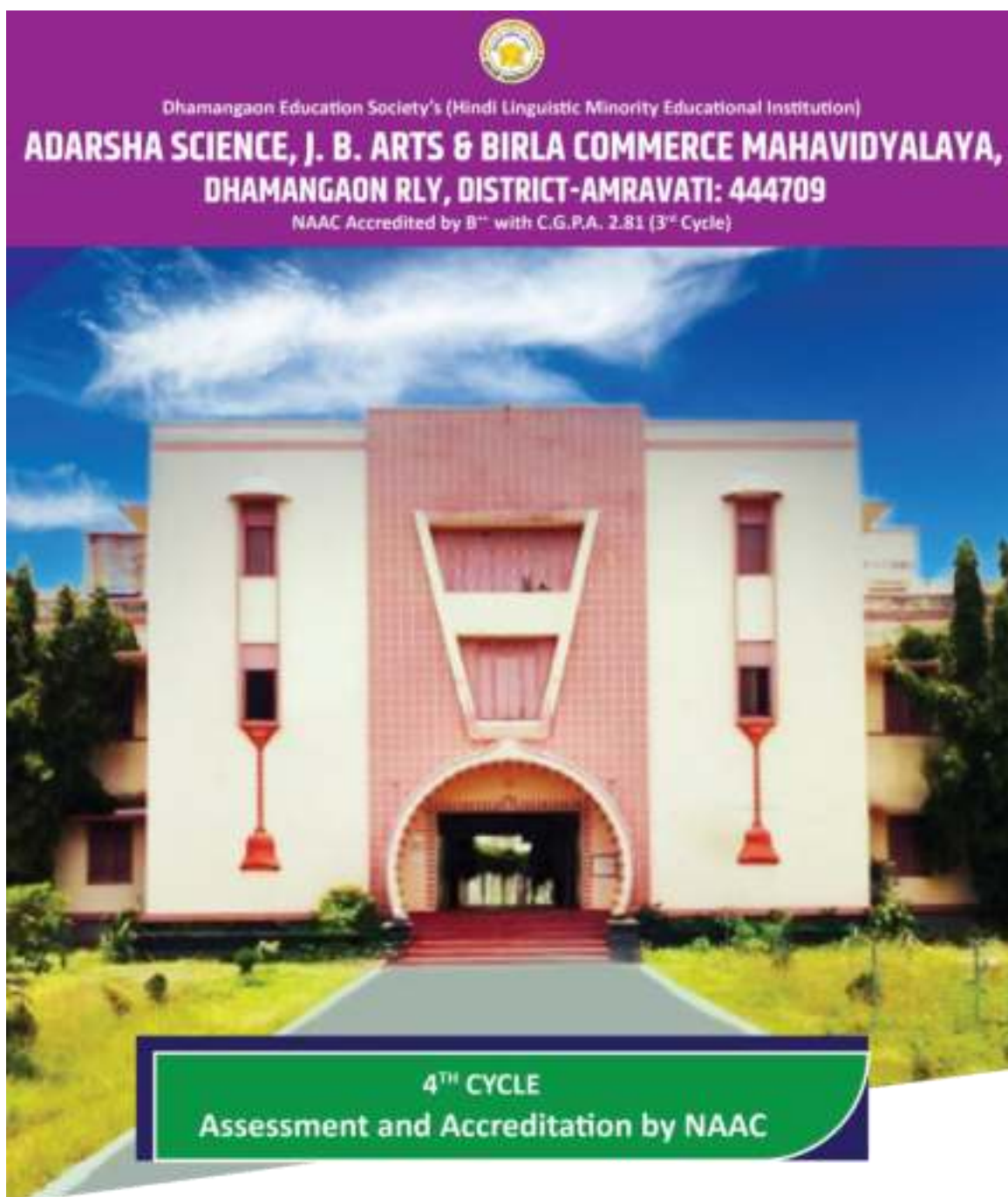




Criterion – III - Research, Innovations and Extension

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Contents

2017-18	7
Separation of Plant Pigments from Medicinal Plants of Solanaceae Family by Using Paper Chromatography: Sandip T. Pendam	7
Octadecanethiol-grafted molybdenum disulfide nanosheets as oil-dispersible additive for reduction of friction and wear: H. P. Mungse	11
Chemically functionalized graphene for lubricant applications: Microscopic and spectroscopic studies of contact interfaces to probe the role of graphene for enhanced tribo-performance : H. P. Mungse	12
GENERALIZED TWO DIMENSIONAL FOURIER-LAPLACE TRANSFORM AND TOPOLOGICAL PROPERTIES: A. N. Rangari	13
NEONATAL SEPSIS DUE TO KLEBSIELLA: FREQUENCY, ANTIBIOTIC SUSCEPTIBILITY AND MOLECULAR CHARACTERIZATION BY 16S RNA ANALYSIS: G. N. Budhlani.....	21
Antibacterial activity of spice essential oils against uropathogenic bacteria: V. A. Kamble	25
Physicochemical Analysis of Water Bodies in Yavatmal: Jitendra Ghanshyam Suryawanshi	28
Studies on Adaptation of NIAS-MaBr-92, a Cell line From Hemocytes of Cabbage Armyworm, Mamestra Brassica to MGM-448 Culture Medium Supplemented with 3% Fetal Bovine Serum: Syed Obaid Qureshi	33
The Effect of Demographic Factors on the Stress and Social Support Related to the Executive Organizational Commitment-Nagpur Region : Anand Naranje	39
Complex analysis of classified of Soil parameters and its relationship identification using PCA : Dr. Maya V. Mawale.....	44
Study on photoluminescence and thermoluminescence properties of UV-irradiated CaSrAl ₂ SiO ₇ :Ce ³⁺ phosphors: Shweta S. Sharma	55
2018-2019	63
RECENT TRENDS IN E-COMMERCE INDUSTRY IN INDIA: RUPESH R. DAHAKE.....	63
E-business - Challenges & Opportunities: Dr Narendra Kawduji Nagpure	69
Negative Capability in Keats' Ode to Psyche: Sanjay Datta Patil	75
Women in Buddhism and Present Scenario: Sanjay Datta Patil	79
Existential Reading of Hamlet: Jeetendrasingh Gangasingh Barulkar	82
Women's Health Problems and Fourier Transform: A Review: Ashwina Nilkanth Rangari ..	87

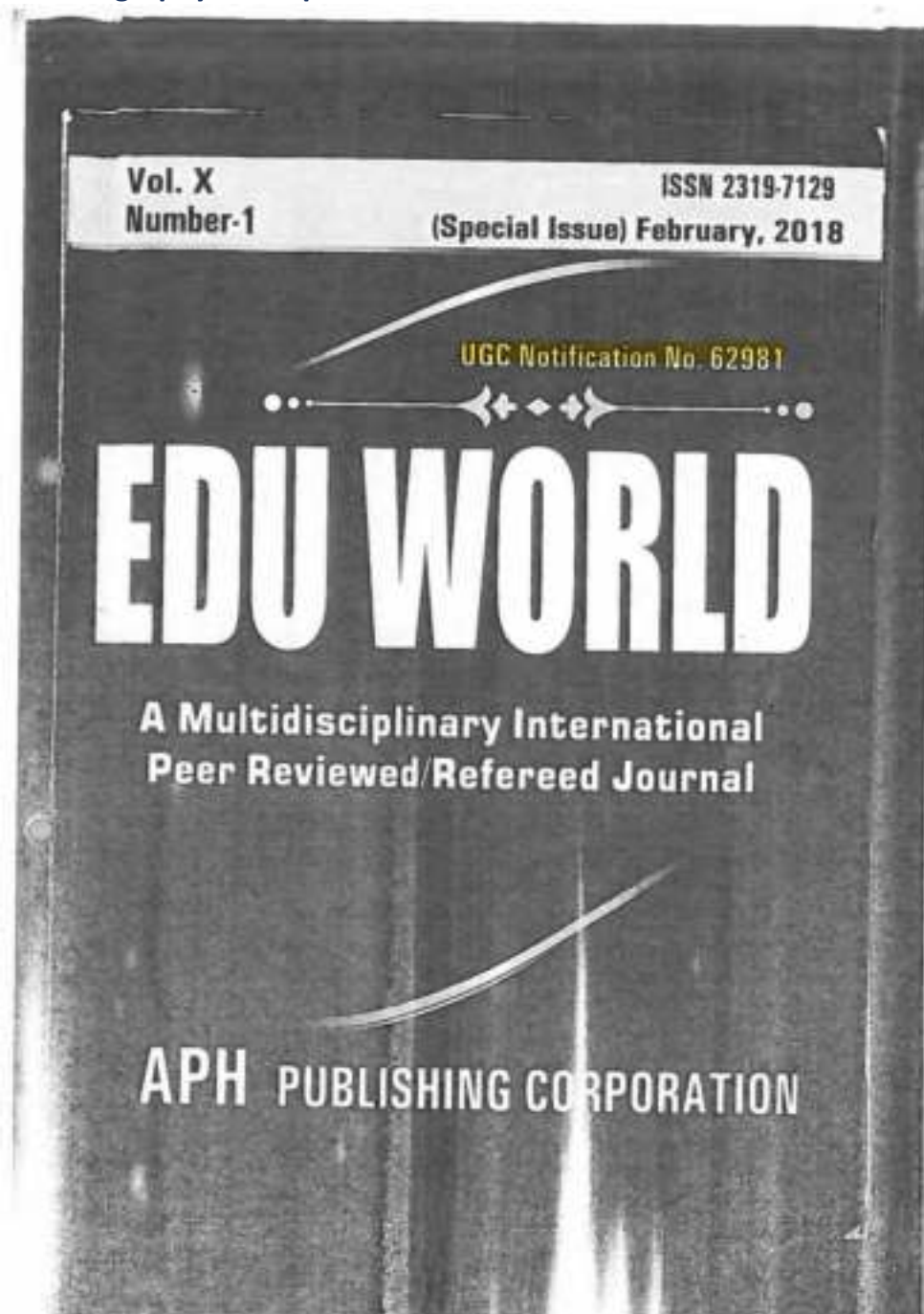
Introduction of Extended Finite Mellin Transform: Ashwina Nilkanth Rangari	93
ANALYTICAL BEHAVIOUR OF DISTRIBUTIONAL TWO-DIMENSIONAL FOURIER LAPLACE TRANSFORM: Ashwina Nilkanth Rangari	98
Two Dimensional Fourier-Laplace Transform and Testing Functional Spaces: Ashwina Nilkanth Rangari	108
Exigobacterium aurantiacum virulent pigment producing a novel pathogenic bacteria associated with cases of corneal ulcers : Dr. Vilas Adinath Kamble.....	117
Dr. Babashaeb Ambedkranche Rajkiy Vichar : : Amol Bhauraoji Band	126
Bhartiya Sansdiya shasan Paddhatil Dosh ani Upay : Amol Bhauraoji Band	132
Argo Entrepreneurship: A vital tool for sustainable agriculture: Anand Gangadhar Naranje	141
SYNTHESIS OF SCHIFF BASES BY CONDENSATION OF AROMATIC AMINES WITH 3-(5-BROMOTHIOPHEN-2-YL)-1-PHENYL-1-H-PYRAZOLE-4-CARBALDEHYDE. : Sachin V. Manohare	147
Investigation of BMI-PF6 ionic liquid/graphite interface using frequency modulation atomic force spectroscopy: Harshal P. Mungase.....	151
Samajsudharna chalvalit Rashtasant Tukdoji Aitihashik Yogdan : Kishor H. Barapatre.....	152
Luminescence properties of near-uv excitable yellow-orange light emitting warm CaSrAl ₂ SiO ₇ :Sm ³⁺ phosphors: Dr. Shweta S Sharma	157
Thermoluminescence glow curve of UV induced Sr ₃ MgSi ₂ O ₈ phosphor with its structural characterization: Dr. Shweta S Sharma	166
Jagtikikarnanantar krushi Kshetrachi disha ani dasa : Dr. Dipak K. Shrugare	173
2019-2020	180
Cr(III),Mn(III),Fe(III) and VO(IV) complexes of 2,4-dihydroxyacetophenone and sulphapyridine.: Vishnu Anandrao Sadafale.....	180
SYNTHESIS AND ANTIBACTERIAL SCREENING OF SCHIFF BASES DERIVED FROM 3-(5-BROMOTHIOPHEN-2-YL)-1-(4-CHLOROPHENYL)-1H-PYRAZOLE-4-CARBALDEHYDE: Sachin V. Manohare.....	187
Synthesis and antimicrobial evaluation of pyrimidines derived from chalcones: Sachin V. Manohare	194

Influence of Dy ³⁺ concentration on spectroscopic behavior of Sr ₃ MgSi ₂ O ₈ :Dy ³⁺ phosphors: Influence of Dy ³⁺ concentration on spectroscopic behavior of Sr ₃ MgSi ₂ O ₈ :Dy ³⁺ phosphors: Dr. Shweta S Sharma	201
Synthesis, Characterization and Gas Sensing Study of SnO ₂ Thick Film Sensor towards H ₂ S, NH ₃ , LPG and CO ₂ : Dr. Suresh Gulabrao Onkar	202
Studies on Cytopathic Effects of BmNPV infection in Three Lepidopteran Insect Cell Lines in MGM-448 Medium: Syed Obaid Qureshi	203
Alkylated graphene oxide and reduced graphene oxide: Grafting density, dispersion stability to enhancement of lubrication properties: Dr. Harshal P. Mungse	207
Synthesis and evaluation of a novel series of 6-bromo-1-cyclopentyl-1H-indazole-4- carboxylic acid-substituted amide derivatives as anticancer, antiangiogenic and antioxidant agents: Vilas A. Kambale	214
IOT Based Flamaeble Gas Monitoring System Using ESP32 for Underground Environment: Suryakant Anandarao Rodge	215
2020-2021	220
Surface Chemistry of Graphene and Graphene Oxide:A Versatile Route for Their Dispersion and Tribological Applications : Ajay Chouhan, Harshal P. Mungse, Om P Khatri.....	220
Generation of cold white light by using energy transfer process in single phase Ce ³⁺ /Tb ³⁺ co-doped CaSrAl ₂ SiO ₇ phosphor: Dr. S. S. Sharma	248
Investigation of structural and thermal response of Sm ³⁺ doped Sr ₃ MgSi ₂ O ₈ phosphors: Dr. S. S. Sharma.....	260
Adaptation of an indigenous insect cell line DZNU-Bm-1 to Mitsunashi and Maramorosch (MM) Culture Medium supplemented with 3% Foetal Bovine Serum: S. O. Qureshi	278
Vedic paramparetun manviya vikasache prapta jhalele yathartha Gyan : Dr. Poonam M. Gahukar	284
String Source in cosmological model for theory of gravitation: Dr. S. N. Bayaskar	291
2021-2022	298
Enhanced thermoluminescence properties of CaSrAl ₂ SiO ₇ : Ce ³⁺ , Tb ³⁺ phosphor : Dr. S. S. Sharma, Dr. S. G. Onkar, V. S. Thool, Dr. S. G. Vidhale, G. S. Mendhe	298
PHOTO-ACTIVITIES OF SrNb ₁₀ O ₆ NANOPARTICLES SYNTHESIS BY COPRECIPITATION METHOD: S. G. Onkar	309

Introductory study on machine learning tool WEKA : Dr. S. A. Rodge, S. K. Shelke, Dr. S. A. Ghogare	322
Development of Linear Array Transducer using Field-II : Dr. S.A.Rodge	328
Simulation of 32 Element linear array Transducer using Field-II GUI: Dr. S.A.Rodge.....	337
Study the response of Ultrasonic linear array transducer with different number of elements: Dr. S.A.Rodge.....	347
Simulation of linear array Transducer with center frequency 5 MHz: Dr. S. A. Rodge.....	354
Comparison of Responses of 2 element and 4 element array transducer at center frequency 5 MHz. : Dr. S. A.Rodge, S. K. Shelke	361
Comparative Study of 4-Element and 8-Element Array Transducers at Center Frequency 5 MH: Dr. S. A. Rodge.....	370
Development of 16 elements linear transducer using field II :Dr. S. A. Rodge; R. D. Chaudhary	379
Linear array Transducer with center frequency 7.5 MHz :Dr. S. A. Rodge.....	388
Design of hybrid model of ultrasonic transducer: Dr. S. A. Rodge	394
Analysis of 4 element and 16 elements linear array transducer 5 MHz : Dr. S. A. Rodge...	401
Surface charge dependent structure of ionic liquid/ alkali halides interfaces investigated by atomic force micros copy: H. P. Mungse	410

2017-18

Separation of Plant Pigments from Medicinal Plants of Solanaceae Family by Using Paper Chromatography: Sandip T. Pendam



Separation of Plant Pigments from Medicinal Plants of Solanaceae Family by Using Paper Chromatography

Sandip T. Pedam*

ABSTRACT

Plants are one of five big groups of living things. The whole plant biodiversity is the largest source of herbal medicine and among these India have 15 agro climatic zones and 17000-18000 species of flowering plants of which 6000-7000 are estimated to have medicinal usage. The Indian medicinal plants and their products also account of exports in the range of Rs. 10 billion. It raises a need of studies in medicinal plants as these are involved in maintaining traditional therapies and also they lead to economical support to the country. In this research paper three different varieties of medicinal plants belongs to Solanaceae families which are well known medicinal plants are selected for the chromatographic experiments and the Rf values were noted down.

Keyword: Medicinal plants, Plant pigments, Paper Chromatography.

INTRODUCTION

Medicinal plants are an accessible, affordable and culturally appropriate source of primary health care for more than 80% of Asia's population (WHO). Marginalized, rural and indigenous people, who cannot afford or access formal health care systems, are especially dependent on these culturally familiar, technically simple, financially affordable and generally effective traditional medicines. As such, there is widespread interest in promoting traditional health systems to meet primary health care needs. This is especially true in South Asia, as prices of modern medicines spiral and governments find it increasingly difficult to meet the cost of pharmaceutical-based health care. Throughout the region, there is strong and sustained public support for the protection and promotion of the cultural and spiritual values of traditional medicines. Many of the modern medicines are produced indirectly from medicinal plants. Plants are directly used as medicines by a majority of cultures around the world. Cultivation and preservation of medicinal plants protect biological diversity.

MEDICINAL PLANTS SUBJUGATED FOR THE RESEARCH WORK

Three different species of medicinal plants belongs to solanaceae family were collected from the different locality. Three plants of each variety were collected, among those three one plant is well known medicinal plant. Later on, these plant samples were collected for the plant pigment separation by using paper chromatography technique. The Plant of our choice was *Datura metel*, *Physalis minima* and *Withania somnifera* as they possess lot of medicinal value.

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Photosynthesis is the main metabolism for the survival of plants. Photosynthesis is the process which transforms light energy from the sun into chemical energy for the plants. This mechanism is done by the presence of plant pigments which are the macro molecules produced by the plants. These pigments absorb specified wavelengths of visible light to provide the energy required for photosynthesis. Chlorophyll is necessary for photosynthesis, but accessory pigments collect and transfer energy to chlorophyll. Although pigments absorb light, the wavelengths of light which are not absorbed by the plant pigments are reflected back. Plant contains different pigments and some of pigments observed include are Chlorophylls (green), Carotenoids (yellow, orange-red), Anthocyanins (red to blue, depending on pH), Betalains (red or yellow).

In Paper chromatography, paper is marked with the plant extract and is placed in a developing chamber with a specified solvent. The solvent carries the dissolved pigments as it moves up the paper. This shows the variation of the pigments from one plant to another. The distance of the pigment travels is unique for the pigment in set conditions and is used to identify the pigment. The ratio is the retention factor R_f value.

MATERIALS AND METHODS

Developing Paper chromatography

1. Cut a piece of Whatman No.1 filter paper or chromatography paper to the dimensions of 15cm long whereas one of the edges must be blunt and other end should be pointed.
2. With a pencil lightly make a line of 1.5-2cm from pointed edge of the paper.
3. Take the extracted plant pigment was painted onto the strips of chromatography paper with the help of V-shaped tips or glass tubes or by using small paint brush. (Fig. A and B)
4. Allow the line to dry, and then repeat the process until a dark green line of pigment is evident to see.
5. Next, Chromatography solvent is used to separate the mixture of pigments painted on the paper.
6. Chromatography solvent is prepared by adding nine parts of petroleum ether to one part of acetone.
7. This chromatography solvent is highly volatile and flammable.
8. The V-Shaped tip of the paper is placed in the chromatography solvent and acts as a wick to draw the solvent up the paper, separating pigments according to their relative solubility and molecular weights.
9. The paper is allowed to remain in the solvent until the uppermost pigment band nears the top of the paper.

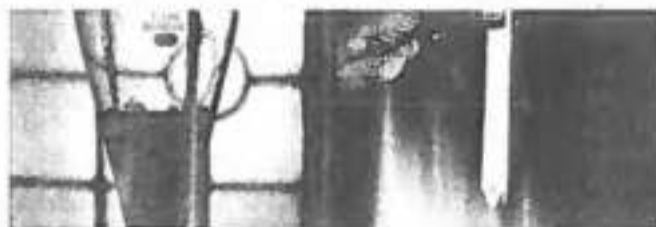


Fig. A

B

This work can be further developed by using more sophisticated equipment like HPLC, TLC, which may lead to create new innovations in pharmacological fields.

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在 1998 年 12 月 1 日以前

Hydrolysis of Me₆Si-ODT by slowly stirring at the constant temperature because of liquid state and exhibits high mechanical strength, which promotes to generate an independent appearance of polymer degradation of Me₆Si, in the future, which has been a great advantage. Here, the polymerization approach is presented to illustrate the Me₆Si monomer and ligand (Me₆Si-ODT) with the substituted Si-OTf. The structural defects and the reaction sites on the Me₆Si-ODT are targeted for grafting in ODT on the Me₆Si-ODT linkage. The chemical and structural defects are characterized by FTIR, NMR, GPC and DSC-TGA analysis. The dispersion stability of Me₆Si-ODT in the polymer latex film is examined by UV-Visible spectroscopy. The case also shows the trend the structural chain of Me₆Si-ODT and the distribution of grafted and free Me₆Si-ODT in Me₆Si-ODT monomers. Thermogravimetric analysis revealed that means during 400 °C and 500 °C, grafted Me₆Si-ODT monomers, as well as, to be free from degradation, whereas, the free Me₆Si-ODT monomers. The structural study of Me₆Si-ODT monomers, as well as, the free Me₆Si-ODT, shows the free monomers in the presence of grafted monomers in the polymer film. Me₆Si, functional and high mechanical strength of Me₆Si-ODT, effectively, enhance the polymer. Thermogravimetric analysis Me₆Si-ODT monomers in an additive in the polymer latex film.

hydrogen and carbon monoxides), crystallization of the polymer [11]. The fraction of MoS₂ homogeneously incorporated in the polymer matrix is determined by the type of layers in the matrix [9]. Calculations of mechanical and electrical properties of nanocomposites with polythiophenes have been established as a function of the content of MoS₂ [12]. The results of the thermal oxidation, photochemical synthesis, electrochromic, gas-phase reduction of MoS₂, and electrochromic properties of MoS₂ nanocomposites have been reported in [13–24]. During the synthesis of MoS₂ nanocomposites, the polymer is considered as a filler, and the MoS₂ is dispersed in the matrix. MoS₂ nanocomposites have been synthesized by the intercalation of MoS₂ into the polymer matrix. The temperature, surface area and complexing agent are the main parameters, which control the size and amount of MoS₂ in the polymer matrix [25].

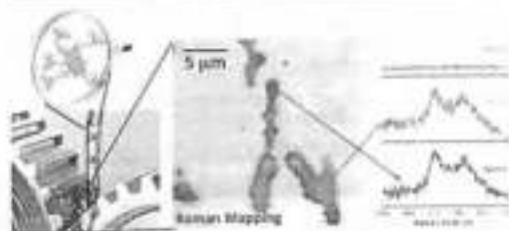
The MoS₂ nanoparticles and nanosheets were used as lubricants in both the tribological tests and the tribocorrosion tests. The MoS₂ nanoparticles, the nanosheet particles and sheets were used as the tribointerfaces and provide significantly improved tribological wear properties [27]. Nanoparticles of MoS₂ and MoS₂ nanosheets in solid lubricants provide excellent triboprotection under dry/severe conditions, where fluids are not available to support a lubricating film. The MoS₂ nanosheet particles are very thin and are separated on the surface of the tribopartner. The tribopartner can be protected by the nanosheet and nanosheet particles in the lubricant. The MoS₂ nanosheet particles are very thin and are separated on the surface of the tribopartner. The tribopartner can be protected by the nanosheet and nanosheet particles in the lubricant.

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Chemically functionalized graphene for lubricant applications: Microscopic and spectroscopic studies of contact interfaces to probe the role of graphene for enhanced tribo-performance: H. P. Mungse



GRAPHICAL ABSTRACT



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Three reduced graphene oxide (rGO) based contact systems were studied by microscopic analyses of steel balls lubricated with chemically functionalized graphene-based oil (G-DO). The 3,3'-diethoxy-4-hydroxybenzophenone (DEHBA) grafted graphene (G-DEHBA) was prepared by two-step approach using epichlorohydrin as a precursor. Chemical and structural analysis of G-DEHBA was carried out by FTIR, XPS, TGA, and DSC. The results show that the DEHBA group is attached to the rGO surface. The tribological studies of G-DEHBA in the G-DO base oil, which is a high viscosity oil, showed that the performance of G-DEHBA as a lubricant additive. The tribological studies of G-DEHBA in the G-DO base oil showed the significant reduction in the wear and friction. The results suggest that the G-DEHBA can be used as a lubricant additive. The tribological studies of G-DEHBA in the G-DO base oil showed the significant reduction in the wear and friction. The results suggest that the G-DEHBA can be used as a lubricant additive. The tribological studies of G-DEHBA in the G-DO base oil showed the significant reduction in the wear and friction. The results suggest that the G-DEHBA can be used as a lubricant additive.

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GENERALIZED TWO DIMENSIONAL FOURIER-LAPLACE TRANSFORM AND TOPOLOGICAL PROPERTIES: A. N. Rangari

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GENERALIZED TWO DIMENSIONAL FOURIER-LAPLACE TRANSFORM AND TOPOLOGICAL PROPERTIES

A. N. Rangari*

V. D. Sharma**

Abstract

Due to wide spread applicability in integral transform for partial differential equations involving distributional conditions, many of integral transform extended to generalized function and in last few years, the theory of generalized integral transforms have been of ever increasing interest due to its application in physics especially in quantum field theory, engineering and pure as well as applied mathematics. It provided new aspect to many mathematical disciplines such as ordinary and partial differential equation, operational calculus transformation theory and functional analysis.

This paper presents the generalization of two dimensional Fourier-Laplace transform in the distributional sense. The testing function spaces using Gelfand-Shilov techniques are defined. Also some topological properties of S-type spaces for distributional generalized two dimensional Fourier-Laplace transform are proved.

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Keywords:

Fourier Transform;
Laplace Transform;
Two dimensional Fourier-Laplace Transform ;
Generalized Function;

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1. Introduction

More recently, a number of results on multidimensional Laplace transforms were proposed by Dahiya. Even with these enormous numbers of useful applications of Laplace transform a systematic account of general theory of operational calculus using Laplace transforms of double, triple and higher dimensions need serious attention. The two dimensional Laplace transformation is useful in the solution of non-homogeneous partial differential equations [1]. Mathematically the two dimensional Laplace transform is defined and denoted by the formula given below:

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The Two Dimensional Laplace transform with the parameters p, v , of function $f(x, y)$ denoted by $L[f(x, y)] = F(p, v)$ and is given by,

$$L[f(x, y)] = F(p, v) = \int_0^{\infty} \int_0^{\infty} e^{-px - vy} f(x, y) dx dy \quad (1.1)$$

The two dimensional Fourier Transform has applied in image filtering in the frequency domain and in signal processing. The Fourier transform itself is translation invariant and its conversion to log polar co-ordinates converts the scale and rotation differences to vertical and horizontal offsets that can be measured [10]. And the formula for this two dimensional Fourier transform is defined and denoted below:

The Two Dimensional Fourier transform with the parameters s, u , of function $f(t, z)$ denoted by $F[f(t, z)] = F(s, u)$ and is given by,

$$F[f(t, z)] = F(s, u) = \int_{-\infty}^{\infty} \int_{-\infty}^{\infty} e^{-i(st+uz)} f(t, z) dt dz \quad (1.2)$$

The Fourier and Laplace transforms play a predominant role in the theory of signals and systems. Mechanical networks consisting of springs, masses and dampers, for the production of shock absorbers for example, processes to analyze chemical components, optical systems, and computer programs to process digitized sounds or images, can all be considered as system for which one can use Fourier and Laplace transform as well [3]. Many authors studied on various integral transforms separately. B. N. Bhosale and M.S. Choudhari [4] and S.M. Khairnar et al. [6] has discussed double transform. Motivated by this we have also defined a combination of integral transform in distributional generalized sense namely two dimensional Fourier-Laplace transform which is given by the formula:

The Two Dimensional Fourier-Laplace transform with parameters s, u, p, v , of $f(t, z, x, y)$ is defined as,

$$FL\{f(t, z, x, y)\} = F(s, u, p, v) = \int_{-\infty}^{\infty} \int_{-\infty}^{\infty} \int_0^{\infty} \int_0^{\infty} f(t, z, x, y) e^{-i(st+uz)-i(px+vy)} dt dz dx dy \quad (1.3)$$

where the kernel $K(s, u, p, v) = e^{-i(st+uz)-i(px+vy)}$.

Along with this we have developed some testing function spaces and proved some topological properties.

This paper is summarized as follows: Some testing function spaces which are useful for proving topological properties are given in section 2. In section 3, we have given definition of Distributional generalized Two dimensional Fourier-Laplace transform. Some topological properties of S-type spaces are proved in section 4. And in section 5, some results on strict inductive limit spaces are given. Finally we conclude the paper.

The notations and terminologies are as per Zemanian [11], [12].

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2. Testing Function Spaces

2.1. The Space $FL_{a,b,c,d,\alpha}$ (S_a -type space):

Let I be the open set in $R_s \times R_s$ and E_s denotes the class of infinitely differentiable function defined on I , the space $FL_{a,b,c,d,\alpha}$ is given by

$$FL_{a,b,c,d,\alpha} = \left\{ \phi : \phi \in E_s / \gamma_{a,b,c,d,\alpha} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} \left| t^k z^r K_{a,b}(x) R_{c,d}(y) D_t^j D_z^q D_x^s D_y^m [\phi(t, z, x, y)] \right| \leq C_{i,q,s,m} A^k k^{i\alpha} B^r r^{j\alpha} \right\}$$

(2.1)

where the constants A, B and $C_{i,q,s,m}$ depend on the testing function ϕ .

2.2. The space $FL_{a,b,c,d,\beta}$ (S_r -type space):

This space is given by,

$$FL_{a,b,c,d,\beta} = \left\{ \phi : \phi \in E_s / \xi_{a,b,c,d,\beta} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} \left| t^k z^r K_{a,b}(x) R_{c,d}(y) D_t^j D_z^q D_x^s D_y^m [\phi(t, z, x, y)] \right| \leq C_{k,r,j,q} A^k q^{r\beta} B^m m^{r\beta} \right\}$$

(2.2)

where the constants A, B and $C_{k,r,j,q}$ depend on the testing function ϕ .

2.3. The space $FL_{a,b,c,d,\alpha,\beta,h}$:

It is defined as,

$$FL_{a,b,c,d,\alpha,\beta,h} = \left\{ \phi : \phi \in E_s / \gamma'_{a,b,c,d,\alpha,\beta,h} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} \left| t^k z^r K_{a,b}(x) R_{c,d}(y) D_t^j D_z^q D_x^s D_y^m [\phi(t, z, x, y)] \right| \leq C_{i,q,s,m,\alpha,\beta,h} (g + \delta)^k k^{i\alpha} (h + \mu)^r r^{j\beta} \right\}$$

(2.3)

For any $\delta > 0$, $\mu > 0$ and g, h are the constants depending on the function ϕ .

The space $FL_{a,b,c,d,\alpha}$ and $FL_{a,b,c,d,\alpha,\beta,h}$ are equipped with their natural Hausdorff locally convex topologies $T_{a,b,c,d,\alpha}$ and $T_{a,b,c,d,\alpha,\beta,h}$. These topologies are respectively generated by the total families of seminorms $\{\gamma_{a,b,c,d,\alpha}[\phi]\}$ and $\{\gamma'_{a,b,c,d,\alpha,\beta,h}[\phi]\}$ given by (2.1) and (2.3).

3. Distributional Generalized Two Dimensional Fourier-Laplace Transform (2DFLT):-

For $f(t, z, x, y) \in FL'_{a,b,c,d,\alpha}$, where $FL'_{a,b,c,d,\alpha}$ is the dual space of $FL_{a,b,c,d,\alpha}$. It contains all distributions of compact support. The distributional two dimensional Fourier-Laplace transform is a function of $f(t, z, x, y)$ is defined as,

$$FL\{f(t, z, x, y)\} = F(u, p, v) = \langle f(t, z, x, y), \phi(t, z, x, y, s, u, p, v) \rangle,$$

(3.1)

Where $\phi(t, z, x, y, s, u, p, v) = e^{-\frac{\alpha}{2}(t^2 + z^2 + x^2 + y^2 + s^2 + u^2 + p^2 + v^2)}$ and for each fixed $t(0 < t < \infty)$, $z(0 < z < \infty)$, $x(0 < x < \infty)$ and $y(0 < y < \infty)$. Also $s > 0$, $u > 0$, $p > 0$ and $v > 0$. The right hand side of (3.1) has a sense as an application of $f(t, z, x, y) \in FL_{a,b,c,d,n}^*$ to $\phi(t, z, x, y, s, u, p, v) \in FL_{a,b,c,d,n}$.

4. Topological Properties of S-type spaces:

4.1. Theorem: $(FL_{a,b,c,d,n}; T_{a,b,c,d,n})$ is a Frechet space.

Proof:- As the family $D_{a,b,c,d,n}$ of seminorms $\{Y_{a,b,c,d,r,q,m,l,n}\}_{a,b,c,d,r,q,m,l,n=0}^{\infty}$ generating $T_{a,b,c,d,n}$ is countable, it suffices to prove the completeness of the space $(FL_{a,b,c,d,n}; T_{a,b,c,d,n})$.

Let us consider a Cauchy sequence $\{\phi_n\}$ in $FL_{a,b,c,d,n}$. Hence for a given $\varepsilon > 0$, there exist an $N = N_{a,b,c,d,r,q,m,l,n}$ such that for $m, n \geq N$,

$$Y_{a,b,c,d,r,q,m,l,n}(\phi_m - \phi_n) = \sup_{I_1} |t^k z^r K_{a,b}(x) R_{c,d}(y) D_i^l D_s^q D_z^m D_v^n (\phi_m - \phi_n)| < \varepsilon \quad (4.1.1)$$

In particular for $k = r = q = m = l = n = 0$, for $m, n \geq N$,

$$\sup_{I_1} |K_{a,b}(x) R_{c,d}(y) (\phi_m(t, z, x, y) - \phi_n(t, z, x, y))| < \varepsilon \quad (4.1.2)$$

Consequently, for fixed (t, z, x, y) in I_1 , $\{\phi_n(t, z, x, y)\}$ is a numerical Cauchy sequence.

Let $\phi(t, z, x, y)$ be the point-wise limit of $\{\phi_n(t, z, x, y)\}$ using (4.1.2) we can easily deduce that $\{\phi_n\}$ converges to ϕ uniformly on I_1 . Thus ϕ is continuous.

Moreover, repeated use of (4.1.1) for different values of k, r, q, m, l, n yields that ϕ is smooth that is $\phi \in E_s$. Further from (4.1.1) we get,

$$Y_{a,b,c,d,r,q,m,l,n}(\phi_m) \leq Y_{a,b,c,d,r,q,m,l,n}(\phi_N) + \varepsilon, \quad \forall m \geq N \\ \leq C_{lqmn} A^k k^{ka} B^r r^{ra} + \varepsilon.$$

Taking $m \rightarrow \infty$ and ε is arbitrary we get,

$$Y_{a,b,c,d,r,q,m,l,n}(\phi) = \sup_{I_1} |t^k z^r K_{a,b}(x) R_{c,d}(y) D_i^l D_s^q D_z^m D_v^n \phi(t, z, x, y)| \leq C_{lqmn} A^k k^{ka} B^r r^{ra}$$

Hence $\phi \in FL_{a,b,c,d,n}$ and it is the $T_{a,b,c,d,n}$ limit of ϕ_n by (4.1.1) again. This proves the completeness of $FL_{a,b,c,d,n}$ and our proof is completed. For the justification of our study of two dimensional Fourier-Laplace spaces, that is the non triviality of these spaces we have proved the following theorem.

4.2. Theorem: The space $D(I_1)$ is a subspace of $FL_{a,b,c,d,n}$ such that the injection mapping from $D(I_1)$ to $FL_{a,b,c,d,n}$ is continuous that is $T_{a,b,c,d,n}/D(I_1) \subset T(I_1)$.

67

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Proof: For $\phi(t, z, x, y) \in D(I_1)$, set

$$L = \sup_{I_1} \{t, z : (t, z, x, y) \in \text{supp } \phi\} \text{ and } C_{\text{qgm}} = \sup_{I_1} \{K_{a,b}(x) R_{c,d}(y) D_1^j D_2^q D_3^r \phi(t, z, x, y)\}$$

then,

$$\begin{aligned} \gamma_{a,b,c,d,h,r,q,m,j,n} \phi(t, z, x, y) &= \sup_{I_1} |t^k z^r K_{a,b}(x) R_{c,d}(y) D_1^j D_2^q D_3^r \phi(t, z, x, y)| \leq C_{\text{qgm}} L^k N^r \\ &= C_{\text{qgm}} L^k N^r \frac{k^{ka}}{k^{ka}} \cdot \frac{A^k}{A^k} \cdot \frac{r^{ra}}{r^{ra}} \cdot \frac{B^r}{B^r} \\ &= C_{\text{qgm}} \left(\frac{L}{Ak^a}\right)^k \left(\frac{N}{Br^a}\right)^r A^k k^{ka} B^r r^{ra} \end{aligned}$$

(4.2.1)

$$\text{Since } \left(\frac{L}{Ak^a}\right) \leq 1 \text{ and } \left(\frac{N}{Br^a}\right) \leq 1 \text{ iff } k \geq \left(\frac{L}{A}\right)^{\frac{1}{a}} \text{ and } r \geq \left(\frac{N}{B}\right)^{\frac{1}{a}},$$

$$\text{define } k_0 = \left\lceil \left(\frac{L}{A}\right)^{\frac{1}{a}} \right\rceil + 1 \text{ and } r_0 = \left\lceil \left(\frac{N}{B}\right)^{\frac{1}{a}} \right\rceil + 1, \text{ where } [t, z] \text{ denotes the Gaussian symbol,}$$

that is the greatest integer not exceeding t, z . Therefore for $k > k_0, r > r_0$, we have

$$\gamma_{a,b,c,d,h,r,q,m,j,n} \phi(t, z, x, y) \leq C_{\text{qgm}} A^k k^{ka} B^r r^{ra} \quad (4.2.2)$$

If $k \leq k_0$ and $r \leq r_0$, let us write,

$$C = \max \left\{ \left(\frac{L}{A}\right) \left(\frac{N}{B}\right), \left(\frac{L}{A_2}\right)^2 \left(\frac{N}{B_2}\right)^2, \left(\frac{L}{A_3}\right)^3 \left(\frac{N}{B_3}\right)^3, \dots, \left(\frac{L}{A_{k_0}}\right)^{k_0} \left(\frac{N}{B_{r_0}}\right)^{r_0} \right\}$$

Then again from (4.2.1)

$$\gamma_{a,b,c,d,h,r,q,m,j,n} \phi(t, z, x, y) \leq CC_{\text{qgm}} A^k k^{ka} B^r r^{ra} \quad (4.2.3)$$

Hence the inequalities (4.2.2) and (4.2.3) together yield,

$$\gamma_{a,b,c,d,h,r,q,m,j,n} \phi(t, z, x, y) \leq C_{\text{qgm}}^* A^k k^{ka} B^r r^{ra}, \quad \forall k \geq 0, r \geq 0, \text{ where } C_{\text{qgm}}^* = CC_{\text{qgm}}.$$

Implying that $\phi \in FL_{a,b,c,d,h,r,q,m,j,n}$. Consequently, $D(I_1) \subset FL_{a,b,c,d,h,r,q,m,j,n}$. To prove that continuity of the injective map, consider a sequence $\{\phi_n\}$ in $D(I_1)$, converging to zero. Then

$$\gamma_{a,b,c,d,h,r,q,m,j,n}(\phi_n) = \sup_{I_1} |t^k z^r K_{a,b}(x) R_{c,d}(y) D_1^j D_2^q D_3^r \phi_n(t, z, x, y)| \quad (4.2.4)$$

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We find a compact subset Q_n (say) of I_1 such that $\phi_n \in D(Q_n)$ for each $n \geq 1$ and $\phi_n \rightarrow 0$ in

$D(Q_n)$. If $P = \sup_{Q_n} |t^k z^r K_{\alpha, \beta}(x) R_{\gamma, \delta}(y)|$; then from (4.2.4),

$$\gamma_{\alpha, \beta, \gamma, \delta, r, q, m, j, n}(\phi_n) \leq P \sup_{Q_n} |D_1^{i, q} D_2^{n, m} \phi_n(t, z, x, y)| \rightarrow 0 \text{ as } n \rightarrow \infty$$

Hence $\phi_n \rightarrow 0$ in $FL_{\alpha, \beta, \gamma, \delta, r}$ and therefore $T_{\alpha, \beta, \gamma, \delta, r} / D(I_1) \subset T(I_1)$.

5. Results on strict Inductive Limit Spaces:

5.1. Theorem: If $g_1 < g_2$, $h_1 < h_2$, then $FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_1, h_1} \subset FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_2, h_2}$. The topology of $FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_1, h_1}$ is equivalent to the topology induced on $FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_1, h_1}$ by $FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_2, h_2}$ i.e. $T_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_1, h_1} \square T_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_2, h_2} / FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_1, h_1}$.

Proof: For $\phi \in FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_1, h_1}$

$$\begin{aligned} \gamma_{\alpha, \beta, \gamma, \delta, r, q, m, j, n}(\phi) &\leq C_{lqnmjpr} (g_1 + \delta)^k k^{ku} (h_1 + \mu)^r r^{ra} \\ &\leq C_{lqnmjpr} (g_2 + \delta)^k k^{ku} (h_2 + \mu)^r r^{ra} \end{aligned}$$

Thus $FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_1, h_1} \subset FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_2, h_2}$.

The second part is clear from the definition of topologies of these spaces.

5.2. Theorem: $FL_{\alpha, \beta, \gamma, \delta, r} = \bigcup_{i=1}^{\infty} FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_i, h_i}$ and if the space $FL_{\alpha, \beta, \gamma, \delta, r}$ is equipped with the strict inductive limit topology $S_{\alpha, \beta, \gamma, \delta, r, q, m, j, n}$ defined by the injective map from $FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_i, h_i}$ to $FL_{\alpha, \beta, \gamma, \delta, r}$ then the sequence $\{\phi_n\}$ in $FL_{\alpha, \beta, \gamma, \delta, r}$ converges to zero if $\{\phi_n\}$ is contained in some $FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_i, h_i}$ and converges to zero.

Proof: Once we show that

$$FL_{\alpha, \beta, \gamma, \delta, r} = \bigcup_{i=1}^{\infty} FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_i, h_i}.$$

Clearly $\bigcup_{i=1}^{\infty} FL_{\alpha, \beta, \gamma, \delta, r, q, m, j, n, g_i, h_i} \subset FL_{\alpha, \beta, \gamma, \delta, r}$.

For proving the other inclusion, let $\phi \in FL_{\alpha, \beta, \gamma, \delta, r}$ then

$$\begin{aligned} \gamma_{\alpha, \beta, \gamma, \delta, r, q, m, j, n}(\phi) &= \sup_{I_1} |t^k z^r K_{\alpha, \beta}(x) R_{\gamma, \delta}(y) D_1^i D_2^q D_3^r D_4^m \phi(t, z, x, y)| \\ &\leq C_{lqnmjpr} A^k k^{ku} B^r r^{ra} \end{aligned}$$

(5.2.1)

where A and B are some positive constants. Choose an integer $g = g_A$, $h = h_B$ and $\delta > 0$,

$\mu > 0$ such that $C_{lqnmjpr} A^k B^r \leq C_{lqnmjpr} (g + \delta)^k (h + \mu)^r$

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Then from (5.2.1) we immediately get $\phi \in FL_{a,b,c,d,\alpha,\beta,\gamma,\delta}$, implying that

$$FL_{a,b,c,d,\alpha} \subset \bigcup_{i=1}^{\infty} FL_{a,b,c,d,\alpha,\beta_i,\gamma_i,\delta_i}.$$

6. Conclusion

This paper is concerned with the generalization of two dimensional Fourier-Laplace Transform in the distributional sense. Testing function space using Gelfand-Shilov technique is developed. Topological properties are proved by using the testing function spaces. Also results on strict inductive limit spaces are proved.

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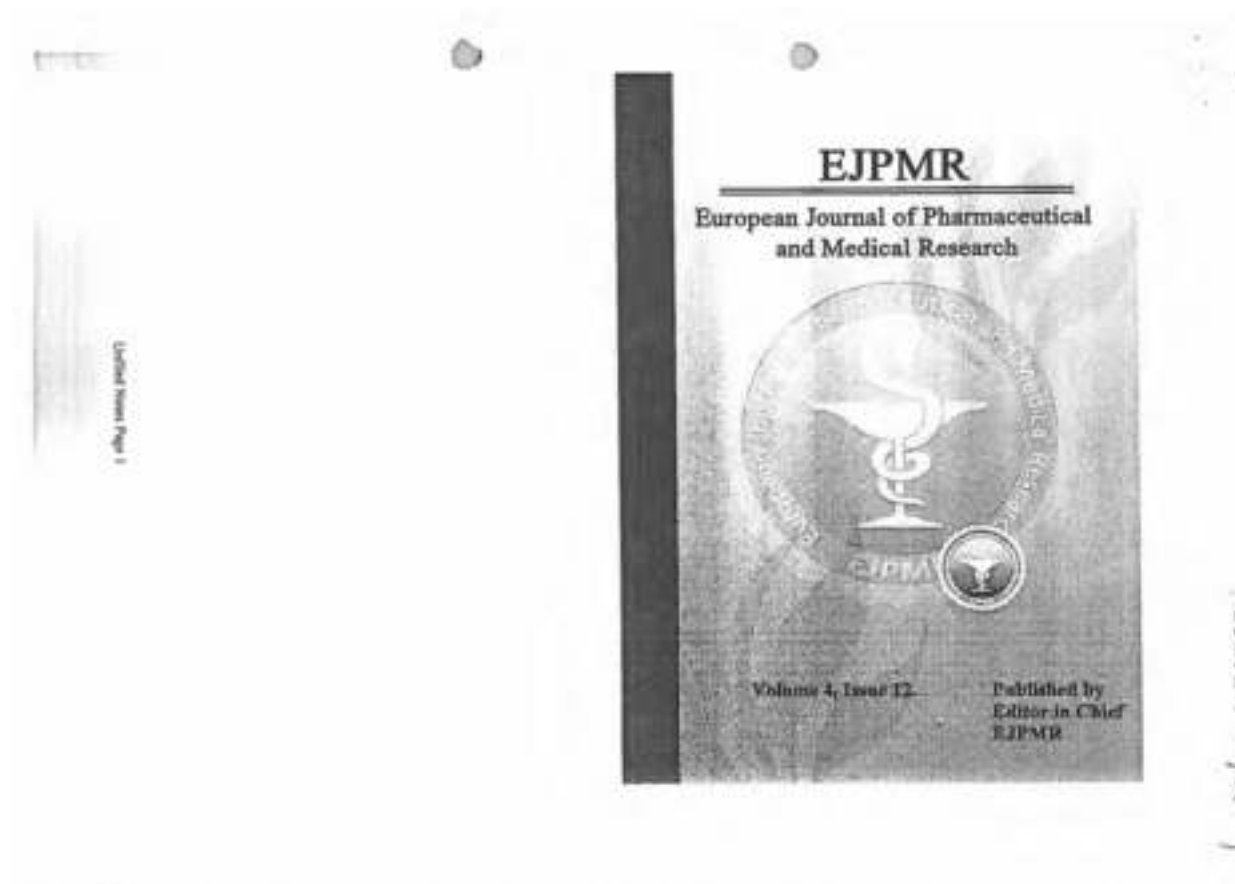
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NEONATAL SEPSIS DUE TO KLEBSIELLA: FREQUENCY, ANTIBIOTIC SUSCEPTIBILITY AND MOLECULAR CHARACTERIZATION BY 16S RNA ANALYSIS: G. N. Budhlani





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EJPMRNEONATAL SEPSIS DUE TO KLEBSIELLA: FREQUENCY, ANTIBIOTIC
SUSCEPTIBILITY AND MOLECULAR CHARACTERIZATION BY 16S RRNA
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ABSTRACT

Sepsis is a significant cause of morbidity and mortality in neonates. The focus of this study was to isolate and to identify *Klebsiella pneumoniae*, and its confirmation by 16s rRNA analysis. Present study revealed that *Klebsiella pneumoniae* is the commonest organism responsible for neonatal sepsis. One hundred and nineteen (119) neonates had positive blood cultures due to *Klebsiella* infection out of 1000 cases. Patients with *Klebsiella* septicemia were categorized into two groups of early and late-onset sepsis. Present study showed Ampicillin resistance in 97.5% isolates and Penicillin resistance in 96.6 % isolates. Above 50% isolates exhibited resistance against Erythromycin, Norfloxacin, Tetracycline and Ciprofloxacin. 97.5% isolates of *Klebsiella* spp. were found to be most sensitive to Imipenem. This study of *Klebsiella* spp. causing neonatal sepsis and their sensitivity pattern is useful so that guidelines can be prepared for empirical antibiotic therapy.

KEYWORDS: *Klebsiella*, early and late onset, antibiotic susceptibility, 16s rRNA analysis.

INTRODUCTION

Sepsis is normally defined as bacteremia in combination with systemic inflammatory response syndrome, but there is no widely accepted definition for neonatal sepsis (Haque KN, 2005). Neonatal sepsis may be categorized as early-onset or late. Early onset sepsis (EOS), present within 48-72 hours of life. Onset is most rapid in premature neonates. Early-onset sepsis is associated with acquisition of microorganisms from the mother and source of infection is generally the maternal genital tract. Clinically, neonates are observed usually with respiratory distress and pneumonia. Presence of some prenatal risk factors has been associated with an increased risk of early onset sepsis (Kahan H, Kinney JS, 1998). Late-onset sepsis occurs (LOS) after 72 hours of age. The source of infection is either nosocomial or community-acquired and neonates usually present with septicemia, pneumonia or meningitis (Baltimore RS., 1998; Wolach B, 1997).

According to recent data from National Neonatal Perinatal Database (NNPD) 2020, the incidence of neonatal sepsis has been reported to be 38 per 1000 intramural live births in tertiary care institutions (National Neonatology Forum, 2000). Septicemia is the commonest clinical category with an incidence of 24 per 1000 live births. Meningitis is diagnosed in 0.5 per 1000 live births. Neonatal sepsis is one of the common causes

of neonatal mortality contributing to 23% of all neonatal deaths (National Neonatology Forum, 2000). *Klebsiella pneumoniae* is the most frequently isolated pathogen (31.2%), followed by *Staphylococcus aureus* (17.5%) among the intramural live births. Among extramural babies admitted for neonatal problems, *Klebsiella pneumoniae* is the commonest organism (36.4%), followed by *Staphylococcus aureus* (14.3%) and *Pseudomonas* (13.2%).

Neonatal nosocomial infections are an important cause of neonatal morbidity and mortality, however it's reporting in India has been non-uniform (Pawa *et al.*, 1997). This may be due to the lack of surveillance work and scanty studies on this aspect. The reported incidence of nosocomial sepsis in India ranges from 1.5-379%. Although any pathogen may be acquired by the neonates in the hospital, *Klebsiella* is lately emerging as an important cause of neonatal nosocomial infection (Gupta *et al.*, 1993).

Sepsis with Gram negative microorganisms are increasingly reported nowadays particularly in Asian countries (Joshi *et al.*, 2000). The inadvertent use of broad-spectrum antibiotics has led to the emergence of multidrug resistant Gram-negative bacteria (Koksal *et al.*, 2001). *Klebsiella* species are of significant importance in this regard (Roidides *et al.*, 2000).

MATERIALS AND METHOD

Our study is based on a prospective analysis of 1000 neonates who admitted to preterm unit and intensive care unit (ICU) in different hospitals of Akola city. All newborns admitted during the period of study with one or more symptoms/signs suggestive of sepsis with or without risk factors of sepsis were recruited into the study. Babies who had received antibiotics prior to presentation as well as those whose mothers had received antibiotics within one week prior to delivery were excluded from the study.

During a period of research work, neonates who admitted were investigated for early onset sepsis (0-7 days of age) and late onset sepsis (>7-90 days of age). For every neonate recruited, 2 ml of venous blood was collected in sterile tubes containing anticoagulant from a peripheral vein. The blood culture specimens were processed according to standard methods in the microbiology laboratory.

All blood cultures were incubated in brain heart infusion broth at 37°C and inspected daily for 3 days for presence of visible microbial growth by observing any of one of the following: turbidity, haemolysis, air bubbles (gas production) and coagulation of broth, otherwise the results were considered as negative for microbial growth. Subcultures were made during three successive days on nutrient agar, blood agar, and MacConkey's agar. The inoculated plates were incubated under aerobic conditions for 24 hrs. *Klebsiella* isolates obtained were identified by standard microbiological techniques, namely, colony characteristics, and biochemical properties including cultural characteristics, IMViC tests and carbohydrate utilization tests. For molecular level confirmation, multidrug resistant strains were sent for characterization by 16s rRNA analysis in Yeast Xenomics DNA sequencing service, Madurai (Chennai Branch), Tamil Nadu, (India).

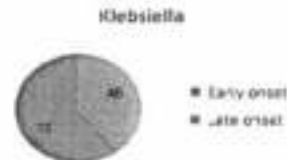
Simultaneously, by using Kirby-Bauer disc diffusion method (1966) antibiotic susceptibility patterns of the *Klebsiella* isolates was determined against Chloramphenicol, Ampicillin, Cefotaxime, Penicillin, Cefazidime, Cefixoxime, Ciprofloxacin, Erythromycin, Carbapenem, Norfloxacin, Imipenem, Gentamicin, Meropenem, Nalidixic acid, Tetracycline, Amoxycylav, Vancomycin, Amikacin, Fuszolidone, Azithromycin.

RESULTS AND DISCUSSION

One hundred and nineteen (119) neonates had positive blood cultures due to *Klebsiella* infection out of 1000 cases. Patients with *Klebsiella* septicemia were categorized into two groups of early and late-onset sepsis. Out of 119 cases, 46 (38.6%) were early onset sepsis (EOS) and 73 were late-onset sepsis (LOS).

Table 1: Frequency in early onset and late onset of *Klebsiella*.

Organisms	Frequency N (%)	Early onset N (%)	Late onset N (%)
<i>Klebsiella</i>	119(16.1)	46(28.6)	73(61.3)



The antibiotic susceptibility pattern of *Klebsiella* isolates was analysed against several antibiotics by disc diffusion method on Mueller Hinton Agar (Table 1). Present study showed Ampicillin resistance in 97.5% isolates and Penicillin resistance in 96.6 % isolates. Above 50% isolates exhibited resistance against Erythromycin, Norfloxacin, Tetracycline and Ciprofloxacin. 97.5% isolates of *Klebsiella* spp. were found to be most sensitive to Imipenem. These findings were similar to that reported by Misra *et al.*, as they found *Klebsiella* isolates were resistant to Ampicillin. Similarly in the study of Roy *et al.*, Ampicillin resistance was observed in 100% isolates and 100% isolates of *Klebsiella* spp. were found to be sensitive to Imipenem and 40% isolates exhibited resistance against Erythromycin, Gentamicin, Tetracycline and Ciprofloxacin.

Table 2: Antibiotic activity against *Klebsiella* Spp. n = 119

Antibiotics	<i>Klebsiella</i> Spp. n = 119	
	S%	R%
Chloramphenicol	82 68.9%	37 31.1%
Ampicillin	3 2.5%	116 97.5%
Cefotaxime	57 47.9%	62 52.1%
Penicillin	4 3.4%	115 96.6%
Cefazidime	52 43.6%	67 56.4%
Cefixoxime	78 65.5%	41 34.5%
Ciprofloxacin	46 38.7%	73 61.3%
Erythromycin	59 49.5%	60 50.5%
Carbapenem	97 81.5%	22 18.5%
Norfloxacin	32 26.8%	87 73.2%
Imipenem	116 97.5%	3 2.5%
Gentamicin	76 63.8%	43 36.2%
Meropenem	114 95.8%	5 4.2%
Nalidixic acid	64 53.7%	55 46.3%
Tetracycline	41 34.5%	78 65.5%
Amoxycylav	82 68.9%	37 31.1%
Vancomycin	88 73.9%	31 26.1%
Amikacin	106 89%	13 11%
Azithromycin	91 76.5%	28 23.5%
Fuszolidone	102 85.7%	17 14.3%

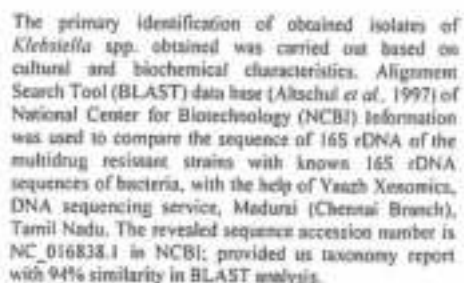


Table 3: Sequences producing significant alignments

Accession No.	Description	Total Score	Query Coverage	E value	Max Ident.
NC_014833.1	<i>Streptococcus pneumoniae</i> subsp. <i>pneumoniae</i> H511286 plasmid pKP95L, complete sequence	897	94%	0.0	93%



CONCLUSION

Study concluded that *Klebsiella* spp. was the predominant organism of neonatal septicemia, which was most frequently observed in late onset so it can be refer as hospital acquired infection and it was found to be almost 100% resistant to commonly used antibiotic Ampicillin and Penicillin whereas the highest sensitivity were observed against Imipenem, Meropenem and Amikacin. Molecular characterization by 16s rRNA analysis confirmed that neonatal septicemia majorly cause by the genus *Klebsiella pneumoniae* pathogen.

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Antibacterial activity of spice essential oils against uropathogenic bacteria: V. A. Kamble

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RESEARCH ARTICLE

Antibacterial activity of spice essential oils against uropathogenic bacteria

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adaptations are made.

ABSTRACT

The medicinal values of many spices and condiments used in Indian cooking have been known from centuries. The aim of this study is to evaluate the antibacterial activity of spice essential oils which are used in kitchen as uropathogenic bacteria. Urinary tract infection is second most common infection next to Respiratory tract in our human body. The indiscriminate use of antimicrobial drugs has led to the resistance in uropathogens globally. In present scenario, alternative and safe therapy is urgently needed. Essential oils of ajeain (*Trachyspermum ajacoides*), cardamom (*Amomum subulatum*), clove (*Syzygium aromaticum*), cinnamon (*Cinnamomum zeylanicum*), black pepper (*Piper nigrum*) and turmeric (*Curcuma longa*) were used in this study. Their antibacterial activity against various uropathogenic bacteria viz. *Escherichia coli*, *Staphylococcus aureus*, *Klebsiella pneumoniae*, and *Pseudomonas aeruginosa* was determined by disc diffusion method. The minimal inhibitory concentrations (MICs) of oils were determined by broth microdilution method. Among all oils ajeain showed highest inhibitory activity with zone of inhibition ranging from 40 to 35 mm diameter against all uropathogenic bacteria. Turmeric was found to be ineffective against all uropathogenic bacteria. Cardamom and clove also showed strong activity for all uropathogens. Activity of black pepper and cinnamon oil was found to be moderate against uropathogens.

Keywords: Spice, Spice essential oils, Uropathogen, Antibacterial activity.

INTRODUCTION

Urinary Tract Infection (UTI) is the second most common infection in humans (Tablan et al., 2008). Different sex and age group of people are affected by urinary tract infection. Sometimes, the UTI is symptomatic or asymptomatic and complicated or uncomplicated. Usually UTI infection is confirmed by significant bacteriuria, i.e., 10^5 organisms/ml (Astrand et al., 2015). UTI refers to the presence of clinical signs and symptoms arising from the genitourinary tract associated with the presence of one or more microorganisms. UTIs are usually localized to the bladder, kidneys or prostate. *Escherichia coli* is the predominant uropathogen

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37

UGC Approved List of Journals-June 2017

Sl.No.	Journal No	Title	Publisher	ISSN	E-ISSN
1	138	Aaps Journal	Springer	15507416	15507416
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3	140	Aaps Pharmstech	Springer	15309932	15309932
4	466	Acta Farmaceutica Bonaerense	Colegio De Farmaceuticos De La Provincia De Buenos Aires	03262383	
5	692	Acta Pharmaceutica	Hrvatsko Farmaceutsko Drustvo (Hfd)-Croatian Pharmaceutical Soc	13300075	18469558
6	693	Acta Pharmaceutica Fennica	Suomen Farmaseutinen Yhdistys	03563456	
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Physicochemical Analysis of Water Bodies in Yavatmal

Jilendra Ghanshyam Suryawanshi*

ABSTRACT

Water is immense natural resource on planet earth. Increase in urbanization, agriculture activity and lots human activities have increased the pollution of surface water as well as ground water. The nature of drinking water is affected by various factors which include chemical, physical and biological factors. The quality of drinking water is important for the health. In view of significant health issues arising out of polluted water bodies, quality of the water must be tested for various contaminants. The objective of this research is to provide information on the physicochemical and ecological parameters of water bodies in Yavatmal. While experimentation on drinking water supplied by municipal corporation, it was found that various parameters such as water temperature, total dissolved solids, hardness, pH, alkalinity, dissolved oxygen, chemical oxygen demand, biological oxygen demand, conductivity and chloride analyzed were within the limits according to WHO (1971) and BIS (1991). The physicochemical properties of water samples found influencing the biotic communities and primary productivity of the water bodies at different sites. Present study depicts the physico-chemical analysis of drinking water in Yavatmal, District Amravati, Maharashtra (India).

Keywords: Water, Physico-chemical investigation, Ecology.

INTRODUCTION

Water is important natural resource on earth. Increase in industrialization, urbanization, agriculture activity and various human activities have increased the pollution of surface water as well as ground water. The nature of drinking water is affected by various factors which include chemical, physical and biological factors. The quality of drinking water is important for the health. In view of significant health issues arising out of polluted water bodies, quality of the water must be tested for various contaminants.

Physico-chemical analysis of drinking water of Yavatmal of Maharashtra state has been studied from January 2016-2017. Municipal corporation water and Bore well water is generally used for drinking and other domestic purposes in Yavatmal. The anthropological activities are the main source of water pollution in Yavatmal. In order to know water quality, this research is being carried out.

MATERIALS AND METHODS

General description of study area

Yavatmal is situated in Maharashtra, India. Its geographical coordinates are 20.11700° North, 78.11080° East. As of 2011 India census, Yavatmal has a population of 116,551. Males are 58,549 of the population and females are 58,002. Yavatmal has an average literacy rate of 82.82%. Yavatmal

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titrated against standardized EDTA solution until the color changes from wine red to sky blue which indicated the end point. The final reading of the burette was noted and the titration was repeated to get constant value. Finally using the analytical calculation, total hardness of water sample was determined in terms of mg/L of CaCO_3 .

Dissolved Oxygen

Dissolved Oxygen is one of the important parameter of water quality which depict physical and biological process occurring in water. When temperature increases gas solubility of water decreases and microbial activity increases; both these changes can reduce DO in water. DO shows significant difference at different study samples. The level of the oxygen in water (3 to 5 mg/l) is an indicator of healthy state of water and values below 3 mg/l are hazardous to human.

Table-1: Comparison of chemical parameters of water sampling sites

Parameters	Sampling sites			BIS standards		WHO Standard
	Yavatmal	Nilona Dam	Dhumnapur	Acceptable limit	Maximum limit	
pH	7.2	7.1	7.2	6.5-8.5	6.5-9.2	6.5-8.5
PA	00	00	00	—	—	—
TA	195	205	205	200	600	—
DO	5.0	4.8	4.9	—	—	—
TDS	503	480	510	500	2000	500
To. Hardness	80.0	95.0	110.0	—	—	—
Cl-	130.11±3.22	132.48±5.23	547.32±7.05	200	1000	250

RESULTS AND DISCUSSION

In order to explore drinking water quality, Physico-chemical study of Drinking water in different areas of Yavatmal, experiments were conducted and observed with respect to the analysis of Physico-chemical parameters. The physico-chemical data of the drinking water sample collected in January 2016-2017. The results of the samples vary with different collecting places because of the different contaminants. In the present study temperature ranges from 27.00C to 33.00C. PH: In the present study pH ranged from 7.1 to 7.2. The tolerance pH limit is 6.5 – 8.5. TDS: in the present study TDS ranged from 480-510 mg/L. According to WHO and Indian standards, TDS value should be less than 500 mg/L for drinking water. D.O. : The D.O. range from 4.8 to 5 mg/L in present samples. The minimum tolerance range is 4.0 mg/L for drinking water. In the present study chloride ranged from 130-548 mg/L. While the tolerance range for chloride is 200 – 1000 mg/L. Total Alkalinity: The total alkalinity content in the samples is in between 160 to 748 mg/L. The chloride concentration indicates the sewage pollution. Atmospheric sources or sea water contamination is reason of the chloride concentration in groundwater. Soil Porosity also plays a key role to increase chlorides concentration. The chloride content of water samples analyzed was within permissible limit of 250 mg/L. In study, the results of chlorides in all sampling sites ranged from 135.22 to 554.46 mg/L.

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CONTENTS

Teacher Education in India: Innovative Strategies to Strengthen a Critical Analysis of NCFTE-2009 <i>Dr. Narbada Devi</i>	1
Teacher Education: Present Scenario and Future Challenges <i>Dr. Narbada Devi</i>	7
The Importance of Art in a Child's Development <i>Nalin Chandra</i>	14
The Effect of Demographic Factors on the Stress and Social Support Related to the Executive Organizational Commitment-Nagpur Region <i>Dr. Anand G. Naranje</i>	22
Studies on Adaptation of NIAS-MaBr-92, a Cell line From Hemocytes of Cabbage Armyworm, <i>Mamestra brassicae</i> to MGM-448 Culture Medium Supplemented with 3% Fetal Bovine Serum <i>Syed Obaid Qureshi</i>	31
Development of Pisciculture in Bardia Reservoir of District Pratapgarh, Rajasthan with Special Reference to Limnochemical Parameters <i>Sourindra Mohan Roy</i>	37
Internet Addiction in Juveniles: Measuring the Validity of Internet Addiction Scale and Estimating the Level of Internet Addiction and Anti-Social Internet Behavior <i>Anu Singla and Saba Rashid</i>	46
Teacher Education in Post- Independence India <i>Dr. Kamini Rajput</i>	54
Green Corporate Image Through Green Marketing and Sustainable Development <i>Shadma Parween</i>	59
Human Rights of Women in Islam : An Analysis <i>Dr. Nooreen Zaidi</i>	63
Marxist Movement in India: A Theoretical-Empirical Analysis <i>G. N. Trivedi</i>	72

Studies on Adaptation of NIAS-MaBr-92, a Cell line From Hemocytes of Cabbage Armyworm, *Mamestra Brassicae* to MGM-448 Culture Medium Supplemented with 3% Fetal Bovine Serum

Syed Obaid Qureshi*

ABSTRACT

Serum-dependence of insect cell lines is a factor that escalates the cost of maintaining of a cell line. Vertebrate sera are heterogenous in composition, lack reliability and pose an inherent potential risk for transmission of infectious agents. Reducing the serum-dependence of a cell line can allow researchers to use it for production of proteins of interest. During present study NIAS-MaBr-92 cell line was successfully adapted from MGM-448 growth medium supplemented with 10% FBS to MGM-448 growth medium supplemented with 3% FBS. Adaptation was carried out by decreasing the volume of original medium (MGM-448 supplemented with 10% FBS) by 20% and increasing the volume of MGM-448 medium supplemented with 3% FBS by 20% at each passage. The initial cell density, final cell density, number of days taken to attain final cell density and population doubling time were noted at each passage. Cell population of NIAS-MaBr-92 was heterogenous, comprising of five different types of cells viz. large round, small round, spindle-shaped, giant and tadpole-shaped cells. Large round cells were the most common type. The cell line took about 40 days for adaptation. During early steps of adaptation, the cells showed reduction in final cell density. The growth of cells during middle steps was slowest. These steps also returned highest population doubling time values with a peak value of about 76 hours. By last step the population doubling time was reduced to about 67 hours. Continuous passaging of cells in MGM-448 with 3% FBS reduced the population doubling time to about 65 hours.

Keywords: NIAS-MaBr-92; insect cell line; adaptation; population doubling time.

INTRODUCTION

Trager (1935) successfully demonstrated that mixture of inorganic salts, maltose, digested egg albumin and *B. mori* hemolymph can be used for maintaining ovarian tissues of *B. mori*. This paved way for a series of experiments to formulate optimum growth media for long-term culture of insect cells. Mitsuhashi (1982, 1984) successfully modified Grace's medium several times for cultivation of different cell lines. Supplementation with vertebrate sera like fetal bovine serum (FBS) or other proteinaceous materials proved to yield high growth rate (Weiss and Vaughn, 1986). Over years, many serum-free media have been developed (Hink, 1981; Donaldson and Shuler, 1998). It is possible to achieve high growth rate by increasing total Pluronic F-68 concentration to 0.3% with IPL41 serum-free medium (Caron et al., 1990). Lepidopteran cell lines offer a cost-effective

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and size. The cell size was determined by ocular micrometer taking about 50-100 readings of each cell type. The relative percentage of each type in population was determined by counting different cell types in optical fields under the inverted microscope.

ADAPTATION TO MGM-448 MEDIUM SUPPLEMENTED WITH 3% FBS

The cell line was adapted to MGM-448 supplemented with 3% FBS to reduce its serum dependence as described by Mitsuhashi and Grace (1969) through passaging of the cells. This was done by decreasing the volume of original medium (MGM-448 supplemented with 10% FBS) by 20% and increasing the volume of MGM-448 medium supplemented with 3% FBS by 20% at each passage (steps "a" through "e"). The initial cell density, final cell density, number of days taken to attain final cell density and population doubling time were noted at each passage.

POPULATION DOUBLING TIME

A small quantity of cell suspension was taken in Pasteur pipette from culture flasks at the time of seeding the culture. The cell suspension was allowed to flow in the chambers of Neubauer hemocytometer chamber and cells were counted in four large corner squares each of which is divided into 16 small squares. The volume of one large square is 0.1 mm^3 . Therefore, average cell count was multiplied by 10^4 to give number of cells per ml. The cell number was determined as an average of readings from two culture flasks. The population doubling time (PDT) was calculated using the exponential formula given by Hayflick (1973).

$$r = 3.32 (\log x_2/x_1)/(t_2-t_1)$$

where,

r = Multiplication rate

x_1 = Initial cell number at selected time t_1

x_2 = Final cell number at selected time t_2

Generation time (g) = $1/r$

The procedure was repeated at the time of attainment of final cell density.

RESULTS AND DISCUSSION

Cells of NIAS-MaBr-92 grew freely suspended. Cell clumping reported in studies on *Bombyx mori* ovarian cells (Bahekar and Qureshi, 2013) was not observed during present study. Cell population of NIAS-MaBr-92 was heterogeneous, comprising of five different types of cells viz. large round, small round, giant, spindle-shaped and tadpole-shaped cells.

- (i) Large round cells were having a diameter of $40 \pm 1.06 \mu\text{m}$. About 40% cells were of this type.
 - (ii) Small round cells measured $25 \pm 2.13 \mu\text{m}$ and constituted 37% of total cell population.
 - (iii) Giant cells: These cells measured about $54 \pm 2.77 \mu\text{m}$ in size. They formed about 12% of the cell population.
 - (iv) Spindle-shaped cells: They formed about 8% of total cell population. They measured $48.42 \pm 1.18 \mu\text{m}$ in length and $17.95 \pm 2.07 \mu\text{m}$ in width.
 - (v) Tadpole shaped cells: These were the least common of all cell types accounting for about 3% cells. They measured about $77.96 \pm 3.87 \mu\text{m}$ in length and $31.21 \pm 1.07 \mu\text{m}$ in width.
- In MGM-448 medium with 10% FBS, cells of NIAS-MaBr-92 grew very slowly for 4 days after subculturing. Thereafter the cells entered exponential growth phase to reach a cell density

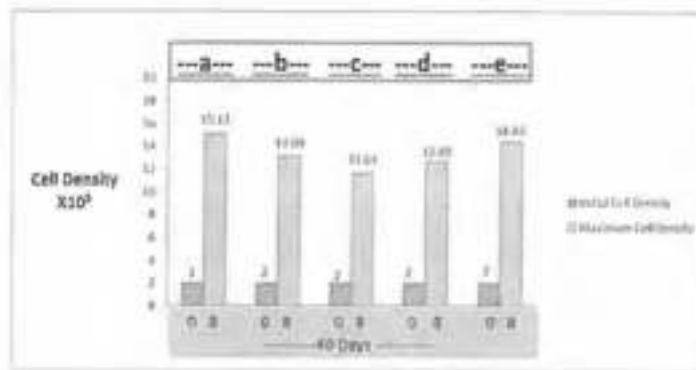


Fig.1. Graph showing adaptation of NIAS-MaBr-92 cell line from MGM-448 medium with 10% FBS (A) to MGM-448 medium with 3% FBS (B)- a-80% A+ 20% B; b-60% A+ 40% B;c- 40% A+ 60% B; d 20% A+ 80% B; e- 100%B

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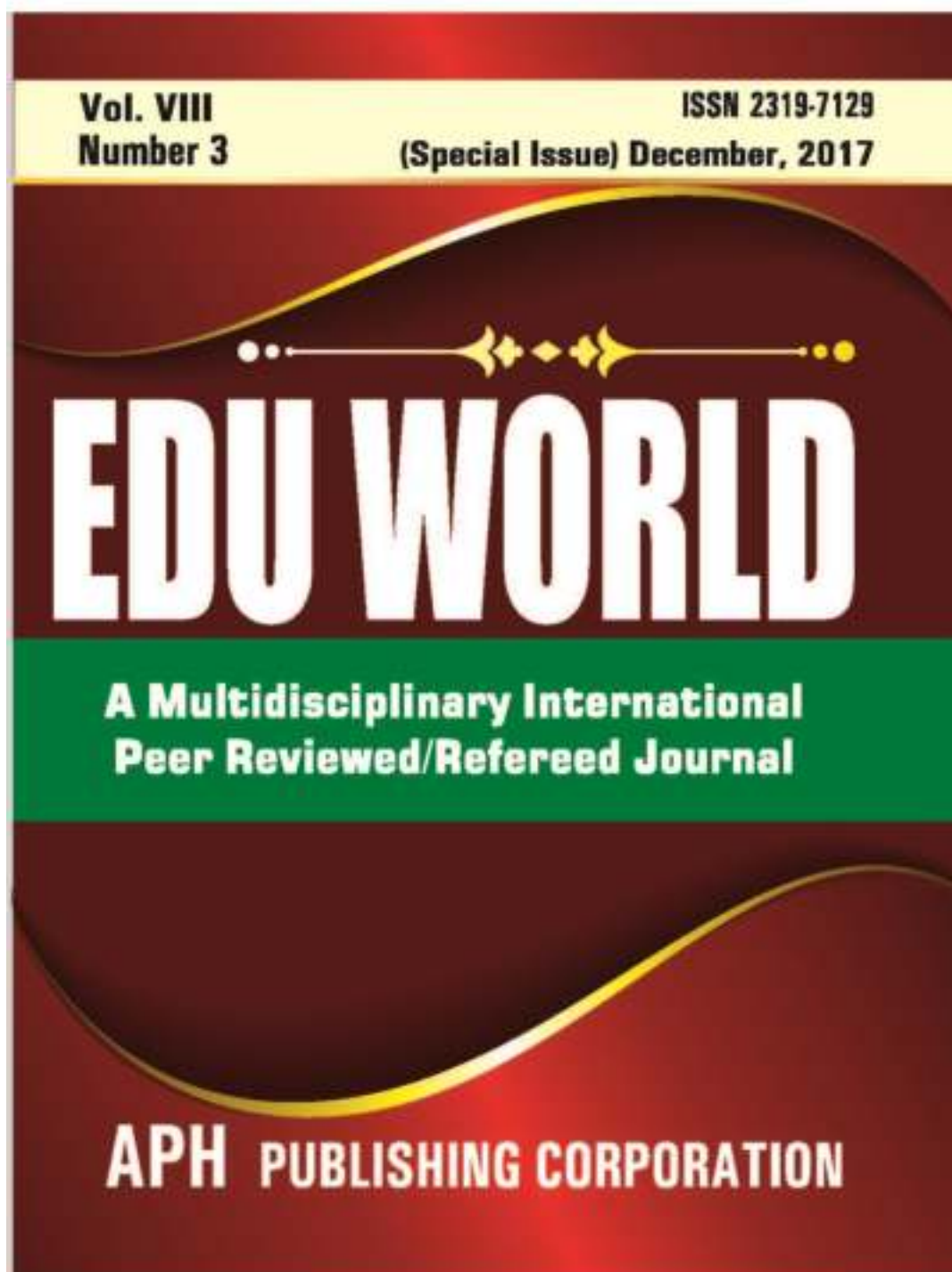
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88

The Effect of Demographic Factors on the Stress and Social Support Related to the Executive Organizational Commitment-Nagpur Region : Anand Naranje



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CONTENTS

Teacher Education in India: Innovative Strategies to Strengthen a Critical Analysis of NCFTE-2009 Dr. Narbada Devi	1
Teacher Education: Present Scenario and Future Challenges Dr. Narbada Devi	7
The Importance of Art in a Child's Development Nalin Chandra	14
The Effect of Demographic Factors on the Stress and Social Support Related to the Executive Organizational Commitment-Nagpur Region Dr. Anand G. Naranje	22
Studies on Adaptation of NIAS-MaBr-92, a Cell line From Hemocytes of Cabbage Armyworm, <i>Mamestra brassicae</i> to MGM-448 Culture Medium Supplemented with 3% Fetal Bovine Serum Syed Obaid Qureshi	31
Development of Pisciculture in Bardia Reservoir of District Pratapgarh, Rajasthan with Special Reference to Limnochemical Parameters Sourindra Mohan Roy	37
Internet Addiction in Juveniles: Measuring the Validity of Internet Addiction Scale and Estimating the Level of Internet Addiction and Anti-Social Internet Behavior Anu Singla and Saba Rashid	46
Teacher Education in Post- Independence India Dr. Kamini Rajput	54
Green Corporate Image Through Green Marketing and Sustainable Development Shadma Parween	59
Human Rights of Women in Islam : An Analysis Dr. Nooreen Zaidi	63
Maoist Movement in India: A Theoretical-Empirical Analysis G. N. Trivedi	72

The Effect of Demographic Factors on the Stress and Social Support Related to the Executive Organizational Commitment-Nagpur Region

Dr. Anand G. Naranje*

ABSTRACT

In today's competitive environment life is infinitely more complex than it has ever been. The present era of globalization has brought forth many issues related to the development and social balance of human society. Amongst the numerous factors it has been studied and found, organizational setup is primarily to improve the level of equality approach in productivity. Organizational and its environmental set up depend on higher-level employees who are mostly involved in the decision making for the development of an organization. Competition in the workplace, the stress that arises from it and the negative environment that results from it, endangers the mental health of employees. In this situation, stress and social support are essential factors affecting work, commitment and performance at organizations. This paper is an attempt to study the impact of stress and social support on the level of commitment of managers. A sample of 590 managers of different levels was taken. Results show the negative and significant relationship between job stress and organizational commitment also it shows that stress is a major cause that decreases the employee's commitment towards the organization.

Keywords: Organizational Commitment, Stress, social support

INTRODUCTION

The present study based on the management approach which is concerned with the social, administration, psychological and human resources aspects of the development process. This study is mainly framed based on different managerial levels of employees from the government and private sectors. The working conditions at the organization influences the people during the initial period of employment, which leads to the development of expectations of the employees in terms of the support they receive from the organization and which along with the satisfaction of employees in terms of financial gains, settlement of life, increasing standard of living, etc. meanwhile, the involvement of employees in their own home with domestic work, child responsibilities, spouse responsibility and even dependent persons of family and community, etc. which may create obstacles in the employees work-life at the organization. Therefore the researchers found it interesting to study stress and social support and their relationship to organizational commitment.

It has been biologically and psychologically proving that the stress of the employees is also an antecedent of commitment. It has been studied that stress is deeply rooted in the workplace environment, the structure of the job, support extended by the organization, home environment, and to some extent by community factors.

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30 Dr. Anand G. Naranje

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Complex analysis of classified of Soil parameters and its relationship identification using PCA : Dr. Maya V. Mawale

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Complex analysis of classified of Soil parameters and its relationship identification using PCA

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Abstract— This study was carried out to predict meaningful information from large data set of soil parameters and representation in graphical manner to make its clear understanding. This analysis help in determining role of dependent variable and independent variable in the system and their relationships, their dependability for designing any prediction system. A field study is carried out to collect information for assessing soil parameter. Soil parameters analysis is done on 902 soil samples collected from Krushi Vigyan Kendra, Ghatkesh, Amravati. The values of C, N, P, K, Mg, Ca, Fe, Cu, Zn, B, Mn, Lime, Saline, CEC, Sds, OM and pH of soil sample collected for the year 2011-2012 and 2012-2013 and Principle Component Analysis (PCA) is used to predict these soil parameters as a dependent and independent parameter that have direct/indirect effects on productivity.

Keywords— complex analysis, soil parameter, Principle Component Analysis, Cu, copper, Fe, iron, K, potassium, Mn, manganese, OC, organic content, P, phosphorus, Zn, zinc.

I. INTRODUCTION

Real world problem needs lots of data and information to handle situation so its size and scale are larger and testing is done on such a large scale is quite difficult. Complex analysis is used to predicate meaningful information from large data and representation of that data in graphical manner. Plant growth is the result of a complex process. Many biological, chemical and physical factors are responsible for determining soil quality [1]. As soil having different texture for different area, the soil properties of soil are in variation and its operation at a range of scales, show intermittent effects, and fluctuating more income regions than in others and prediction of such soil properties become complex [2]. With the help of complex analysis larger data and information is converted into meaningful information. More number of quality variables are good sign for better accuracy in data analysis as each variable shows its significance individually and relationship between each other having same properties. Complex analysis using Principle Component Analysis (PCA) is used to predicate meaningful information from large data set of soil parameters and representation in graphical manner to make its clear understanding [3]. This analysis help in determining role of dependent variable and independent variable in the system and their relationships, their dependability for designing any prediction system [4].

COMPLEX ANALYSIS

During soil collection, the process of soil testing in laboratory is well studied. Testing of each macronutrient, micronutrient and other factor is carried out. As result of laboratory analysis, values of 902 soil sample are taken. To assess the variables relevant for the decision making the framework of complex analysis is used to predicate meaningful information from large data and representation of that data in graphical manner. Soil parameters are identified such that direct variable considered for fuzzy model and indirect variables are used for making platform for direct or relevant variables. Choosing of relevant variables is most crucial process in determining precision decision. Ec, N, P, K, Mg, Ca, Fe, Cu, Zn, pH are dependent variables which are applied directly in fuzzy model and Saline, Organic matter, CEC and Lime are independent variables [5].

The PCA technique of XLSTAT is used to analyses relationship between various variables by Correlation matrix (Pearson (r)), Eigenvalues, factor loading contribution and square cosines of variable and observations. It also plot different chart like score plot, Bi-plot etc. that helps in analyzing the data and draw some valid inference.

Use of PCA in the research

- i. Analysis of independent variable of a data table representing observations described by several dependent variables and their inter-correlation.
- ii. Its goal is to extract the important information from the data table and to express this information as a set of new orthogonal variables called principal components.
- iii. PCA also represents the pattern of similarity of the observations and the variables by displaying them as points in maps.
- iv. Principal components are obtained as linear combinations of the original variables. The first principal component is required to have the largest possible variance. The second component is computed under the constraint of being orthogonal to the first component and to have the largest possible inertia.
- v. The other components are computed likewise. The values of these new variables are factor scores and these factors scores are interpreted geometrically as the projections of the observations onto the principal components.
- vi. Figure No. 1 Interlinking between nutrients and analyzing their dependability

- dimensionally. If you must use mixed units, clearly state the units for each quantity that you use in an equation.
- Do not mix complete spellings and abbreviations of units: -Wb/m² or -webers per
- (Source: Compiled by Researcher)
- Soil parameters are classified into number of set. Each set consist of number of parameter according their dependability, correlation properties. Maximum number of set is prepared with the help of combination of variables.

Table No. 1 Classification of variables

Dependent Variables	Independent Variables
pH, EC, N, P, K, Mg, Zn, Cu, Fe, Mn, B, Mo, C	Lime, OM, Saline, CEC
No. of variables=13	No. of variables=4

(Source: Compiled by Researcher)

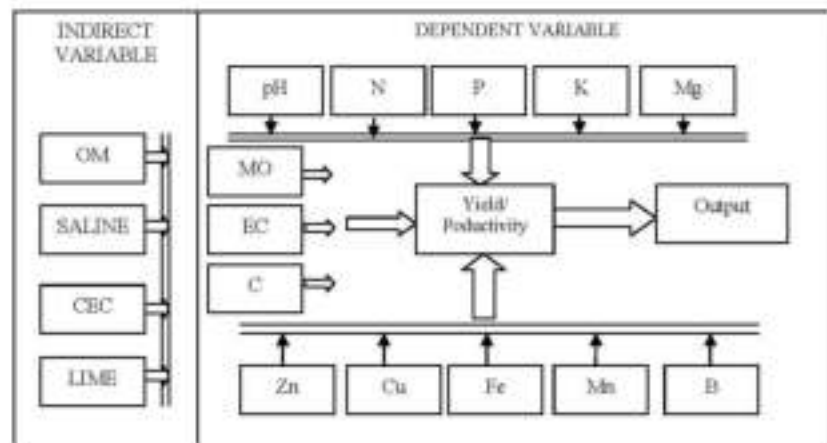


Table No. 2 Preferred combinations used for designing the system

Indirect Variables			Direct Variables			Mix variables		
Cu	Variables	No. of Rule	Cdi	Variables	No. of Rule	Cmi	Variables	No. of Rule
Ci1	{OM}	3	Cd1	{p}	06	Cm1	{Zn,OM}	2
Ci2	{OM,CBC}	1	Cd2	{Mn}	01	Cm2	{pH,CBC}	1
Ci3	{Lime}	1	Cd3	{K}	06	Cm3	{Mg,CBC}	1
			Cd4	{EC}	06	Cm4	{Lime,B}	1
			Cd4	{Zn}	01	Cm5	{K,Lime}	1
			Cd5	{N}	02	Cm6	{OM,B}	1
			Cd5	{Cu}	01	Cm7	{pH,OM}	4
			Cd6	{B}	05	Cm8	{C,Lime}	1
			Cd7	{C}	01	Cm9	{P,OM}	1
			Cd7	{pH}	04	Cm10	{Cu,Lime}	1
			Cd8	{MO}	01	Cm11	{pH,OM,CBC}	1
			Cd9	{K,Mn}	01	Cm12	{K,Cu,Lime}	1
			Cd10	{K,Zn}	01	Cm13	{pH,N,P,K,OM,CBC}	1
			Cd11	{MO,B}	01	Cm14	{Cu,Zn, Mg, Saline, MO}	1
			Cd12	{pH,K}	33	Cm15	{pH,K,Mg,CBC}	1
			Cd13	{pH,N}	33	Cm16	{pH,saline,OM,Lime}	17
			Cd14	{pH,P}	33	Cm17	{Cu, Zn, Mg, Saline, MO}	1
			Cd15	{EC,N}	06	Cm18	{pH, N, K,OM, CBC}	1
			Cd16	{Cu,MO}	01	Cm19		
			Cd17	{pH,B}	01			
			Cd18	{N,B}	01			
			Cd19	{pH,Cu}	03			
			Cd20	{N,P,Zn}	01			
			Cd21	{P,Cu,MO}	01			
			Cd22	{pH,Mn,MO}	01			
			Cd23	{pH,K,Fe,Mn,Zn}	02			
			Cd24	{Cu,Fe,Mn}	01			
			Cd25	{Cu,Zn,MO}	01			
			Cd27	{pH,Cu,P,Cu,Fe,Mn,Mo,Zn}	01			
			Cd28	{pH,Fe,N,P,K,C}	02			

(Source: Compiled by Researcher)

Table No 3: Correlation coefficient of soil parameter

	Lime	Saline	OM	CBC	pH	EC	N	P	K	C	Cu	Fe	MN	Zn	Mg	B	MO
Lime	1.00																
Saline	0.29	1.00															
OM	0.33	0.56	1.00														

CEC	-0.55	-0.05	0.00	1.00														
pH	0.24	-0.02	0.04	0.32	1.00													
EC	-0.36	-0.21	-0.32	-0.40	-0.84	1.00												
N	0.38	-0.00	-0.12	0.16	0.31	-0.50	1.00											
P	0.14	0.00	0.10	0.30	0.85	-0.75	0.30	1.00										
K	0.15	-0.09	-0.50	-0.05	-0.04	0.05	-0.72	0.00	1.00									
C	0.35	-0.13	-0.19	0.22	0.58	-0.64	0.96	0.48	0.63	1.00								
Cu	0.45	0.52	0.04	0.08	0.49	-0.58	0.62	0.10	0.38	0.61	1.00							
Fe	0.44	0.36	0.47	0.35	0.58	-0.74	0.45	0.27	-0.02	0.45	0.75	1.00						
MN	0.39	0.21	0.24	-0.38	0.17	-0.22	0.09	0.30	-0.20	0.16	0.08	-0.04	1.00					
Zn	0.24	0.10	0.09	0.23	0.13	-0.40	0.88	0.17	0.66	0.78	0.51	0.43	0.00	1.00				
Mg	-0.22	-0.42	-0.40	0.02	-0.14	0.34	0.01	0.20	0.31	-0.05	-0.52	-0.52	-0.44	0.05	1.00			
B	-0.40	-0.52	-0.70	-0.03	-0.13	0.51	0.10	-0.10	0.36	-0.09	-0.57	-0.33	-0.22	-0.07	0.68	1.00		
MO	0.05	0.20	0.66	0.37	0.07	-0.47	0.27	0.11	-0.24	0.21	0.28	0.51	0.08	0.37	-0.30	-0.79	1.00	

(Source: Compiled by Researcher)

Table No 4: Correlation coefficient of soil parameter

	Line	Saline	OM	CEC	pH	EC	N	P	K	C	Cu	Fe	MN	Zn	Mg	B	MO
Line	23.55																
Saline	0.04	0.00															
OM	0.01	0.00	0.00														
CEC	-0.65	0.00	0.00	0.06													
pH	0.34	0.00	0.00	0.02	0.08												
EC	-0.12	0.00	0.00	-0.01	-0.02	0.00											
N	86.59	-0.07	-0.04	1.86	4.11	-1.64	2208.75										
P	17.06	0.00	0.02	1.80	5.84	-1.29	349.13	603.01									
K	9.16	-0.07	-0.08	-0.31	-0.27	0.05	892.80	-1.07	705.00								
C	0.10	0.00	0.00	0.00	0.01	0.00	2.67	0.69	0.99	0.00							
Cu	3.24	0.01	0.00	0.03	0.21	-0.05	43.31	5.69	15.04	0.05	2.18						
Fe	4.70	0.02	0.01	0.19	0.38	-0.11	46.35	14.35	-1.15	0.06	2.43	4.85					
MN	15.46	0.06	0.01	-0.89	0.46	-0.15	41.25	46.96	-51.03	0.09	1.17	0.89	94.46				
Zn	1.67	0.00	0.00	0.07	0.05	-0.04	58.69	4.08	25.00	0.07	1.06	1.35	-0.03	2.01			
Mg	-0.05	0.00	0.00	0.00	0.00	0.00	0.05	0.22	0.37	0.00	-0.03	-0.05	-0.19	0.00	0.00		
B	-0.18	0.00	0.00	0.00	0.00	0.00	-0.37	-0.19	0.78	0.00	-0.04	-0.09	-0.18	-0.03	0.00	0.01	
MO	0.00	0.00	0.00	0.00	0.00	0.00	0.09	0.02	-0.04	0.00	0.00	0.01	0.01	0.00	0.00	0.00	0.00

Table No. 3 shows the correlation coefficient of the two soil parameters ranges. It determines the relationship between two Soil properties with the help of correlation coefficient equation. Table determines the degree to which two variable's movements are associated. It gives the strength and direction of a linear relationship between two variables. The table shows that all soil Parameter shows moderate uphill and positive linear relation and almost all parameter shows positive relationship except EC, Fe, and Cu as these soils parameter availability is very low.

Table No. 4 shows covariance, which is the average of the products of deviations for each data point of soil parameters pair and are used for determining the relationship between two data sets. From table it is observed that how much two soil variables change together. The variables tend to show similar behavior, the covariance is positive in the opposite case, when the greater values of one variable mainly correspond to the smaller values of the other, the covariance is negative. The sign of the covariance therefore shows the tendency in the linear relationship between the variables, thus it is concluded that all soil parameter has positive covariance.

Principal component analysis is used to find out relationship between various soil parameters and classified them into dependent and independent variables.

Distribution of soil characteristics in relation to the two first PCA axes are shown in Figure. The eigenvalues and the proportion of variance explained by the axes are listed in Table No. 5. The proportion of the variance is simply the eigenvalue for that axis divided by the total variance, i.e. the sum of the diagonal of the cross-products matrix. Graph No. 5.2 shows Soil characteristics, occupied different regions of the diagram. Table No. 5 also shows Pearson and Kendal correlation coefficients among soil variables, and between them and main axes of PCA.

Table No. 5 PCA Analysis (Min, Max, Mean, SD)

Variable	Observations	Obs. with missing data	Obs. without missing data	Minimum	Maximum	Mean	Std. deviation
Lime	15	0	15	3.73	14.01	9.62	5.02
Saline	15	0	15	0.65	0.75	0.72	0.03
OM	15	0	15	0.23	0.26	0.25	0.01
CBC	15	0	15	10.17	11.14	10.71	0.25
pH	15	0	15	7.60	8.75	7.73	0.29
EC	15	0	15	0.32	0.66	0.53	0.07
N	15	0	15	267.77	468.47	314.97	48.65
P	15	0	15	20.25	128.35	43.58	25.44
K	15	0	15	722.62	826.04	754.23	27.48
C	15	0	15	0.40	0.63	0.47	0.06
Cu	15	0	15	2.84	7.05	4.63	1.53
Fe	15	0	15	5.21	13.68	9.55	2.28

MN	15	0	15	19.12	59.91	30.05	10.06
Zn	15	0	15	0.91	6.78	1.80	1.47
Mg	15	0	15	21.08	21.31	21.20	0.05
B	15	0	15	0.03	0.36	0.06	0.08
MO	15	0	15	0.33	0.36	0.35	0.01

(Source: Compiled by Researcher)
Table No. 6
PCA Analysis (Correlation matrix (Pearson (n)))

Soil Parameter	Time	Saline	OM	CEC	pH	EC	N	P	K	C	Ca	Fe	MN	Zn	Mg	B	MO
Time	1.00	0.29	0.51	-0.15	0.24	-0.36	0.30	0.14	0.15	0.30	0.45	0.44	0.19	0.23	-0.22	-0.40	0.03
Saline	0.29	1.00	0.6	-0.05	-0.02	-0.21	-0.06	0.00	-0.07	0.13	0.32	0.30	0.21	0.10	-0.02	-0.32	0.30
OM	0.51	0.56	1.00	-0.004	0.04	-0.32	-0.12	0.10	-0.53	-0.10	0.04	0.47	0.24	0.09	-0.00	-0.70	0.66
CEC	-0.15	-0.07	0.00	1.00	0.31	-0.40	0.10	0.30	-0.05	0.22	0.00	0.35	-0.18	0.21	0.02	-0.09	0.37
pH	0.24	-0.02	0.04	0.31	1.00	-0.04	0.31	0.03	-0.04	0.50	0.40	0.50	0.17	0.13	-0.14	-0.33	0.07
EC	-0.36	-0.21	-0.32	-0.40	-0.04	1.00	-0.30	-0.73	0.03	-0.04	-0.38	-0.70	-0.22	-0.40	0.34	0.31	-0.07
N	0.30	-0.06	-0.12	0.10	0.31	-0.30	1.00	0.36	0.72	0.90	0.62	0.40	0.09	0.00	0.00	-0.30	0.27
P	0.14	0.00	0.10	0.30	0.03	-0.73	0.36	1.00	0.00	0.40	0.44	0.27	0.20	0.17	0.20	-0.30	0.11
K	0.15	-0.00	-0.50	-0.05	-0.04	0.03	0.72	0.00	1.00	0.63	0.70	-0.01	-0.20	0.00	0.30	-0.30	-0.24
C	0.30	-0.13	-0.10	0.22	0.50	-0.04	0.90	0.40	0.63	1.00	0.61	0.40	0.16	0.70	-0.00	-0.00	0.21
Ca	0.45	0.32	0.04	0.00	0.40	-0.38	0.62	0.10	0.70	0.60	1.00	0.71	0.00	0.31	-0.52	-0.37	0.10
Fe	0.44	0.30	0.47	0.35	0.50	-0.74	0.45	0.27	-0.02	0.40	0.72	1.00	0.04	0.45	-0.52	-0.33	0.31
MN	0.19	0.21	0.24	-0.18	0.17	-0.22	0.09	0.10	-0.20	0.10	0.00	0.04	1.00	0.00	-0.44	-0.22	0.00
Zn	0.23	0.10	0.00	0.21	0.13	-0.40	0.00	0.17	0.60	0.70	0.31	0.42	0.00	1.00	0.00	-0.07	0.37
Mg	-0.22	-0.02	-0.00	0.02	-0.14	0.34	0.01	0.20	0.31	0.00	-0.32	-0.51	-0.44	0.00	1.00	0.00	-0.50
B	-0.40	-0.32	-0.70	-0.03	-0.13	0.31	-0.30	-0.10	0.30	-0.00	-0.37	-0.53	-0.22	-0.07	0.00	1.00	-0.70
MO	0.03	0.30	0.66	0.37	0.07	-0.07	0.27	0.11	-0.24	0.20	0.20	0.31	0.00	0.37	-0.50	-0.70	1.00

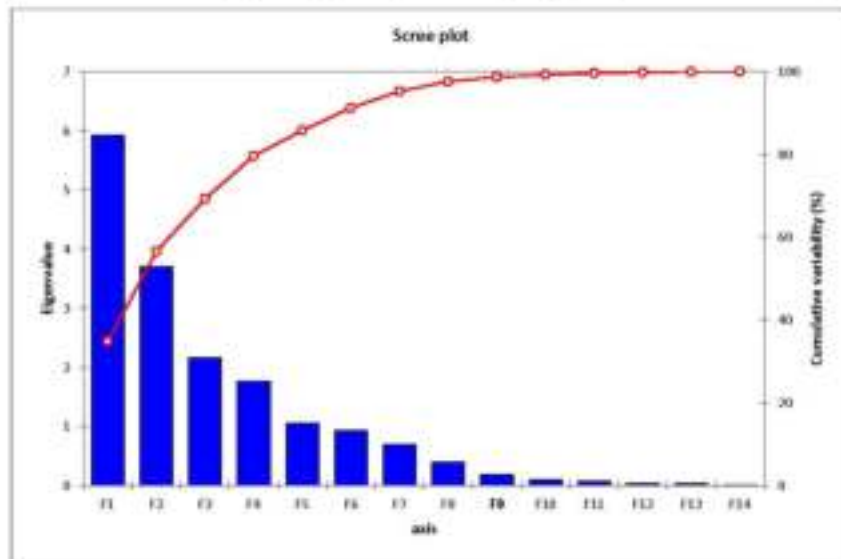
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Table No. 7 PCA Analysis (Eigenvalues)

Function Count	Eigenvalue	Variability (%)	Cumulative %
F1	5.91	34.79	34.79
F2	3.7	21.74	56.53
F3	2.16	12.71	69.24
F4	1.76	10.35	79.59
F5	1.05	6.16	85.75
F6	0.92	5.42	91.17
F7	0.69	4.08	95.25
F8	0.39	2.32	97.57
F9	0.18	1.06	98.63
F10	0.1	0.56	99.19
F11	0.07	0.41	99.6
F12	0.03	0.19	99.79
F13	0.03	0.16	99.95
F14	0.01	0.05	100

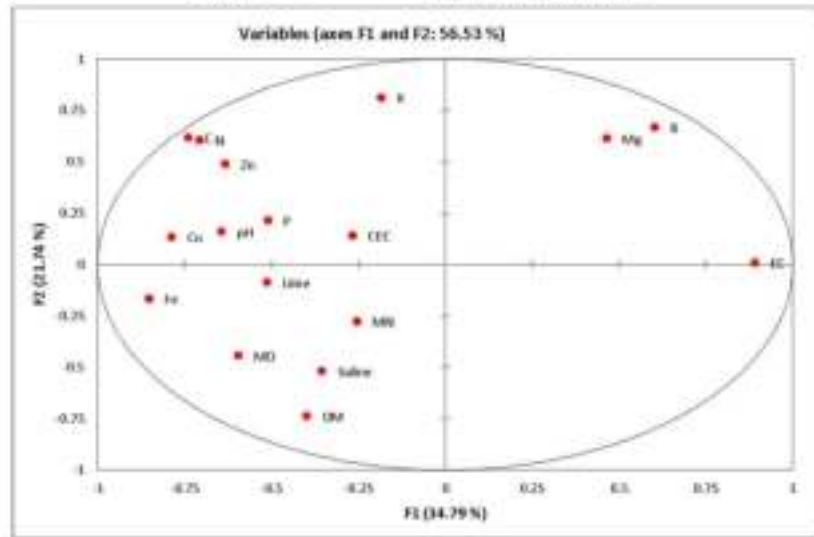
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Graph No. 1 Scree plots of eigenvalue, its variability among them, and cumulative variability in%



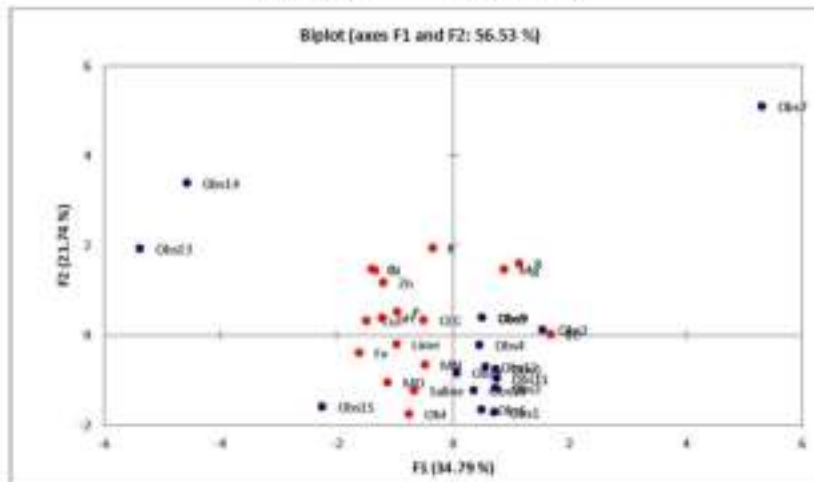
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Graph No. 2 Variables distribution space



(Source: Compiled by Researcher)

Graph No. 3 Biplot of PCA



(Source: Compiled by Researcher)

Principle Component Analysis of data which include mean, standard deviation, min-max values and data does not contain any missing value in total number of observation. Correlation coefficient among all soil parameter is calculated and it shows their positive uphill relationship among two parameters.

The eigenvalue table from the current analysis shows the eigenvalues for components 1 to 5 are 5.914, 3.695, 2.161, 1.760, and 1.047, respectively. Only these components demonstrated eigenvalues greater than 1.00, so the eigenvalue-one criterion would lead to retain and interpret only these components.

The first Principle Component (PC) accounts for the most variance (and hence have the highest eigenvalue), and the next component account for as much of the left over variance, and so on. Hence, each successive component will account for less and less variance.

- This variability in the percent of variance accounted for by each principal component show decreasing pattern for each pc.
- Cumulative % - This cumulative percentage of variance accounted for by the current and all preceding principal components. From Table No 5.11 observed that the first six components together account for 91.169% of the total variance. Scree plot is plotted with these three items. Biot shows that first two component F1, F2 occupies 56.53% of total variance.

Result

- PCA shows positive uphill for coefficient of correlation for soil parameters using independent and dependent variables representing orthogonal space in the scree graph. Scree plot is plotted with Eigen value, variability and cumulative variability for variables. Cumulative percentage of variance accounted for by principal components observes that the first six components together account for 91.169% of the total variance. Biplot shows that first two component F1, F2 occupies 56.53% of total variance.
- The Eigen value shows for components 1 to 5 are 5.914, 3.695, 2.161, 1.760, and 1.047, respectively. Only these components demonstrated Eigen values greater than 1.00, so the Eigen values lead to retain and interpret only these components. The variability is accounted for by each principal component show decreasing pattern for each principal component.

Conclusion

As soil is complex system, its behavior is examined by making complex analysis using PCA because soil consists of number of components, number of micronutrient, micronutrient, related to each other and specifies the dependent variable and independent variable for making prediction system for productivity. This analysis is one of the way for judging yield production like regression analysis, simulation time series and neural network.

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Study on photoluminescence and thermoluminescence properties of UV-irradiated $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphors: Shweta S. Sharma

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Study on photoluminescence and thermoluminescence properties of UV-irradiated $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphors

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Abstract This paper reports synthesis of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphors via traditional high temperature solid state reaction method. The phase analysis of the phosphors was carried out using powder X-ray diffraction (XRD) technique which indicates that all these compounds are of single phase of $\text{CaSrAl}_2\text{SiO}_7$ with tetragonal crystal structure. Average grain size of the phosphor was determined by using Scherrer formula. The photoluminescence (PL) study was carried out using spectrofluorophotometer. PL Excitation (PLE) spectra consist of two peaks at around 253 and 290 nm. $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ emits strong violet emission located at 418 nm when excited with 290 nm. Emission is associated with $5\text{D} \rightarrow 4\text{F}$ transition of Ce^{3+} ions. Thermoluminescence (TL) properties of UV-irradiated phosphor were measured. Effect of different heating rates on TL glow curve was recorded and 5 °C/s is found to be optimum heating rate. There is no shift in glow peak position with increase in UV-irradiation time. TL intensity increases with increase in UV exposure time up to 35 min and then saturates. To analyse kinetic study, glow curve was deconvoluted into four different traps and their activation energies were determined by using peak shape method. Concentration dependence on PL and

TL was investigated; concentration quenching in PL occurs at 1 mol% of Ce^{3+} and quenching in TL arises at 0.5 mol% of Ce^{3+} . TL spectra and decay characteristic were also carried out. This paper deals with possible mechanism of PL and TL.

1 Introduction

Phosphors are the luminescent materials that exhibit emission of light (usually in ultraviolet, visible and infrared region of electromagnetic spectrum) by the conversion of any kind of stimulus [1]. Unlike other luminescent materials, persistent phosphors can continuously emit light for minutes or hours. Such types of materials are applicable in safety signage, dials, decoration and display [2]. ZnS-based materials are first commercially persistent phosphors. These materials absorb moisture from surrounding environment, exhibit formation of sulfate and destruction of sulfide structure. Hence they do not have better afterglow property. To supersede demerits of the sulfides invention is made towards the aluminate based phosphors. In the past few decades rare earth doped aluminates like $\text{SrAl}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} [3], $\text{BaAl}_2\text{O}_7:\text{Eu}^{2+}$, Dy^{3+} [4], $\text{CaAl}_2\text{O}_7:\text{Eu}^{2+}$, Nd^{3+} [5] were invented as efficient long persistent phosphors. Aluminates show afterglow for long duration as compared to sulfides however their properties are very much decreased under the influence of water, besides they have weak chemical stability, which limits their future applications [6]. Consequently silicate based phosphors have attracted much attention. Silicate and aluminosilicate based phosphors show excellent water resistance property, high quantum efficiency, long persistence, colour purity, rigid crystal structure, high chemical and thermal stability, multicolour emission [7, 8].

Rare earth ions are commonly used as the dopant ions in different host lattice due to their high fluorescence

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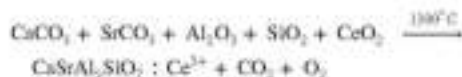
efficiencies and very narrow line fluorescence band. Rare earth ions are useful in modern lighting and display fields because of various optical emission based on their 4f–4f or 5d–4f transitions. Their emission can be tuned by selecting a suitable host [9, 10]. Ce^{3+} activated materials have attracted considerable interest in accordance with their potential as scintillator and tunable laser materials. Ce^{3+} ions consist of simple one-electron system having electronic configurations of their ground and excited states are $4f^1$ and $5d^1$, respectively. Ce^{3+} doped materials have broad emission band in ultraviolet (UV) and visible ranges [11]. Over the last two decades the attention is mainly concentrated on the preparation and optical studies of melilite type materials. Melilite group consists of two members Akermanite ($\text{A}_2\text{MgSi}_2\text{O}_7$) and Gehlenite ($\text{A}_2\text{Al}_2\text{SiO}_7$) here $\text{A} = \text{Ca}$ or Sr . They characterize in tetragonal phase with space group $\text{P4}_2/\text{m}$ [12]. Kodama et al. [13] have investigated long lasting phosphorescence produced by UV excitation in Eu^{3+} -doped melilites like $\text{Ca}_2\text{Al}_2\text{SiO}_7\text{:Eu}^{3+}$, $\text{CaSrAl}_2\text{SiO}_7\text{:Eu}^{3+}$ and $\text{Sr}_2\text{Al}_2\text{SiO}_7\text{:Eu}^{3+}$. Recently rare earth activated $\text{Ca}_2\text{Al}_2\text{SiO}_7$ and $\text{Sr}_2\text{Al}_2\text{SiO}_7$ hosts pay particular attention in the research work. It is reported that $\text{Ca}_2\text{Al}_2\text{SiO}_7\text{:Ce}^{3+}$ [14, 15], $\text{Ca}_2\text{Al}_2\text{SiO}_7\text{:Dy}^{3+}$ [16, 17] phosphors are suitable candidate for UV or gamma dosimetry. Ding et al. have reported photoluminescence (PL) and thermoluminescence (TL) properties of Eu/Dy co-doped $\text{Sr}_2\text{Al}_2\text{SiO}_7$ phosphors. Co-doping of dysprosium enhance the long persistence [6].

Several research works have been made on $\text{Ca}_2\text{Al}_2\text{SiO}_7$ and $\text{Sr}_2\text{Al}_2\text{SiO}_7$ as hosts but only few works [7, 18, 19] done on $\text{CaSrAl}_2\text{SiO}_7$ host. In the present communication authors adopt solid state reaction method to synthesize $\text{CaSrAl}_2\text{SiO}_7$ phosphors with varying concentration of Cerium (Ce). These powders were characterized by X-ray diffraction technique and average grain size was determined by using Scherrer formula. Photoluminescence (PL) studies of phosphors were examined. To the best of our knowledge there is no report on thermoluminescence (TL) properties of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ phosphor. Systematic studies on TL of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ is investigated and reported for the first time.

2 Experimental

A series of powder samples with chemical formula $\text{Ca}_{1-x}\text{Sr}_x\text{Al}_2\text{SiO}_7\text{:xCe}^{3+}$ ($x = 0.1, 0.3, 0.5, 1, 2, 3$ and $4 \text{ mol\%}$) were successfully prepared via conventional solid state reaction method at a high temperature. The starting materials were calcium carbonate CaCO_3 , strontium carbonate SrCO_3 , silicon dioxide SiO_2 , aluminium oxide Al_2O_3 and cerium oxide CeO_2 (all are analytical grade). The stoichiometric amount of the raw materials was thoroughly mixed together and then grounded for 3 h using an agate mortar and pestle. The mixtures were then sintered

at 1300°C temperature for 5 h. The appropriate chemical reaction which is applied for stoichiometric calculation is:



The phase analysis of as-prepared phosphors was investigated by X-ray diffraction (XRD) analysis (model D8 Advance Bruker AXS) using $\text{CuK}\alpha$ radiation ($\lambda = 0.154 \text{ nm}$). The data were recorded over the 2θ range between 10° to 70° . The purity of all the samples was checked by matching their XRD patterns with JCPDS 26-0327. In order to confirm the introduction of Ce^{3+} ions into the powders, PL emission and excitation behaviour of phosphors were recorded at room temperature using a spectrofluorophotometer RF-5301PC assembled with 150W xenon lamp as an exciting light source; excitation and emission spectral bandwidth are well-set as 3 nm. TL study was carried out by using TLD reader, Nucleonix TL1009I with different heating rates. For the production of TL, samples were exposed to 254 nm of UV light. TL spectra were recorded using interference filters and decay characteristic was performed using home-made indigenous setup. Concentration-dependent PL and TL were examined to determine the optimum dopant concentration. All the studies were performed at room temperature.

3 Results and discussion

3.1 XRD characterization

X-ray diffraction technique is a convenient non-destructive tool for determination of crystal structure and average crystallite size of nano or bulk materials. Powder XRD pattern of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ (1 mol%) phosphor is shown in Fig. 1 attached with its JCPDS file. All the diffraction peaks of the synthesized phosphor were in good agreement with standard JCPDS file 26-0327. It is exhibited that no secondary phase is observed, results the sample has a single phase with melilite structure and Ce^{3+} ions have no significant influence on the crystal structure of $\text{CaSrAl}_2\text{SiO}_7$. Hence confirms that the introduction of Ce^{3+} ions does not create a new crystal phase, but it gently replace the cation sites of Ca^{2+} and Sr^{2+} ions in tetragonal crystal lattice with space group $\text{P4}_2/\text{m}$.

The average crystallite size of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ (1 mol%) phosphor was estimated from the broadening of (211) reflection by using Debye–Scherrer formula which is stated as:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

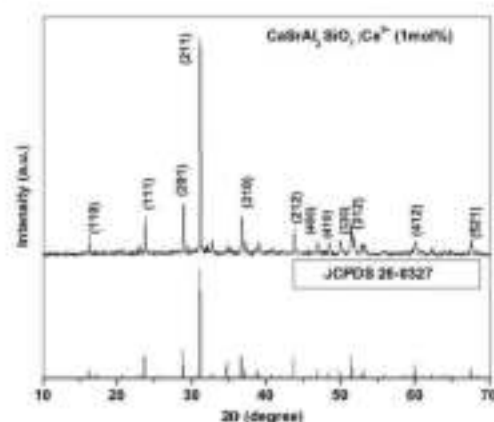


Fig. 1 XRD pattern of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (1 mol%) phosphor with JCPDS file

where D is the crystalline size for (hkl) plane, λ is the wavelength of incident X-ray which is 0.154 nm, β is peak width at half maximum, θ is the Bragg diffraction angle, and K is the Scherrer constant related to crystalline shape and usually taken as value 0.9. The value of β in 2θ axis of diffraction profile must be in radians. The θ can be in degrees or in radians, since the $\cos\theta$ correspond to the same number [20]. From the XRD shown in Fig. 1 it is cleared that (211) is the most intense plane among all the diffraction planes. For (211) plane $\theta = 15.584^\circ$, $\beta = 0.00279$ rad and calculated average crystallite size of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (1 mol%) phosphor is 51.57 nm.

3.2 Photoluminescence (PL) studies

In order to understand the behaviour of 5d electrons Ce^{3+} is a very good candidate among rare earth family. As compared to other trivalent rare earth ions excited energy state structure of Ce^{3+} is easily understood because it has single outer electron and only two spin orbital splitting 4f states, i.e. $^2F_{5/2,7/2}$. Ce^{3+} activated materials show light emission from red to ultraviolet region due to its 5d–4f transition which is an electric dipole allowed transition. In case of other rare earth ions usual transitions responsible for luminescence emission is 4f–4f transitions which are spin forbidden [21].

Figures 2 and 3 display PL excitation (PLE) and PL emission spectra of $\text{Ca}_{1-x}\text{Sr}_x\text{Al}_2\text{SiO}_7:\text{Ce}^{3+}$ ($x = 0.1 - 4$ mol%) phosphors recorded at room temperature. PLE spectra consist of one weak and one strong peaks located at 253 and 290 nm respectively. Among these two bands, peak at 290 nm is used to investigate PL emission of different Ce^{3+} contents in $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$. All the samples exhibited a similar trend with broad emission band from

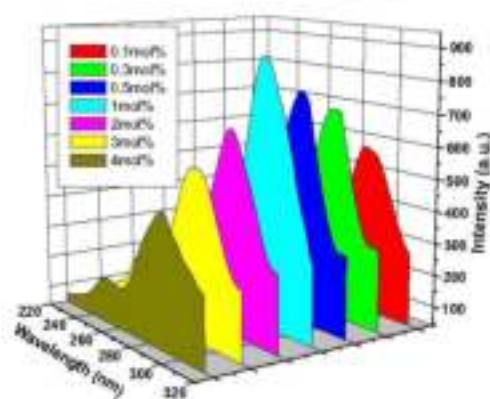


Fig. 2 PL excitation (PLE) spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphors at $\lambda_{\text{em}} = 418$ nm

380 to 560 nm with single peak located at around 418 nm, which is assigned to 5d–4f transition of Ce^{3+} ions.

The inset in Fig. 4 shows the PL intensity as a function of Ce^{3+} concentration x . We can see, a proportional increment was observed in the emission intensity up to 1 mol% of dopant concentration, further increase in the concentration results smaller intensity. This reduction in emission intensity may be ascribed due to concentration-quenching mechanism. Hence optimal doping concentration was stabilised as $x = 1$ mol%. When Ce^{3+} concentration increases the distance between Ce^{3+} ions become shorter, this endorses non-radiative energy transfer between Ce^{3+} ions. One should deal with kind of interaction responsible for non-radiative energy transfer between Ce^{3+} ions that causes concentration

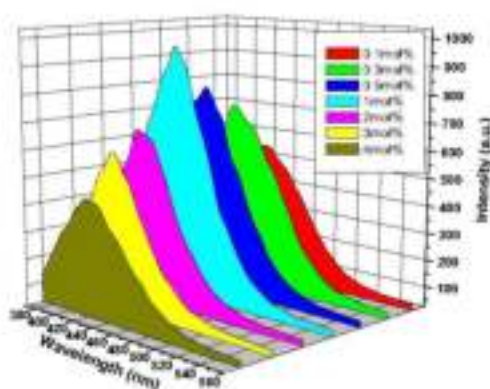


Fig. 3 PL emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphors at $\lambda_{\text{ex}} = 290$ nm

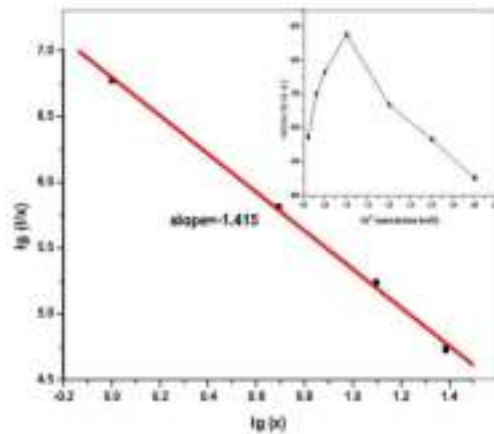


Fig. 4 Dependence of $\lg(I/x)$ on $\lg(x)$. The inset shows PL emission intensity of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphor as a function of Ce^{3+} content (x)

quenching process. According to the Dexter's theory, type of interaction can be determined from Eq. (2), which gives emission intensity per concentration ion.

$$I/x = K [1 + \beta(x)^{\theta/3}]^{-1} \quad (2)$$

where I is emission intensity, x is dopant concentration beyond quenching point, K and β are constants for given excitation wavelength and crystal structure, θ can be 3, 6, 8 and 10 for exchange interaction, dipole-dipole (d-d) interaction, dipole-quadrupole (d-q) interaction and quadrupole-quadrupole (q-q) interaction respectively [22].

If $\beta(x)^{\theta/3} \gg 1$, Eq. (2) can be simplified as:

$$\lg(I/x) = K' - \theta/3 \lg(x) \quad (\text{with } K' = \lg K - \lg \beta) \quad (3)$$

From Eq. (3), it is cleared that θ can be obtained from the slope of $\lg(I/x)$ versus $\lg(x)$, which is plotted in Fig. 4. θ is found to be 4.245, which is very close to 3 rather than 6, hence concentration quenching is originated from exchange interaction in $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphor.

3.3 Thermoluminescence (TL) studies

Thermoluminescence is the phenomenon of stimulated emission of light from an insulating or a semiconducting type material followed by previous absorption of energy from ionizing radiation such as X-rays, α -rays, β -rays, γ -rays, UV-rays etc. Here heat is not an exciting source but it is used to trigger the luminescence emission. Due to irradiation electrons may transit into the conduction band creating hole in the valance band, hence electron-hole pairs generated due

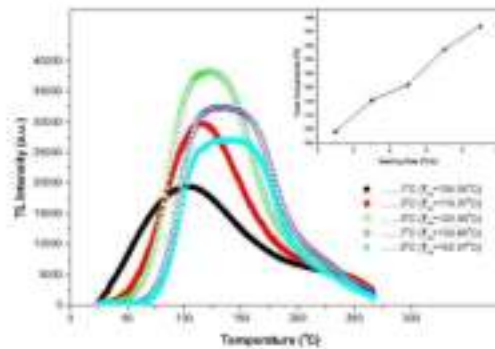


Fig. 5 TL glow curve of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%) exposed to 10 min UV with various heating rates. Inset represents glow peak temperature versus heating rates

to irradiating the material. These charge carriers may wander through the lattice until it may get trapped at the defect levels which are called traps, trapped carriers are thermally released under the condition of heating the material and once again they are free to move in delocalized levels (conduction band for electrons and valance band for holes) for some time. Now they may recombine with a charge carriers of opposite sign, if this recombination is radiative then the emission of thermoluminescence results [23].

3.3.1 Effect of different heating rates

Effect of different heating rates on TL response of 10 min UV irradiated $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%) phosphor has been investigated. Figure 5 represents change in TL intensity and shift in peak temperature at different heating rates from 1 to 9 °C/s. An increment was found in TL intensity and area of glow peak as the heating rate varies from 1 to 5 °C/s and then attenuate for further heating rates. It has been observed that whole glow curve shifts towards higher temperature side as the heating rate increases from 1 to 9 °C/s. This behaviour is originated from the thermal quenching effect. At lower heating rates charge carriers moving towards recombination centres have adequate time to get retrapped and hence producing somewhat low TL response. At higher heating rates, effect of thermal quenching occurs. For higher heating rates, charge carriers released from traps do not directly approach to recombination center and energy transfers via non-radiative transitions [24]. For occurrence of thermal quenching heating rate should be optimized. In the present case maximum recombination takes place for 5 °C/s, thus it is treated as optimum heating rate; hence further TL measurements were performed at heating rate of 5 °C/s.

3.3.2 Effect of concentration and UV exposure time

Figure 6 shows the comparison of TL response of $\text{Ca}_{1-x}\text{Sr}_x\text{Al}_2\text{SiO}_5\text{:Ce}^{3+}$ ($x=0.1-4$ mol%) phosphor followed by 10 min UV-irradiation. Inset in Fig. 6 exhibits that TL intensity linearly increases up to 0.5 mol% and then declines with further increase in concentration due to the concentration quenching mechanism. TL glow curves of all the samples were integrated between temperature range 23–300 °C. The comparison of glow curves of $\text{CaSrAl}_2\text{SiO}_5\text{:Ce}^{3+}$ (0.5 mol%) phosphor with various UV-irradiation time is shown in Fig. 7. It is seen that each glow curve has main glow peak nearly at 141 °C. Inset in Fig. 7 shows plot of total TL intensity versus UV-irradiation time. It is cleared that TL intensity nonlinearly increases from 5 to 35 min UV-irradiation. Maximum TL intensity was obtained for 35 min UV-irradiation time. This may be due to the fact that with increase in irradiation time more and more traps responsible for TL glow peaks are getting filled and subsequently charge carriers get liberated from these traps on heating the material. Finally recombination of these charge carriers with their counterparts results different glow peaks [25]. As we increase UV exposure time after 35 min saturation in TL intensity is found. Because further exposure time could not increase the concentration of trapped charge carriers, since traps become saturated and sealed to fill further charge carriers.

3.3.3 Analysis of TL glow curve

3.3.3.1 T_m - T_{trap} method In order to determine the approximate number of glow peaks and their positions within the glow curve of $\text{CaSrAl}_2\text{SiO}_5\text{:Ce}^{3+}$ (0.5 mol%) phosphor T_m - T_{trap} method is used. T_{trap} was varied from 50 to 290 °C

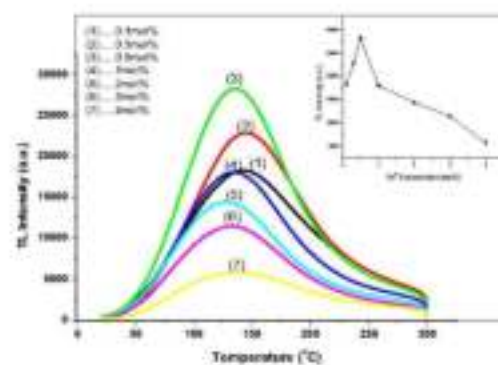


Fig. 6 TL glow curves of $\text{CaSrAl}_2\text{SiO}_5\text{:Ce}^{3+}$ phosphor for 10 min UV exposure. Inset shows influence of Ce^{3+} concentration on TL intensity

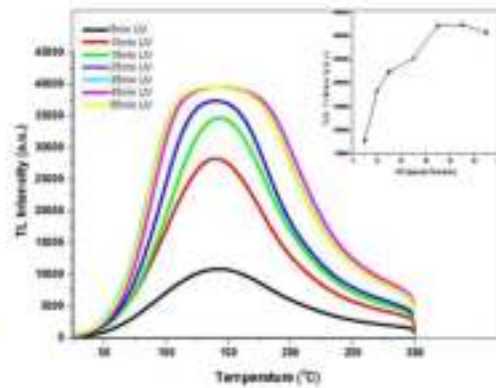


Fig. 7 TL glow curve of $\text{CaSrAl}_2\text{SiO}_5\text{:Ce}^{3+}$ (0.5 mol%) phosphor for different UV irradiation time. Inset shows total TL intensity versus UV exposure time

with difference of 10 °C. For each T_{trap} small amount of sample was irradiated to UV light for 1 min. Once the sample is irradiated two sets of heating treatment were performed on it. In the first step sample was heated from room temperature up to T_{trap} . The sample was then cooled down to room temperature. Second step involves reheating of the same sample up to maximum temperature 300 °C at the same heating rate. In each step TL glow curve was recorded at a constant heating rate of 5 °C/s. For each T_{trap} position of first maximum is noted from the second measurement. Now the entire process is repeated on a freshly irradiated sample at a new T_{trap} . From this analysis staircase shaped T_m - T_{trap} curve was obtained as shown in Fig. 8. Each plateau region in the T_m -

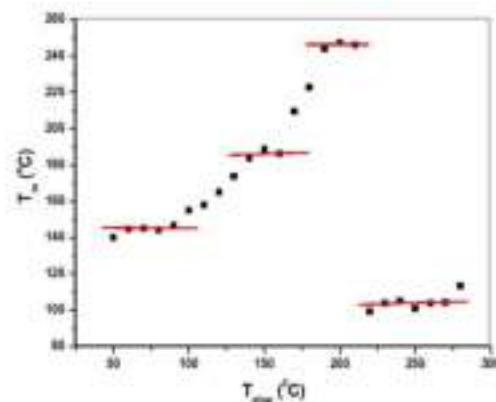


Fig. 8 Plot of T_m against T_{trap} for the glow curve of $\text{CaSrAl}_2\text{SiO}_5\text{:Ce}^{3+}$ (0.5 mol%)

T_{dec} curve corresponds to the occurrence of individual glow peak. Figure 8 shows that there are four noticeable plateau regions found at around 102, 144, 186 and 242 °C. Hence main glow peak at 141 °C of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ (0.5 mol%) phosphor is composed of four Gaussian peaks.

Figure 9 shows glow curve of 35 min UV irradiated $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ (0.5 mol%) sample with its peak deconvolution. This broad glow curve contains four different species of traps at 102.24, 144.29, 186.33 and 242.39 °C respectively which are consistent with $T_{\text{m}}\text{--}T_{\text{dec}}$ method. In order to completely analyse the TL properties of the sample, trapping parameters such as activation energy (E), frequency factor (S), order of kinetics (b) and geometrical shape factor (μ_g) should be determined. In general, activation energy is larger for higher temperature peak and smaller for lower temperature peak that means formation of deeper traps at higher temperature and shallower traps at lower temperature side. Higher activation energy gives better trap stability and hence less fading of high temperature peak [26]. Trapping parameters and activation energies for all the peaks were calculated by using Chen's set of empirical formulae, this method is based on shape of the glow curve and required three temperature points on the glow curve i.e. temperature at maximum intensity T_m ; temperature on either side of T_m at half intensity (T_1 and T_2).

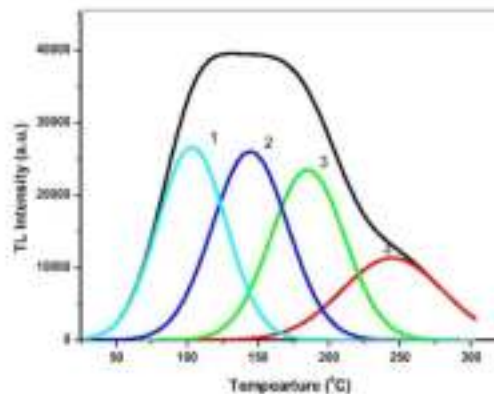


Fig. 9 Peak deconvolution of glow curve of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ (0.5 mol%) phosphor exposed to 35 min UV-irradiation

Table 1 TL parameters for $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ (0.5 mol%) phosphor

Peak	T_m (°C)	μ_g	b	E_a (eV)	E_c (eV)	E_d (eV)	E_m (eV)	S (s ⁻¹)
First	102.24	0.5	2	0.64	0.67	0.66	0.66	10.5×10^9
Second	144.29	0.5	2	0.66	0.71	0.69	0.69	2.61×10^9
Third	186.33	0.46	1	0.76	0.81	0.79	0.79	5.9×10^9
Fourth	242.39	0.53	2	0.94	0.97	0.96	0.96	2.7×10^{10}

The equation used for determination of activation energies, which is true for any kinetics, is given as:

$$E_a = c_a (kT_m^2/a) - b_a (2kT_m) \quad (4)$$

Here a stands for τ , δ and ω ; τ denotes low temperature half width ($T_m - T_1$), δ is high temperature half width ($T_2 - T_m$) and ω denotes full width at half intensity ($T_2 - T_1$). c_a and b_a are given by the following expressions:

$$c_\tau = 1.51 + 3.0(\mu_g - 0.42), \quad b_\tau = 1.58 + 4.2(\mu_g - 0.42),$$

$$c_\delta = 0.976 + 7.3(\mu_g - 0.42), \quad b_\delta = 0,$$

$$c_\omega = 2.52 + 10.2(\mu_g - 0.42), \quad b_\omega = 1.$$

The geometrical shape factor μ_g is defined as ratio δ/ω depending on its value order of kinetics is differentiated. The calculated values of μ_g for first, second and fourth peaks are very close to the theoretical value for a second order TL peak, i.e. $\mu_g = 0.52$ and the value of μ_g for third peak is very close to the value expected for a first order TL peak, i.e. $\mu_g = 0.42$ [27]. General expression for determination of frequency factor is as follows:

$$S = \beta E/k \exp(E/kT_m) [1 + (b-1) 2kT_m/E]^{-2} \quad (5)$$

where β is linear heating rate which is taken as 5 °C/s, E is activation energy, b is order of kinetics; $b=1$ for first order kinetics and $b=2$ for second order kinetics. Trapping parameters, activation energies and frequency factors for all glow peaks are listed in Table 1.

3.3.4 TL emission spectrum

TL spectrum is recorded using various interference band-pass filters ranging from 400 to 560 nm. Figure 10 represents TL emission spectra of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ (0.5 mol%) phosphor. It contains single peak located around 418 nm, which belongs to UV-violet region. This emission generates due to $5d \rightarrow 4f$ transition of Ce^{3+} ions. TL emission spectrum is almost similar to PL emission spectrum.

3.4 Decay characteristics

The long-lasting phosphorescence decay of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ (0.5 mol%) phosphor after 10 min UV (254 nm) exposure has been measured and decay curve is

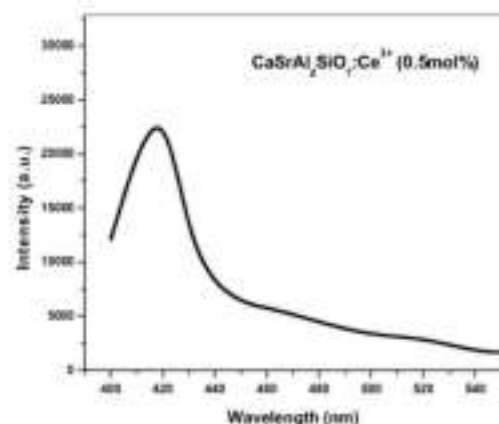


Fig. 10 TL emission spectrum of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%) phosphor

shown in Fig. 11. Afterglow decay is based on exponential function, in the present work decay curve is fitted successfully based on following double-exponential equation:

$$I(t) = I_0 + A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2) \quad (6)$$

where I and I_0 are the phosphorescence intensity at time t and $t=0$, A_1 and A_2 are the fitting constants, τ_1 and τ_2 are short and long lifetimes for exponential component. Calculated decay parameters are mentioned in Table 2

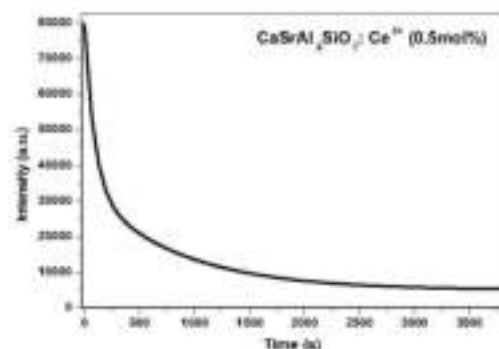


Fig. 11 Decay curve of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%) phosphor after 10 min UV exposure

Table 2 Decay parameters of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%) phosphor

Phosphor	τ_1 (min)	τ_2 (min)
$\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$	1.49	13.88

4 Conclusions

$\text{CaSrAl}_2\text{SiO}_7$ phosphors doped with varying concentration of Ce^{3+} ions were synthesized by conventional solid state reaction method. XRD analysis confirms that the synthesized samples showed tetragonal phase and verified it by matching experimental XRD pattern with JCPDS file. Average crystallite size was determined as 51.57 nm. PL excitation and emission spectra shows characteristic bands of Ce^{3+} ions. Concentration quenching occurs at 1 mol% of Ce^{3+} , which arises due to exchange interaction in $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphor. Phosphor is excited by UV wavelength (290 nm) and gives emission in violet region (418 nm), hence it may be applicable in phototherapy lamps for treating bilirubinemia.

The systematic investigation on TL of UV-irradiated $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphors have been done. Influence of various heating rates on the TL glow was investigated that results shift in peak position to higher temperature side, and decrement in TL intensity after 5 °C/s of heating rate which is originated from thermal quenching process. Concentration dependent TL was also measured and optimal concentration was obtained as 0.5 mol% of Ce^{3+} . For kinetic analysis, glow curve is deconvoluted into three second order peaks and one first order peak; trapping parameters, activation energies and frequency factors were estimated by using Chen's peak shape method. Calculated trap depths are 0.66, 0.69, 0.79 and 0.96 eV. TL intensity increases with UV-irradiation time then becomes saturate. TL emission spectra was also recorded and single emission peak is located in violet region. To study long afterglow property of phosphor, decay curve has been plotted. There is a requirement to improve TL characterization of this material so that its dosimetric properties can enhance and gives applicability of this material in radiation technology.

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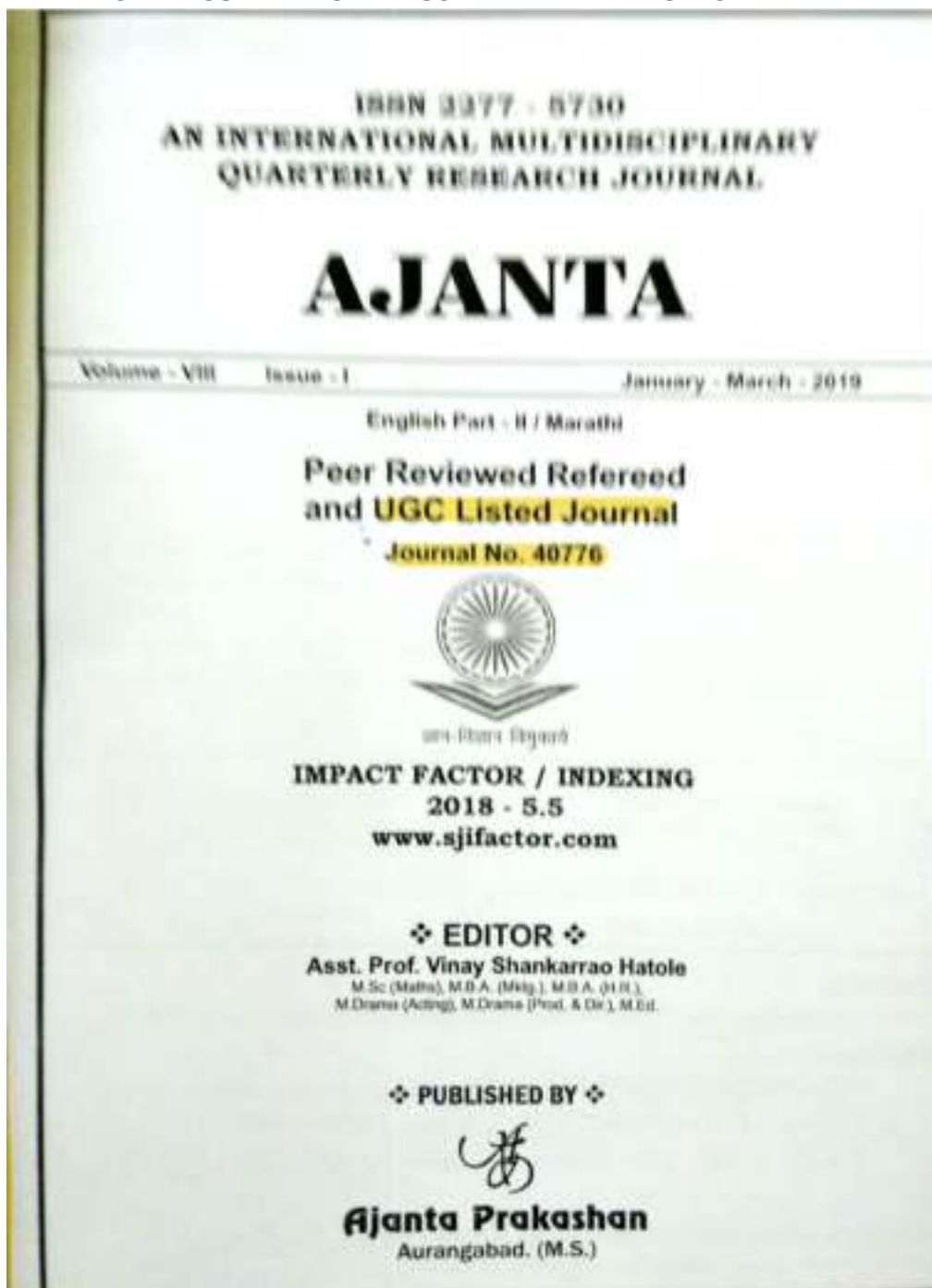
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2018-2019

RECENT TRENDS IN E-COMMERCE INDUSTRY IN INDIA: RUPESH R. DAHAKE



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VOLUME - VIII, ISSUE - I - January - March - 2019
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❧ CONTENTS OF ENGLISH PART - II ❧

S. No.	Title & Author	Page No.
14	Tribal Economic Development in India Dr. Ganesh M. Khekale	66-72
15	Importance of Business Ethics Dr. Rajendra M. Tated Prof. Vinit R. Mahure	73-80
16	Recent Trends in E-commerce Industry in India Prof. Rupesh R. Dahake	81-89
17	Impact of Core Banking System on Increase Employees Productivity and Knowledge in Yavatmal District Central Cooperative Banking, Yavatmal Dr. Radhesham P. Choudhari	90-94
18	Skill Development for Youth in India: Challenges and Opportunities S. M. Shegokar	95-98

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
 AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.ajifactor.com)

16. Recent Trends in E-commerce Industry in India

Prof. Rupesh R. Dahake

Assistant Professor, Adarsha Science J.B.Arts & Birla Commerce
 Mahavidyalaya Dhamangion Railway.

Abstract

Future of ecommerce industry in India now a days most debating topic because so many things materialize with this sector now a day. Reason for making this research paper was to know the future and impact of ecommerce on Indian market. Everyone know India is the 2nd biggest market in the world and most promising market for investors in return point of view that why so many investor investing their moneys in India special in ecommerce sector because of government policy, penetration of internet infrastructure and awareness about the new online market. Now ecommerce is most popular word in India because of huge discount, door to door services, variety access of global product and services through internet. Now Indian consumer more aware and liking online shopping through mobile and computers. There recent statistics which prove above statement The Indian e-commerce industry has been on an upward growth trajectory and is expected to surpass the US to become the second largest e-commerce market in the world by 2034. India's e-commerce industry is expected to grow from US\$ 38.5 billion as of 2017 to US\$ 200 billion by 2026. India's internet economy is expected to double from US\$125 billion as of April 2017 to US\$ 250 billion by 2020, majorly backed by ecommerce. Online shoppers in India are expected to reach 220 million by 2025. Average online retail spending in India was US\$ 224 per user in 2017. (<https://www.ibef.org>) This paper focus on current & future status of ecommerce in India.

Keyword: - Ecommerce, internet penetration, investment, **Product Differentiator**

Introduction

Why Indian ecommerce business so rising which growth is not match with any other country in the world reasons are Rising Internet penetration, access of global majors, rise in smartphone adoption, innovation in mobile technologies, access to capital, optimistic consumers and digital payments is fuelling the growth of the ecommerce market in the country. It also delves into the various business segments, with highlights on growth, presence of start-ups, and

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future prospects. The report also showcases stratus that are emerging in niche segments of ecommerce and expanding the business opportunity.

India is the fastest emergent market for the E-commerce business. Being driven by a young demographic profile, increasing internet penetration and relative better economic performance, India's E-commerce revenue is expected to jump from US\$ 39 billion in 2017 to US\$ 120 billion in 2020, growing at an annual rate of 51 per cent, the highest in the world.

This growth has been enabled in part by the ecommerce market leaders in India—Flipkart, Amazon and Paytm Mall—who have been expanding into new sectors, including grocery. This investment along with India's rising internet and smartphone use, as well as the country's young population and expanding middle class, have contributed to a growing number of digital buyers. We expect a quarter of the population will be digital shoppers this year.

"Ecommerce is booming in India thanks to increased internet users and cheaper smartphones," said Eric Haggstrom, forecasting analyst at eMarketer. "In tandem with this shift to online and mobile usage, Flipkart, Amazon and Paytm Mall have been competing fiercely to claim their share of the Indian market. All three of these companies are making large investments, which include improved logistics and payment systems, as well as offering deep discounts, which will fuel future growth in the market.

Objective

1. To know the future ecommerce industry in India
2. To find out the reason ecommerce industry growth in India.
3. To Find the opportunities and challenges to ecommerce industry
4. To know effect on Indian industry

Research Methodology

This paper based on desk research so I used most of the data is secondary in nature for analysis purpose.

1. Primary Date: - Not applicable
2. Secondary Data: - Research Reports of IBEF, NASSCOM etc., Website, Web Articles-

E-Commerce Industry in India

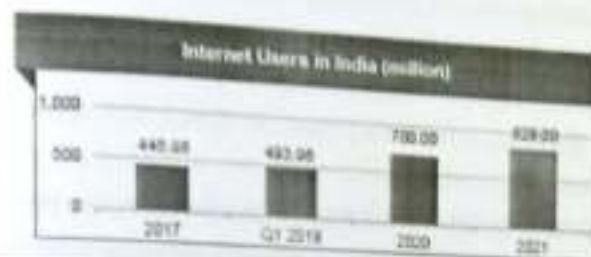
With increasing internet penetration and adoption of smartphone, ecommerce is India's fastest growing and most dynamic channel for commercial transactions. The Indian ecommerce is expected to outshine the United States to become the second largest ecommerce market in the

ENGLISH PART - II / Peer Reviewed Refereed and UGC Listed Journal No. : 40778

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VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
 AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.ajfactor.com)

world. The future of ecommerce industry in India so optimistic statistics show positive upward trends in growth by 2026. In 2017 ecommerce market value was 38.50 \$ billion dollars this will go further which shown in chart. Forecasting show promising growth in 2026 the market value of ecommerce industry is 200 \$ billion dollars. Internet user in India now 445.96 million it will reach 829.00 million in 2021 so their bright future of ecommerce in India. (According to report of IBEF 2018)



Online Retail Market in India

Most selling item on online shopping is electronics product around 48% sales coming from this segment and most popular product in India is apparels around 29 % market related with this it's clear both this product belongs to young generation and statistics prove that due to young population in India ecommerce market outshine. (According to report of IBEF 2018)



ENGLISH PART - B / Peer Reviewed Refereed and UGC Listed Journal No. - 46773

63

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Why Indian E-commerce Market is Growing?

- **Growing Demand:** - India is the fastest growing market for the E-commerce sector. Being driven by a young demographic profile, increasing internet penetration and relative better economic performance, India's E-commerce revenue is expected to jump from US\$ 39 billion in 2017 to US\$ 120 billion in 2020, growing at an annual rate of 51 per cent, the highest in the world.
- **Increasing Investment:** - The recent rise in digital literacy has led to an influx of investment in E-commerce firms, levelling the market for new players to set up their base, while churn out innovative patterns to disrupt old functioning. E-commerce industry in India witnessed 21 private equity and venture capital deals worth US\$ 2.1 billion in 2017 and 40 deals worth US\$ 1,129 million in the first half of 2018.
- **Policy Support:** - In India 100 per cent FDI is permitted in B2B E-commerce, as per new guidelines on FDI in Ecommerce, 100 per cent FDI under automatic route is permitted in marketplace model of E-commerce.
- **Attractiveness Opportunities:** - A lot of India's blue-chip PE firms had previously avoided investing in E-commerce but are now looking for opportunities in the sector. India's start-up ecosystem is growing supported by favourable FDI policies, Government initiatives like Start-up India and Digital India, as well as rising internet penetration driven by market players like Reliance Jio.

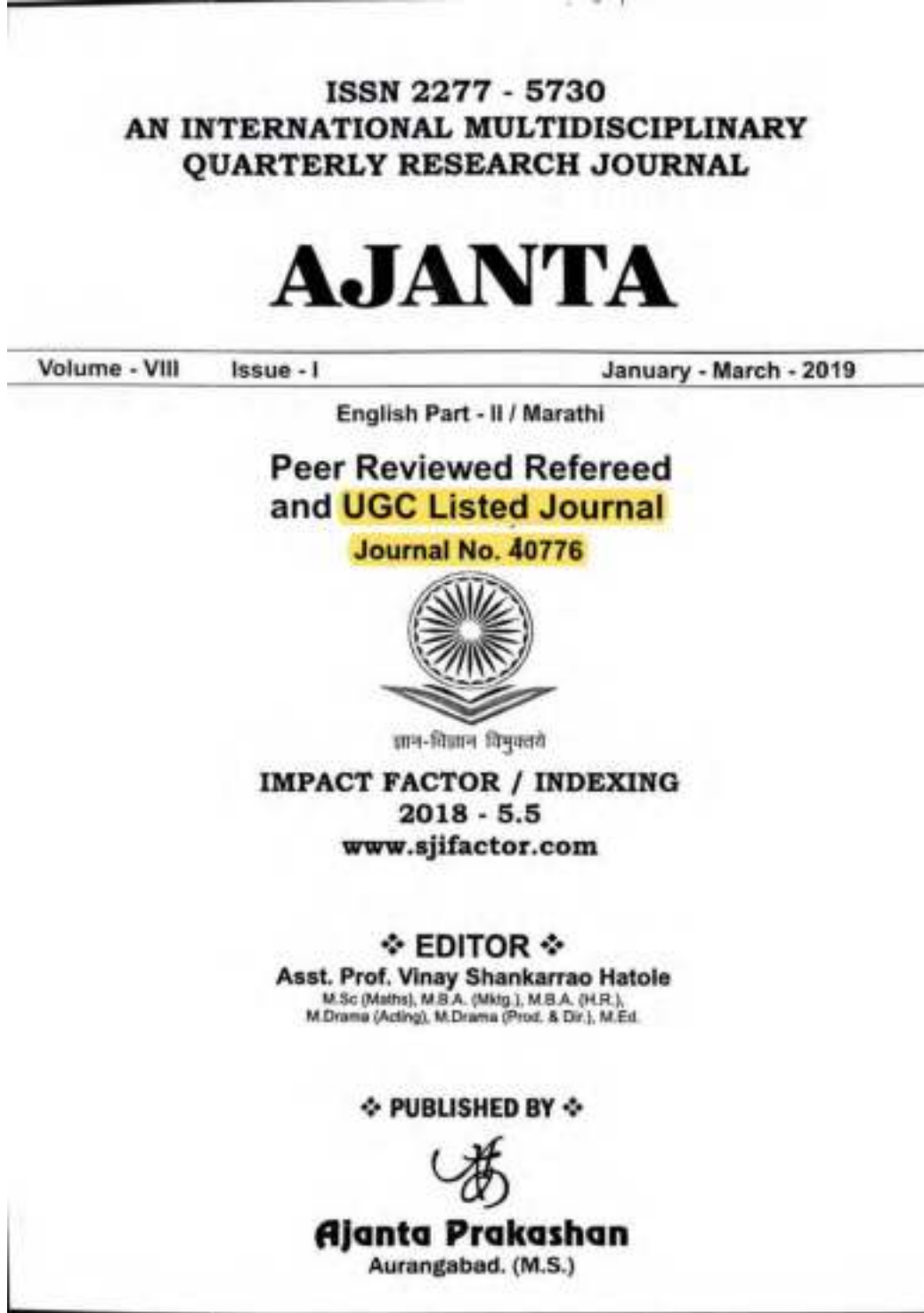
Current Market Trends in Ecommerce Market in India

eCommerce in India can be broadly categorized as: domestic and cross-border, B2B and B2C, marketplace and inventory based, and lastly single brand and multi brand. Technology-enabled innovations such as digital payments, hyper-local logistics, analytics-driven customer engagement and digital advertisements, have enabled the eCommerce industry in India to grow at a much faster rate. Government initiatives such as Digital India, Skill India, Startup India and Make in India are also contributing to the growth of the eCommerce industry.

Some of the Major Developments in the India Ecommerce Sector are

- Walmart's acquisition of Flipkart, an Indian e-Commerce platform, for \$16 billion;
- Launch of the Paytm Payment Bank, India's first bank with zero charges on online transactions, no minimum balance requirement and free virtual debit card

E-business - Challenges & Opportunities: Dr Narendra Kawduji Nagpure



CONTENTS OF ENGLISH PART - II

S. No.	Title & Author	Page No.
1	Emerging Trends in E-Commerce: An Outline Dr. Yogesh L. Patinge	1-4
2	Changing Trends of Indirect Taxes in India Swati A. Saurangpate	5-12
3	Skill Development: Challenges and Opportunities Prof. Dr. Sunil N. Dhere	13-16
4	Comparative Study of VAT and GST in Indian Pharmaceutical Sector Industry Dr. Anil Marotrao Raut	17-21
5	A Skill Development - A Step towards Kaushal Bharat Dr. P. S. Vishwakarma	22-26
6	Business Ethics: An Integrated Approach Dr. Naglaxmi Narsingrao Tirmanwar	27-30
7	Role of Skill India in Economic Development Dr. Ravindra Bhaurao Tembhurne	31-35
8	Advertising World with Augmented Reality Technology Prof. Dr. Aruna R. Chudasama	36-42
9	Emerging Trends in Indian Agricultural Dr. Ulhas N. Medshikar	43-48
10	Impact of Demonetization on Retail Sector Prof. Kishor Narayanrao Tayade	49-53
11	Impact of Goods and Services Tax on Manufacturing Sector with Reference to Amravati City Dr. Nitin Wamanrao Jaswante	54-58
12	Agro Entrepreneurship: A Vital Tool for Sustainable Agriculture Dr. A. G. Naranje	59-61
13	E-Business - Challenges and Opportunities Dr. Narendra K. Nagpure	62-65

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
 AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

13. E-Business - Challenges and Opportunities

Dr. Narendra K. Nagpure

Asst professor, Adarsha Mahavidyalaya, Dhamangaon Rly., Dist. Amravati.

Abstract

The Internet as a fundamental platform of worldwide e-business has emerged both as the world's major distribution channel for goods and services and as its platform for international co-operation between business partners. This is profoundly changing economics, markets and industry structure, products and services and their flow, consumer segmentation, consumer values, consumer behaviours, jobs and labour markets.

No types of e-business are necessarily better than any other. It's up to each business to determine what types of e-business are right for them. E-business affects how a company views its strategy and operations: It is more than just technology. Most businesses have started the process of transformation into e-business by investing in email, a web presence and intranets. These form the foundation for a company's e-business strategy and create a basis from which an improved business to business relationship and business to consumer relationship can be established. This paper tries to attempt to know the objectives of e-business, relationship between e-business and e-commerce, growth of e-business, key principles, growth of e-business benefits and opportunities and challenges hindrances of e-business.

Introduction

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ENGLISH PART - II / Peer Reviewed Refereed and UGC Listed Journal No. : 40776

Improved business to business relationship and business to consumer relationship can be established.

Incorporating ICT technology into core business processes enables new levels of relationship to be established with customers, distributors, suppliers and partners. The main areas e-business can maximize business values are; customer relationship supply chain management and e-commerce. By using ICT technology these three business-critical processes can be transformed into a single, streamlined system.

Definition of E-Business

E-Business can be defined as the use of the internet to network and empowers business processes, electronic commerce, organizational communication and collaborations with a company and with its customers, suppliers and other stakeholders.

Key Principles of E-business

E-business is global nature. However, that is more a vision of the future than the reality of today. The business of big companies is more and more international and e-business tools are critical to their success. E-business simplifies international business also for SME's and offers big markets, but most of SME's are today satisfied with its. Thus not even enterprises with only local operation can totally forget e-business.

The e-business communication networks is global even where e-business is local. E-business is simultaneously an opportunity and a threat. As enterprises can enter new markets with it, so can a foreign company enter domestic markets with it.

A high level of trust in the global information infrastructure and services should be pressured by mutual agreement, education and further technological innovation to enhance security and reliability adoption of adequate dispute resolution mechanisms and private sector self-regulations.

E-business has both industry and enterprises level impacts. It has redefines enterprises and industries boundaries creating digital winners and loser along the way.

Benefits and Opportunities of E-Business

- **New business models and more revenue:** The enterprise can create additional revenue. Some business are fortunate because they provide information, goods and service that users are willing to purchase online. Such sectors are travels, financial

order catalogues.

- **Extended hours (24x7/365):** Increasingly today, the terms 'available' and accessible a business mean twenty-four hours a day, seven days a week access to information products and services. Customers, suppliers and interested parties are less and less satisfied with being restricted to the traditional opening hours of business and will not be impressed if they cannot do such things as order items or discover information for themselves in the comfort of their own homes and offices when it suits them.
- **Global reach and up-to-date company material:** E-Business can improve marketing and be a highly effective promotional tool. It is another promotional tool that should complement other forms of promotion. Besides being visible to potential customers and suppliers. It is also very easy to collate and analyse information about those visiting and buying from the site and equally easy to change the promotional aspects to maximize their impact on sales.
- **Cost reduction:** There are many ways in which an enterprise can save costs and improve operational efficiency E-Business. It helps streamline processes.
- **Improved customer relations and services:** An enterprise can make it easier for companies and people to do business. A website is an additional tool for facilitating business. It should be quick and easy to navigate, customer focused, offer abundant relevant information, provide opportunities for two-way communication and provide a variety of convenient ways to do e-Business.

Challenges and hindrances of E-Business

- **Cultural Issues:** Automated business processes require a cultural change to the way business is conducted and services offered. People typically resist change, especially if they are not sure about the outcome. Not all people know E-Business well and the effects of E-Business are so many that there is space for resistance.

Conclusion

In today's environment more than ever, managers of "old economy" companies need the right tools to support and improve their effectiveness when making major strategic moves, allocating scarce resource, and managing risk. Why? Because the large "old economy" companies from consumer products to industrial manufacturing have begun to see relatively

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AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

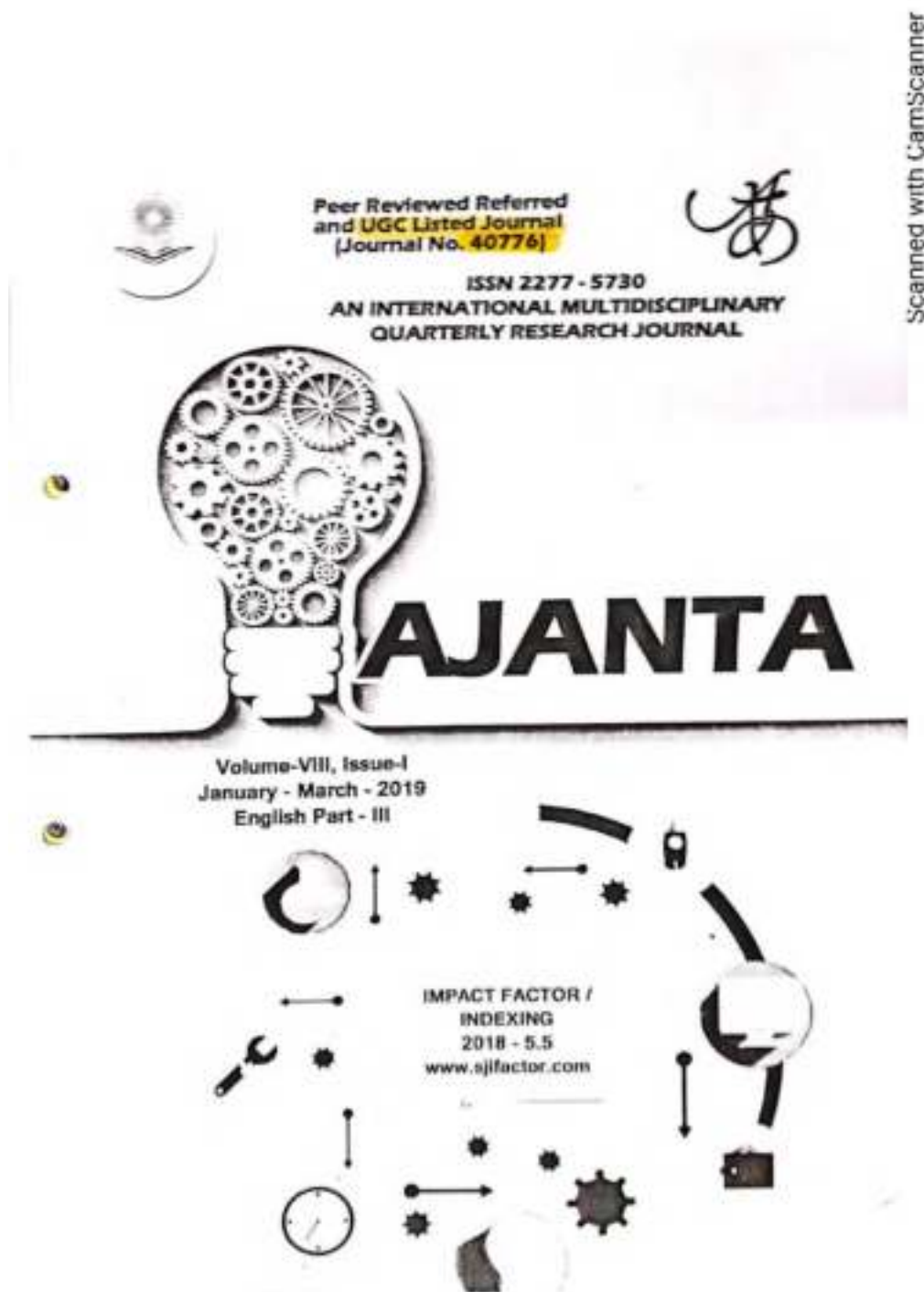
small pieces of their markets taken away by new, Web-enabled firms. As a result, they're waking up to the e-business threat (and opportunity) and have started to push toward more efficient digital strategies based on optimizing customer experiences, integrating their value chains, and accelerating information flow.

Clearly, we're in the early stages of a revolution that's changing the business landscape. As with any revolution, there will be moments of extreme optimism when the potential reveals itself; there will also be moments of extreme pessimism when scepticism rules. However, one thing is certain. E-business is creating new opportunities for companies willing to adapt. For other companies, this same revolution represents a destabilizing threat to the status quo of "business as usual."

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Negative Capability in Keats' Ode to Psyche: Sanjay Datta Patil



2018-2019

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

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14. Negative Capability in Keats' Ode to Psyche

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John Keats is one of the greatest of the English Romantic poets. Besides his unique achievement in poetry, Keats made a niche in the field of criticism by his conception of 'Negative Capability'. The English Romantic Movement has been a spring of critical theories about poet, poetry and poetic method. Wordsworth, Coleridge, Hazlitt have contributed immensely to the Romantic theory of poetry, with their dynamic interpretation of Shakespeare. Wordsworth's ideas of poetic diction, Coleridge's theory of Fancy and Imagination, Hazlitt's conception of the 'Spirit of the Age' have widened the venues of English literary criticism. Living in the age of these great critics, Keats too did not lag behind in evolving new concepts of poetry. He coined the phrase 'Negative Capability' to stress the objective or impersonal poetry as against the poetry of self expression which was the essence of the Romantic poets and the conception of the Romantic poetry. Even at a very young age, Keats started reflecting the seminal problems of poetry, its nature, function and aim and also resolved them, partially, and most of these meditations found their way into his letters and also poems. In the history of English poetry, there is no other poet whose poetry is so much about poetry, as Keats' is.

Keats coined the term 'Negative Capability' to describe the exclusive and unique ability of Shakespeare. The discovery of Shakespeare's capacity for understanding contrary points of view led Keats to celebrate this new celebrated formulation. The Negative Capability for Keats is the surrender or annihilation of one's self, both in experience of life and also in the aesthetic creation. The phrase 'Negative Capability' first appeared in Keats' letter to George and Tom in December 1817 where he mentioned--- I mean Negative Capability, that is when a man is capable of being in uncertainties, mysteries, doubts, without any irritable reaching after fact and reason.

John Keats was the last to be born and first to die among the English Romantic poets. Keats is remembered for his opening lines of his first long poem 'Endymion',

*"A thing of beauty is a joy for ever;
It's loveliness increases; it will never*

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

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*Pass into nothingness; but still will keep**A bower quiet for us; and sleep**Full of sweet dreams, and healthy, and quiet breathing."*

However, it is an attempt to comprehend the Negative Capability in 'Ode to Psyche'. It is normally deemed that 'Ode to Psyche' is the first chronologically and it is straight-forward and descriptive and less searching in thought. In classical legend, Cupid, the winged love-god had fallen in love with the nymph Psyche and had often made love to her by night in blissful Arcadian bowers; eventually, after Cupid's intercession with Jupiter, Psyche became a winged goddess. In the 'Ode to Psyche', Keats changes the legend so that she is already 'winged at the time' of the sylvan embraces. In the ode, the poet first describes his vision of the two lovers embracing 'on the bedded grass'; he then praises the beauty of Psyche. Regrets that she was admitted too late to the Pantheon to be the object of rituals of worship, and says that to make amends he will himself be her priest and will create in his imagination a natural sylvan and floral temple for her glory and pleasure. What emerges is a celebratory vision of idealized love, enthusiastic and affirmative, unclouded by Keats' doubts misgivings and sense of mortality. The poet is too much at ease in this dream of 'soft delight' but there are still some ripples of linguistic energy.

It is Keats' mastery to combine the two models of a sonnet and an ode. The Ode to Psyche is alleged to be devoid of explicit ideas. The rich sensuous imagery of the poem and its lyric ardour and exaltation do not detract our attention of the main motif of the poem. The primary intention of the poet is a kind of reorientation towards ancient mythology. It is opined that Keats takes delight in the exquisitely and spiritually symbolic product of the mythological genius of expiring paganism. But to restore the lost innocence in an unbelieving age requires a special imaginative effort. A detailed reading of the poem reveals a fundamental reappraisal of the value of mythology and of mythology-oriented aesthetics.

In the ode Venus becomes jealous of Psyche because of her beauty and sends Cupid to make Psyche fall in love with an unsightly and vile creature. But Cupid himself falls in love with Psyche. The oracle of Apollo asks Psyche's father to leave her on a barren rock where a serpent would marry her. The unhappy Psyche being left alone is carried in to a deep valley. She finds a palace where she would find her husband. Cupid visits her there every night and asks her not to try to see her husband. Due to her curiosity and her friend's persuasion, Psyche one night looks

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

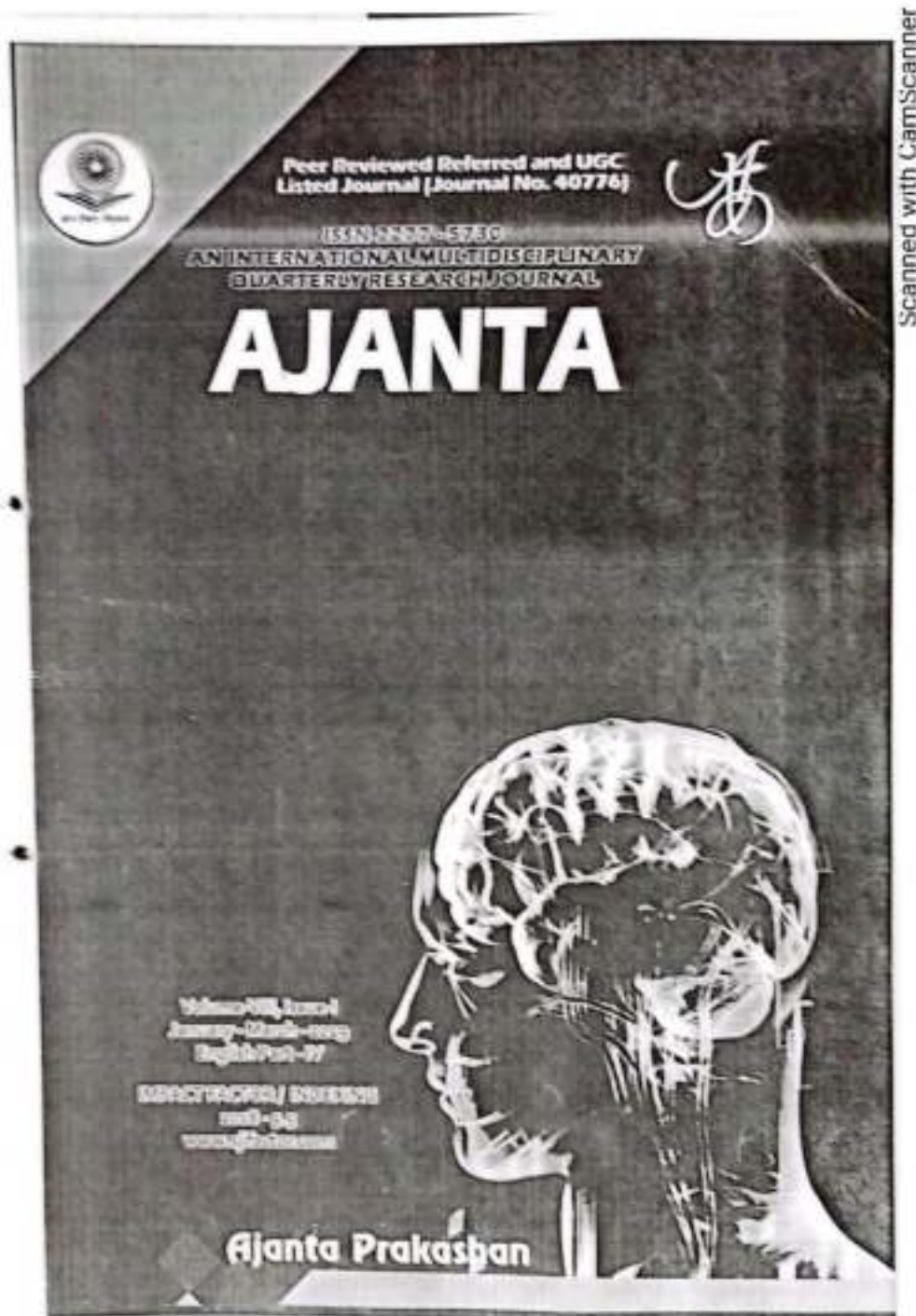
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at her lover. Actually she wants to cut his head off but she was startled to see the beauty of her lover and trembling falls at his feet. Cupid awakes in anger and disappears. Psyche remorsefully sends her husband all over the earth. During her wandering, she falls in to the hands of Venus. Finally Jupiter, at Cupid's prayer, consents to their marriage. Psyche is made immortal and is reunited to Cupid. This theme serves two purposes of Keats: the journey of a mortal creature finally attaining to immortality. From this angle he can prove the transformation of mind into soul through the trials of experience. Secondly it shows the emergence of human consciousness as a new deity, as the unique feature in the evolution of human history.

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Women in Buddhism and Present Scenario: Sanjay Datta Patil



2018-2019

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

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14. Women in Buddhism and Present Scenario

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There was a great difference between Vedic period and Buddhist period as far as the status of women in the society is concerned. Before Buddha, girl child was considered as a burden on the family. Their birth was regarded no more than a problem for the parents. But during Buddha time the status of women in the society was far better than earlier. During Buddha period the word "putta" was used for a new born baby, which was common to both for a male child and a female child. On the basis of gender there was no discrimination. But if we see the period before Buddha, we find this discrimination on large scale. The birth of a female child was seen as a curse on the family. But this misconception, prevailing in the society, regarding women, was altogether different during Buddha period.

There was a revolution and woman was no exception to this. During this period, women enjoyed more liberty and equality as men were entitled to, and they got respectable status in the society. However, their work was restricted to social, economic, religious and household affairs only but their place was equal to men in the society than earlier. They freed themselves from the clutches of male dominated society. This agitation of Buddha's time later on developed and spread all over. Buddha himself was at the centre of this agitation and women themselves led this agitation. They with their intellect, cleverness and self-confidence, established prominent place in the society.

They started getting venerable place and honour in the society. They were free from mental and physical slavery. Child marriages were stopped. They could choose the groom of their choice. Girls from high society and princess started selecting 'swayamwar'. The status women had 'Dasi' (slave) earlier, came to an end. Now she became co-traveller and sometime guide of her partner. She started to get honourable position in the family and she was given power to manage the financial affairs of the family.

Woman is one of the most valuable gifts in the universe. Buddha said, woman is the best of all gifts and her necessity is beyond measure as Bodhisatva and Chakravarti Samrat could take birth because of them. In Buddhism the life of woman has got new dimension and important meaning and she realized that her life was of her own and would remain free till death. She

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

became independent and self-reliant and there was no need to be dependent on man. This fact was first understood in its real sense by the women, during Buddha period.

The revolt that was undertaken against social condition, (in which woman was suppressed) by Buddhist religion, was fruitful. The women that had taken "Deeksha" as "Bhikkhuni" (disciple) became famous for spiritual and moral life. Bhikkhuni Khema was one of the disciples of Tathagat. She was popular as just, skilled, scholar and expert in debate. Women in Buddha period were always ready to give challenges to earlier philosophy. The meaning of a quote by one of the disciples (woman) is very appropriate here-

'One can win thousands of people in battle, but the best winner is he who wins himself'.

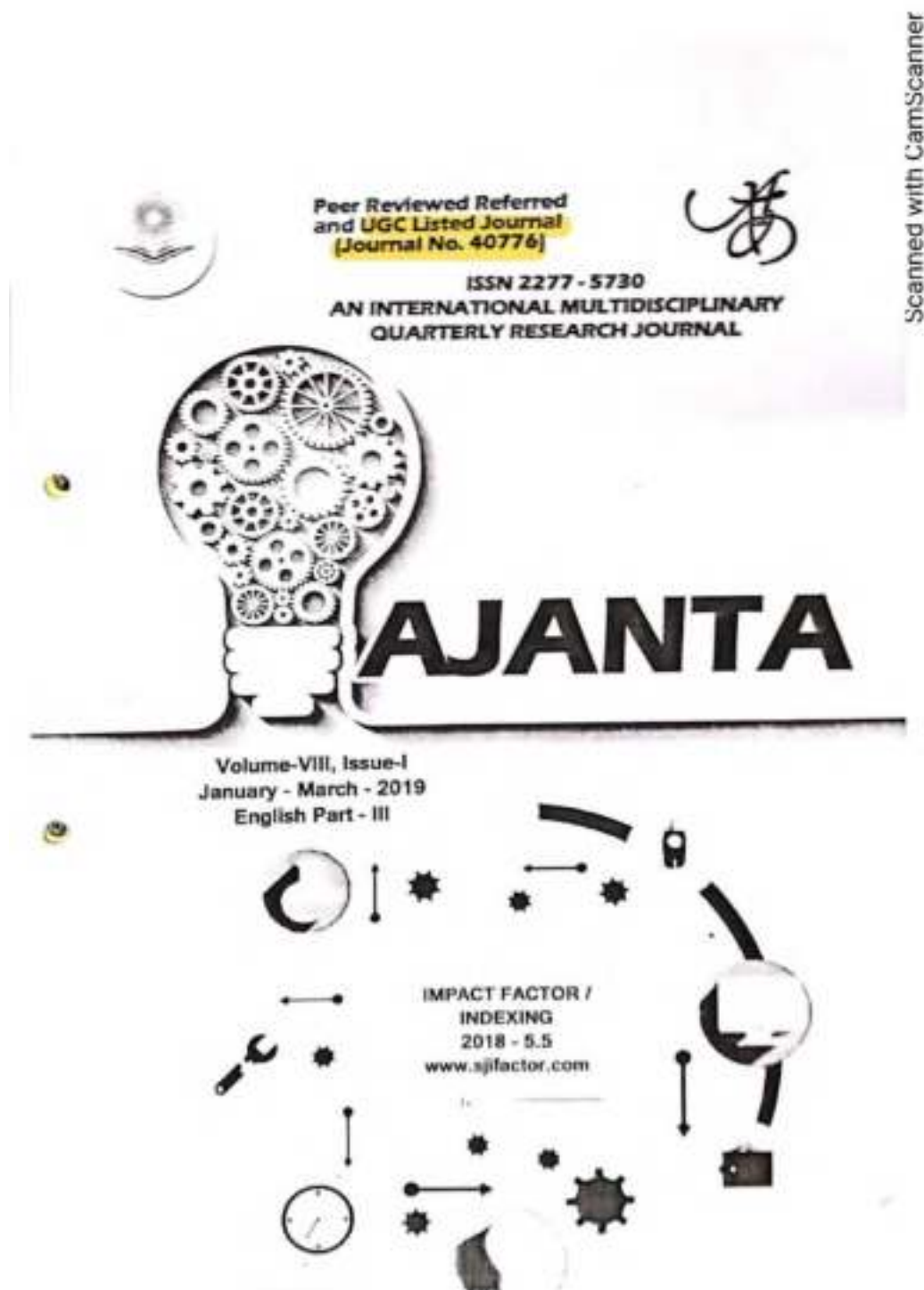
With the same thought, women in present days are marching ahead and it is taking place due to Dr. B. R. Ambedkar who, through Indian constitution made man and woman equal. He destroyed caste discrimination, made reservation policies for the backward class people. Women are being empowered day by day. In 21st century the condition of women is of a very strong kind. Earlier women were not allowed to prosper but today they are free to do whatever is proper and right for them. Today they are being benefitted by the policies made by the constitution and we are witnessing it. Today we celebrate World Woman's Day on 8th March. Everywhere we see women organizing symposia, lecture series etc. Today women are not lagging behind to get the position that men are getting for years. They are achieving everything on their own. They are getting the highest positions like president, ministers, IAS, IPS etc. This is possible because of the equal rights given to them, education and their hard work. Smt. Pratibhatai Patil became the first president of the country. Late Indira Gandhi led the nation as a prime minister for many years. There are so many women who are making their contribution in different fields like sports, science, politics, literature, etc. and proving their talent.

The credit for the prosperous condition of women goes to Buddhism and the laws made by Dr. B. R. Ambedkar. This could be possible due to education that women are allowed to get. It is the outcome of those people who fought for the freedom of women.

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Existential Reading of Hamlet: Jeetendrasingh Gangasingh Barulkar



VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
 AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

CONTENTS OF ENGLISH PART - III

S. No.	Title & Author	Page No.
13	Existential Reading of Hamlet Jeeendra Barulkar	79-81
14	Importance & Techniques of Fitness Mr. Abhay S. Chandekar	82-85
15	Cardiovascular Endurance Improve by Exercises Ganesh Vishwakarma	86-89
16	Grammatical Relations Asst. Prof. G. R. Khanderao	90-97
17	Green Library - A New Concept of Library Dr. Virendrakumar L. Barde	98-103
18	Green Library: Implications at the National Level Dr. Neeta A. Kene	104-108
19	Impact of Cashless Economy on the Following Areas Banking & Finance, International Business, Marketing, Information Technology, Human Resource Ms. Komal Ashok Gupta	109-114
20	Emerging Trends in Teaching English Language and Literature Asst. Prof. Ku. Goldie K. Jambhulkar	115-124
21	Impact of Social Media on College Libraries Dr. Shashikant V. Wankhade	125-128
22	Implementation of TELL to Improve English Communication Skill for EFL Learners Gajanan B. Mane	129-133
23	Initiative of Green Library Today's Need Dr. Sarika M. Sable	134-138
24	Social and Economic Implications of Digitalization Ms. Vidula Ramachandra Katkamwar	139-144
25	Online Marketing: Emerging Trends and Issues Prof. Rahul G. Mahure	145-150

VOLUME - VII ISSUE - I JANUARY - MARCH - 2019
 AJANTA - ISSN 2278 - 5730 - IMPACT FACTOR - 5.5 www.ajfactor.com

13. Existential Reading of Hamlet

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Abstract

This paper attempts to explore the tenets of Existentialism in Shakespeare's Hamlet. Though indecision and revenge apparently seem to be the themes of the play, existential anguish and freedom of choice are inseparable from the play. Hamlet and the philosophy of existentialism both are equally fascinated by issues such as freedom of choice, inwardness, and alienation and becoming.

Keywords: Existentialism, Freedom, Alienation, Choice

Existentialism, one of the major schools of thought, emerged as post World War I phenomenon. It has a specific intellectual history which begins with the precursors like Kierkegaard, Nietzsche, and Dostoyevsky; it got fullest expression in the philosophies of Sartre, Heidegger, Jaspers and Beauvoir. However, it did not emerge as a single philosophical system. It was rather an attitude of life. According to *The Cambridge Dictionary of Philosophy*, "Existentialism focuses on the uniqueness of each human individual as distinguished from abstract human qualities" (*The Cambridge Dictionary of Philosophy*, "Existentialism", 1999, pp. 296). In this sense both Shakespeare and the existential thinkers seem to investigate into human existence- how humans live in the world, how do they interact and react with the surroundings. Shakespeare's plays, particularly his tragedies, resemble closely with modern existential thought. In 1956, Walter Kaufmann published *From Shakespeare to Existentialism: An Original Study*. It was the earliest observation to relate these two topics.

Shakespeare in his tragedies deals with the deeper, inward experience of characters and forces us to question what it means to exist as humans. He is concerned with not just who we are but also with how do we exist and how do we interact. Existentialism comes through in some of the darkest moments of the Shakespearean plays. *Hamlet* is full of passages and line that are extraordinarily existential in nature. Karl Jaspers has rightly observed, in Shakespeare's plays, "Human Life understands itself in terms of its potentialities and perils, its greatness and nothingness, its human and diabolical strains, its nobleness and meanness, its sheer joy at being

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
 AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

alive, and its bewildered terror at failure and destruction, its love, dedication, and openness of heart, and then again its hatred, narrowness and blindness." (Jaspers, 1953, pg. 29-30)

The basic principle of Existentialism is that "we human beings exist without justification... condemned to assume full responsibility for our free actions and for the very values according to which we act..." (The Cambridge Dictionary of Philosophy, "Existentialism", 1999, pg. 298) Hamlet is the existential hero who is in search of his true identity. He faces and suffers from existential anguish of freedom. He seems to be the man on the mission to find the meaning of being. Hamlet poses before us the question common to every man: "What a piece of work is a man!" (II, ii, 296), for him the question is-

"To be or not to be, that is the question
 Whether 'tis nobler in the mind to suffer
 The slings and arrows of outrageous fortune,
 Or to take arms against a sea of troubles,
 And by opposing end them? To die: to sleep. (III, i, 56).

Hamlet has traditionally been categorized as a revenge-tragedy but the character of Hamlet is far more than a revenge hero. Unlike other revenge-tragedies, Hamlet suffers from problems of his own character. He suffers from the anxiety of choice that he has got to make in order to take revenge. Hamlet frequently denies playing his role of a revenge taker. He suffers from inaction and avoids responsibility. Hamlet gets into the role of a modern man who is suffering and looking for a refuge or an escape from the situation. In his dilemma whether to avenge his father's death or not, Hamlet laments:

The time is out of joint. O cursed spite
 That ever I was born to set it right. (I, v, 189-90)

The tragedy of Hamlet portrays before us the predicament of modern man, tangled between confused self and tragic circumstances. Existentialism speaks about the confused, unpredictable nature of man who struggles make his choices. Hamlet is always in the middle of confused state and contradictory situations. Hamlet's delay in taking revenge of his father's death emphasizes the plight he takes from the situation. Various characters in the play including Ophelia and Polonius try to discover the true character of Hamlet but they failed to find a fixed nature of his character. The real Hamlet is unknown to all, even to himself. Hamlet undergoes various changes through the play. He is often confused about what to do. He even turns mad. His

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
 AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

madness baffles people around him and the queen calls him "my too much changed son" (II, ii, 36). But the changes that appear in the character of Hamlet are often believed to be superficial. King Claudius observes:

... Something have you heard
 Of Hamlet's transformation: So call it,
 Since nor th' exterior nor the inward man
 Resembles that it was... (II, ii, 4-7)

Harold Bloom argues that Shakespeare's characters develop because they reconceive themselves (Bloom, 1998, pg. xvii). This is a very appropriate observation in relation with existential thought. It is by living and experiencing and evolving that Hamlet's character defines himself. In existentialism, a loss of self is a necessary precondition for repossession or redefining of the self.

Another existential feature that is prominent in Hamlet is the absence of orderly world. In this play, Shakespeare does not offer us a just and orderly world. The lack of an orderly world offers us a modern portrait of human condition. This trait of lack of cosmic order and justice is apparent in his major tragedies as well as some of the comedies. Thus, it becomes very clear that Shakespeare's approach to human life is existential in nature, especially when he portrays the character of Hamlet.

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Women's Health Problems and Fourier Transform: A Review: Ashwina Nilkanth Rangari

Women's Health Problems and Fourier Transform: A Review

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Abstract: Women's health has long been a concern for World Health Organization (WHO) but today it has become an urgent priority. It also highlights the interplay of biological and social determinants of women's health, and draws attention to the role of gender inequality in increasing exposure and vulnerability to risk, limiting access to health care and information, and impacting on health outcomes. While the paper calls for greater attention to health problems. Also draws attention to the consequences and costs of failing to address health issues at the appropriate points of women's lives. In a world with an ageing population, the challenge is to prevent and manage the risk factors of today to ensure that they do not lead to the chronic health problems of tomorrow.

Women's health matters not only to women themselves. It is also crucial to the health of the children they will bear. All children's are generally more dependent on their mother. So it is very important to take care of her health issues and give attention to women's health problems. After the age of 30s women suffer from various types of diseases like Uterine cysts, Breast Cancer, Heart disease and HIV/AIDS. To resolve these issues and health problems Fourier Transform can help a lot. The main aim of this paper is to discuss the various issues of women's health and how Fourier Transform is work useful for women's health problems.

Keywords: Fourier Transform, Uterine cysts, Uterine fibroids, Breast Cancer, Heart Rate Variability (HRV), Spectral Analysis, HIV/AIDS.

1. Introduction

Is a simple matter of biology, Women's health issues require separate and unique attention from the medical community. From menstruation through pregnancy, breast cancer and menopause, women's health demands attention to both the biological component of women, as well as the mental health requirements unique to women, including body image and violence issues.

The simple, yet unavoidable realities of menstruation, along with the unique ability of women to become pregnant, breast feed and eventually go through menopause requires the special attention of gynecologists and obstetricians as separate medical specialties. Generally referred to as an OB / GYN, these doctors are focused on the exclusive needs of women as they move throughout their lifespan. While pregnancy, for example, is a natural life process, the potential for complications to both mother and child means that continuing the circle of life has special medical changes and challenges that must be cared for in the medical profession.

Women are not unlike an artist's canvas, as they strive to capture forever the intrinsic and observable environments around us. Furthermore, they often represent the transitions that society is also facing in regards to economics, demographics and health and wellness issues. In addition to increased independence for the aging woman, thanks to the greatly acclaimed feminist movement, women have assumed a more independent role in directing their own destiny. Today more women are active professionally and personally, in politics and the business world. This new sense of independence has a very positive effect on the medical and mental health of today's women. A health mind does, indeed, produce a healthy body. Further, wherein a few years ago marathons and competitive sports were reserved mostly for men, women today are finally beginning to realize the benefits of keeping healthy through exercise and proper diet. Although further progress in needed women are finding that a healthy diet and exercise can increase longevity and produce a more fulfilling life as well.

Women's health matters not only to women themselves. It is also crucial to the health of the children they will bear. This underlines an important point. Paying due attention to the health of girls and women today is an investment not just for the present but also for future generations. This implies addressing the underlying social and economic determinants of women's health- including education, which directly benefit's women and is important for the survival, growth and development of their children [16].

2. Disparities between women and men

While women and men share many similar health challenges, the differences are such that the health of women deserves particular attention. Women generally live longer than men because of both biological and behavioural advantages. But in some settings, notably in parts of Asia, these advantages are overridden by gender-based discrimination so that female life expectancy at birth is lower than or equal to that of males.

Moreover, women's longer lives are not necessarily healthy lives. There are conditions that only women experience and whose potentially negative impact only they suffer. Some of these – such as pregnancy and childbirth – are not diseases, but biological and social processes that carry health risks and require health care. Some health challenges affect both women and men, but have a greater or different impact on women and so require responses that are tailored specifically to women's needs. Other conditions affect women and men more or less equally, but women face greater difficulties in getting the health care they need. Furthermore, gender-based inequalities – for example in education, income and employment – limit the ability of girls and women to protect their health [16].

3. Different issues related to women's health

3.1 Sexuality and reproduction are central to women's health

Women's health during the reproductive or fertile years (between the ages of 15 and 49 years) is relevant not only to women themselves, but also has an impact on the health and development of the next generation. Many of the health challenges during this period are ones that only young girls and women face. For example, complications of pregnancy and childbirth are the leading cause of death in young women aged between 15 and 19 years old in developing countries. Globally, the leading cause of death among women of reproductive age is HIV/AIDS. Girls and women are particularly vulnerable to HIV infection due to a combination of biological factors and gender-based inequalities, particularly in cultures that limit women's knowledge about HIV and their ability to protect themselves and negotiate safer sex [16].

3.2 Women's Health and Modern Society

Additionally, women's health issues have a psychological aspect that is exclusive to women. Much of modern society puts pressure on women to conform to a particular body image, one that may be physically damaging to the individual and may cause a negative body image. Furthermore, many women are physically vulnerable to issues such as domestic violence, which can greatly impact their health and quality of life.

4. Application of Fourier Transform in Various Women's Health Problem

4.1 What is Fourier Transform:-

Fourier transform is a mathematical tool. It converts a set of time domain data vectors into a set of frequency (or per time) domain vectors. Imagine you wanted to know about changes in soil temperature. Now suppose you to measure the temperature of the soil in your garden at dawn, midday, dusk and midnight, every day for a year. You would then have a list of real numbers representing the soil temperatures. Now if we plot these readings on a graph the vertical y-axis would be labeled 'temperature' and the horizontal x-axis would be labeled 'time' we get a so called 'time domain' graph. From the graph we infer the nights are cold and the days are warm and as it seems obvious, summer is warmer than winter. The graph you have just imagined is (roughly) the sum of two sinusoids (sine waves). One with a frequency of one day as the temperature varies between day and night, the other with a frequency of one year as the temperature varies with the season. The Fourier Transform provides a means of manipulating or transforming this raw data into an alternative set of data, the magnitude of which can be plotted on a graph with differently labeled axis. Never mind the y-axis label for now, but the x-axis would now be labeled 'frequency' or 'per time' this is in the 'frequency domain' graph.

This graph looks very different. It will consist of two vertical lines rising from the frequency axis, one at a frequency (or period) of one day, the other at a frequency (or period) of one year. Thus we have analyzed a seemingly complicated (rather than complex in the mathematical sense) set of data and extracted the most interesting facts from it: days are warmer than nights and summer is warmer than winter. There is still more information we can extract

from the raw results of the transform not shown on such a graph, we can look at the 'angle' information of the raw results which might tell us that the coldest day isn't January 1st, and midnight isn't the coldest part of the night.

We could use exactly the same Fourier Transform for more interesting purposes we could for example sample some music transform it, and plot the frequency spectrum to reproduce those dancing bars of LEDs on the stereo system. The mathematical formula for Fourier Transform is given as,

The Fourier transform with parameter s of $f(t)$ denoted by $F[f(t)] = F(s)$ and is given by

$$F[f(t)] = F(s) = \int_{-\infty}^{\infty} e^{-ist} f(t) dt, \text{ for parameter } s > 0.$$

4.2 Use of Fourier Transform in Uterine Cysts Treatment

In woman as the age increase various health problems are created which is related to menstrual cycle, Maternity period, Pregnancy, Problems of uterus, Breast cancer and Heart disease. 50-75% of the women in their late 30-50 years suffer from uterine cysts, 30% of them are of reproductive age. Out of the different kinds of uterine cysts formed, misdiagnosis often happens between the conditions of adenomyosis and uterine fibroids.

Uterine fibroids are masses of tissue grown in uterine wall. According to U. S. National Institute of Health, 20-25% of women of reproductive age have fibroids. Uterine fibroids are common in the middle-aged women who are in their 40's. A fibroid can be of any size varying from the size of a seed to that of a grapefruit. A fibroid can be present in any number and based on the location of occurrence fibroids are of different types. Generally, endometrial ablation is the treatment given to this condition. One way to diagnose these conditions is to perform surgery and examine the tissue under microscope. But without surgery, imaging technologies helps to examine uterine fibroids and adenomyosis. Uterus is imaged with ultrasound imaging modality due to its advantages of cost effective and most available method. In this way Fourier Transform is used for treatment of uterine cyst [2].

4.3 Use of Fourier Transform in Breast Cancer Treatment

Breast cancer is one of the most common malignant tumor among women throughout the world. Breast cancer affects one in four women. The risk of breast cancer and treatment failure increases with age [5]. Therefore, early detection and proper diagnostic methods have been known to help save women's lives. Conventional diagnostic techniques involve mammography, ultrasonography, magnetic resonance imaging (MRI), positron emission tomography (PET) and elastography, all of which have significant limitations related to efficacy, and can produce false positive or false negative results, therefore the search for new diagnostic technologies and therapeutic approaches is of critical importance. This involves the development of diagnostic tools to facilitate the detection of cancer cells, which is useful for assessing the efficacy of cancer therapies. One of the major challenges for chemotherapy is the lack of tools to monitor efficacy during the course of treatment. Vibrational spectroscopy appears to be a promising tool for such a purpose as it yields Fourier transformation infrared (FTIR) spectra which can be used to provide information on the chemical composition of the tissue. Fourier Transform Infrared (FT-IR) spectroscopy, coupled with PCA-LDA analysis, is a new technique to investigate the characteristics of serum in breast cancer [4], [6], [16].

4.4 Use of Fourier Transform in Measures Heart Rate Variation (HRV) and Spectral Analysis

The tool most commonly used in the frequency domain is spectral analysis, which consists of decomposing the Heart rate variation in a given period into its fundamental oscillatory components, defining them by their frequency and amplitude. One of the mathematical algorithms most commonly use to determine the number, frequency and amplitude of these components is the Fast Fourier transform, means Fourier Transform is used in the measurement and analysis of heart rate variation (HRV) as well as result of data for women and the relationship between aging and hormonal changes i.e. oral contraceptives and hormone replacement therapy, which contribute to modification of the autonomic control of the heart.

The incidence of cardiovascular diseases among premenopausal women is low when compared to that of men in the same age group, but increases significantly after this period [8]. In several countries, cardiovascular diseases are the major cause of morbidity and mortality among postmenopausal women, representing an important public health problem [12]. The increase in the incidence of cardiovascular events among middle-aged women has been associated with the hypoestrogenism typical of this period of women's lives [9].

4.5 Use of Fourier Transform in HIV/AIDS

Global women's health, whether in developed or developing countries, is in need of robust systems diagnostics for major diseases, such as HIV/AIDS, impacting the world populations. Fourier transform Infrared (FTIR) spectroscopy of serum is a quick and reagent-free methodology with which to analyze metabolic alterations such as those caused by disease or treatment.

In addition to immune system disruption, HIV infection is also known to cause metabolic abnormalities ranging from dyslipidemia, hyperglycemia, insulin resistance, and diabetes. Antiretroviral therapy (ART) can normalize CD4+ cell count leading to reduced morbidity and death in HIV-infected individuals but the continued use of ART can lead to metabolic disorders. HIV-1-infected individuals, and especially those on treatment, must be monitored constantly for the onset of metabolic complications in order to initiate interventions to delay and/or prevent severe metabolic diseases such as type II diabetes and heart disease.

The diagnosis and monitoring of metabolic abnormalities resulting from HIV infection and/or ART is based mainly on radioimmunoassay, dual-energy X-ray absorptiometry (DEXA), and numerous other colorimetric assays where one metabolite or a group of related metabolites are detected at a time. These methods are not only laborious to perform but also expensive and can be subjective. A possible solution to some of these issues is offered by metabolomics methodologies where multiple metabolites can be measured in a single analysis.

we explore the use of ATR-FTIR, a methodology incorporating a much simpler data collection approach, for the detection of metabolites in HIV-infected sera compared to negative controls with the secondary goal of determining whether metabolites detected in this manner would be comparable to those identified by NMR and MS-metabolomics and thus leading to the same conclusions.

Vibrational spectroscopic methods such as FTIR- and Raman spectroscopy are multipurpose techniques that offer advantages in simplicity, rapidity, low-cost, and minimal sample preparation [11].

5. Ten leading causes of death in females in the World

Rank	Cause	Death Percentage
1	Ischaemic heart disease	12.2
2	Stroke	11.1
3	Lower respiratory infections	7.3
4	COPD	5.1
5	Diarrhoeal diseases	3.8
6	HIV/AIDS	3.7
7	Diabetes mellitus	2.3
8	Prematurity and low birth weight	2.1
9	Neonatal infections	2.0

10	Hypertensive heart disease	1.9

5. Conclusions

This paper has summarized some of the health challenges faced by women across their lives and has given an overview of some of the underlying determinants of women's health. It is clear that women around the world face health challenges at every stage of their lives from early childhood to old age, and whether at home, at work or in wider society. The paper that follow look in greater detail at these challenges as they manifest at different stages of life and show how health systems are failing to respond adequately to women's needs.

Women's health matters not only to women themselves. It is also crucial to the health of the children they will bear. All children's are generally more dependent on their mother. So it is very important to take care of her health issues and give attention to women's health problems. In reviewing the evidence and setting an agenda for the future, this paper points the way towards the actions needed to better the health of girls and women around the world.

While this paper highlights differences between women and men, addressing women's health is a necessary and effective approach to strengthening health systems overall – action that will benefit everyone. After the age of 30s women suffer from various types of diseases like Uterine cysts, Breast Cancer, Heart disease, and HIV/AIDS. To resolve these issues and health problems Fourier Transform can help a lot.

Improving women's health matters to women, to their families, communities and societies at large.

Improve women's health – improve the world.

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Research Article

INTRODUCTION OF EXTENDED FINITE MELLIN TRANSFORM

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ABSTRACT

Integral transforms facilitate the conversion of complicated algebraic equations into simple and easily solvable expressions. In contrast to Fourier and Laplace transformations that were introduced to solve physical problems, Mellin transformation arose in a Mathematical, Physical and Engineering context. In the proposed work Extended Finite Mellin Transform is introduced.

Key Words:

Finite Mellin Transform, Testing function space, Generalized function, Operators.

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INTRODUCTION

In mathematics, the Mellin transform is an integral transform that may be regarded as the multiplicative version of the two-sided Laplace transform. This integral transform is closely connected to the theory of Dirichlet series, and is often used in number theory, mathematical statistics, and the theory of asymptotic expansion; it is closely related to the Laplace transform, Fourier transform, and the theory of the gamma function and allied special functions.

The Mellin transform is widely used in computer science for the analysis of algorithms because of its scale invariance property. The Mellin transform method is applied to fractional differential equations (Klimek and Dziembowski, 2008) [1]. It is also used in statistics for finding means, variances, skewness, and fuzzy numbers and then apply to the random coefficient autoregressive (RCA) time series models (Agapadoo, Thavaneswaran and Mandel, 2014) [2]. It is also useful for Geophysics. Integral transforms facilitate the conversion of complicated algebraic equations into simple and easily solvable expressions. Their application is now gaining a lot of importance in geophysics, particularly in the area of signal processing and the qualitative interpretation of potential field data. Despite the popularity of integral transform in geophysical data analysis, the Mellin transform has remained

virtually unexploited for geophysical applications (Mohan, Reddy and Ofoegbu, 1989) [3].

The main purpose of this paper is to generalized Finite Mellin transform in the distributional sense; Describing testing function spaces for Finite Mellin transform, Described some operators and properties for Finite Mellin transform. This paper is ordered as follows: Definitions are given in section 2, In section 3, Testing function spaces for Finite Mellin transform are given. The kernel of Finite Mellin transform is a member of testing function space is proved in section 4. In section 5, Definition of Distributional Generalized Finite Mellin transform is given. Some operators on testing function space are given in section 6. In section 7, Adjoint operators of Finite Mellin transform are given. Some properties of Generalized Finite Mellin transform are given in section 8. Lastly we conclude the paper.

The notation and terminology as per A. H. Zemanian [4], [5].

Definition: Finite Mellin Transform

The one Dimensional Finite Mellin transform with parameter s of $f(t)$ denoted by $M_s\{f(t)\} = F(s)$ performs a linear operation, given by the integral transform,

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$$M_f\{f(t)\} = F(s) = \int_0^a f(t)K(s)dt \quad (2.1)$$

$$\text{where the kernel } K(s) = \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right)$$

Testing Function Spaces for Finite Mellin Transform

The Space $M_{f,h,c,\alpha}$:

Let I be the open set in $\mathbb{R}_+ \times \mathbb{R}_+$ and E_+ denote the class of infinitely differentiable function defined on I , the space $M_{f,h,c,\alpha}$ is given by,

$$M_{f,h,c,\alpha} = \left\{ \phi : \phi \in E_+, \gamma_{h,c,\alpha}[\phi(t)] = \sup_t |\lambda_{h,c}(t)| t^{\alpha+1} D_t^\alpha \phi(t) \leq CA^\alpha q^\alpha \right\}$$

For each $q = 0, 1, 2, 3, \dots$ and where the constants A and C depend on the testing function ϕ .

The Space $M_{f,h,c,\alpha}^\nu$:

This space is a subspace of (3.1) which is given by,

$$M_{f,h,c,\alpha}^\nu = \left\{ \phi : \phi \in E_+, \gamma_{h,c,\alpha}[\phi(t)] = \sup_t |\lambda_{h,c}(t)| t^{\alpha-\nu} D_t^\alpha \phi(t) \leq C_\nu (a+\delta)^\nu q^\alpha \right\}$$

for any $\delta > 0$, where m is the constant depending on the function ϕ .

The space $M_{f,h,c,\alpha}^\nu$:

It is a negative space of (3.1) which is given by,

$$M_{f,h,c,\alpha}^\nu = \left\{ \phi : \phi \in E_+, \gamma_{h,c,\alpha}[\phi(t)] = \sup_t |\lambda_{h,c}(-t)| (-t)^\alpha D_t^\alpha \phi(t) \leq CA^\alpha q^\alpha \right\}$$

Here we also set

$$\lambda_{h,c}(t) = \begin{cases} t^{1+h}, & 0 < t < 1 \\ t^{1+c}, & 1 < t < a \end{cases}$$

Lemma

The function $\left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right)$ is a member of $M_{f,h,c,\alpha}$ if

$-b < \text{Re } s \leq b$ for any real number c .

$$\text{Proof: Let } \phi(t) = \frac{a^{2s+1}}{t^{s+1}} - t^s$$

$$\begin{aligned} \text{Consider, } \gamma_{h,c,\alpha}[\phi(t)] &= \sup_t |\lambda_{h,c}(t)| t^{\alpha+1} D_t^\alpha \phi(t) \\ &= \sup_t \left| \lambda_{h,c}(t) t^{\alpha+1} D_t^\alpha \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) \right| \end{aligned}$$

$$= \sup_t \left| \lambda_{h,c}(t) t^{\alpha+1} [P(-s-q)t^{-s-q-1}a^{2s+1} - P(s-q+1)t^{s-2}] \right|$$

where P is a polynomial in s and q .

$$= \sup_t |a^{2s+1}P(-s-q)t^{b-s} - P(s-q+1)t^{b+s+1}| \leq C \quad (4.1)$$

where

$$C = \sup_t \left\{ a^{2s+1}P(-s-q)t^{b-s} - P(s-q+1)t^{b+s+1} \right\}$$

$$\therefore \gamma_{h,c,\alpha}[\phi(t)] \leq C \frac{q^\alpha}{q^\alpha} \text{ by (4.1)}$$

$$= CA^\alpha q^\alpha, \text{ where } A = \frac{1}{q^\alpha}$$

$$\text{Hence } \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) \in M_{f,h,c,\alpha}.$$

Here onwards for simplicity we say that $\phi(t) \in M_{f,h,c,\alpha}$ if

$|\lambda_{h,c}(t)| t^{\alpha+1} D_t^\alpha \phi(t) < \infty$, for $q = 0, 1, 2, 3, \dots$. If as $t \rightarrow 0$, $b-s > 0$ and $b+s > 0$,

i.e. $b > \text{Re } s$ $s > -b$, i.e. $\text{Re } s < b$ $-b < \text{Re } s$

i.e. if $-b < \text{Re } s \leq b$ and for any real number c .

Thus $\phi(t) \in M_{f,h,c,\alpha}$ if $-b < \text{Re } s \leq b$, for any real number c .

Distributional Generalized Finite Mellin Transform ($M_f T$)

For $f(t) \in F_{f,h,c,\alpha}^*$, where $F_{f,h,c,\alpha}^*$ is the dual space of $M_{f,h,c,\alpha}$ and $-b < \text{Re } s < b$. The distributional Finite Mellin transform is defined as,

$$M_f\{f(t)\} = F(s) = \langle f(t), \phi(t,s) \rangle, \quad (5.1)$$

where $\phi(t,s) = \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right)$ and for each fixed t

$(0 < t < a)$. The right hand side of (5.1) is meaningful because according to lemma 4, $\phi(t,s) \in M_{f,h,c,\alpha}$ and $f(t) \in M_{f,h,c,\alpha}^*$.

Operators on the Space $M_{f,h,c,\alpha}$

Proposition: If $\phi(t) \in M_{f,h,c,\alpha}$ and σ is any fixed real number then $\phi(t+\sigma) \in M_{f,h,c,\alpha}$, $t+\sigma > 0$ and $\phi(t+\sigma) \in M_{f,h,c,\alpha}^\nu$, $t+\sigma < 0$.

Proof

$$\gamma_{h,c,\alpha}[\phi(t+\sigma)] = \sup_t |\lambda_{h,c}(t)| t^{\alpha+1} D_t^\alpha \phi(t+\sigma)$$

Consider,

$$= \sup_t |\lambda_{h,c}(t')| (t'-\sigma)^{\alpha+1} D_{t'}^\alpha \phi(t')$$

Where $t' = t + \sigma$ $\therefore t = t' - \sigma$

$$\leq CA^\alpha q^\alpha$$

Thus, $\phi(t+\sigma) \in M_{f,h,c,\alpha}$ for $t+\sigma > 0$.

Similarly it can show that $\phi(t+\sigma) \in M_{f,h,c,a}^u$ for $t+\sigma < 0$.

Proposition: The translation (Shifting) operator $\sigma: \phi(t) \rightarrow \phi(t+\sigma)$ is a topological automorphism on $M_{f,h,c,a}$, for $t+\sigma > 0$, and it is a topological isomorphism from $M_{f,h,c,a}$ onto $M_{f,h,c,a}^u$, for $t+\sigma < 0$.

Proposition: If $\phi(t) \in M_{f,h,c,a}$ and $r > 0$, strictly positive number, then $\phi(rt) \in M_{f,h,c,a}$.

Proof: Consider, $\gamma_{h,c,a}\phi(rt) = \sup_i |\lambda_{h,c}(t) r^{q+1} D_i^q \phi(rt)|$

$$= \sup_i \left| \lambda_{h,c} \left(\frac{T}{r} \right) \left(\frac{T}{r} \right)^{q+1} D_i^q \phi \left(T \right) \right|,$$

Where, $rt = T \therefore t = \frac{T}{r}$

$$= M \sup_i \left| \lambda_{h,c}(T) (T)^{q+1} D_i^q \phi(T) \right|,$$

Where, M is a constant depending on r .

$$\leq M C A^q q^{q\alpha} \leq C' A^q q^{q\alpha}$$

where $C' = MC$.

Thus, $\phi(rt) \in M_{f,h,c,a}$, for $r > 0$.

Proposition: If $r > 0$ and $\phi(t) \in M_{f,h,c,a}$ then the scaling operator $R: M_{f,h,c,a} \rightarrow M_{f,h,c,a}$ defined by $R\phi = \psi$ where $\psi(t) = \phi(rt)$ is a topological automorphism.

Proposition: The operator $\phi(t) \rightarrow D_i \phi(t)$ is defined on the space $M_{f,h,c,a}$ and transforms this space into itself.

Proof: Let $\phi(t) \in M_{f,h,c,a}$, If $D_i \phi(t) = \psi(t)$, we have

$$\begin{aligned} \gamma_{h,c,a}\psi(t) &= \sup_i |\lambda_{h,c}(t) r^{q+1} D_i^q D_i \phi(t)| \\ &= \sup_i |\lambda_{h,c}(t) r^{q+1} D_i^q D_i \phi(t)| \\ &= \sup_i |\lambda_{h,c}(t) r^{q+1} D_i^{q+1} \phi(t)| \\ &\leq C A^{q+1} (q+1)^{(q+1)\alpha}, \end{aligned}$$

$q = 0, 1, 2, 3, \dots$

Therefore, $\psi(t) \in M_{f,h,c,a}$ i.e. $D_i \phi(t) \in M_{f,h,c,a}$.

$$\text{i.e. } \gamma_{h,c,a}(D_i \phi(t)) = \gamma_{h,c,a(q+1)}(\phi(t)).$$

Proposition: The differential operator of M_f -type $M_f: \phi(t) \rightarrow D_i \phi(t)$ is a topological automorphism on $M_{f,h,c,a}$.

Proposition: For $m = (m_1, m_2)$, where $m_1, m_2 = 0, 1, 2, \dots$, if $\phi(t) \in M_{f,h,c,a}$. Then $\psi(t) \in M_{f,h,c,a}$ where $\psi(t) = D^m \phi(t)$. Further the mapping $\Delta = D^m \phi: \phi \rightarrow \psi$ is one-one, linear and continuous.

Proof: For $\psi \in M_{f,h,c,a}$,

$$\begin{aligned} \gamma_{h,c,a}\psi(t) &= \sup_i |\lambda_{h,c}(t) r^{q+1} D_i^q \psi(t)| \\ &= \sup_i |\lambda_{h,c}(t) r^{q+1} D_i^q D^m \phi(t)| \\ &= \sup_i |\lambda_{h,c}(t) r^{q+1} D_i^{q+m} \phi(t)| \leq C A^q q^{q\alpha} \end{aligned}$$

(6.7.1)

Thus $\psi(t) \in M_{f,h,c,a}$ if $\phi(t) \in M_{f,h,c,a}$.

It is obviously linear. It is injective for, if $D^m \phi = 0$ then $\phi = c$, c is a constant. If $c = 0$ then $\phi = 0$ and D is injective. But if $c \neq 0$ then,

$$\sup_i |\lambda_{h,c}(t) r^{q+1} D_i^q c| = \sup_i |\lambda_{h,c}(t) r^{q+1} c|, \text{ for } q = 0.$$

As the right hand side is not bounded we conclude that $\phi \notin M_{f,h,c,a}$, which is a contradiction. Hence ' c ' must be zero and therefore $\phi = 0$. For continuity we observe from equation (6.7.1) that,

$$\gamma_{h,c,a}(D^m \phi) \leq M \gamma_{h,c,a}(\phi),$$

Where M is some constant. Thus the theorem is proved.

Proposition: For $\tau \in \mathbb{R}$ and $\phi(t) \in M_{f,h-c,a,a}$,

$$E(\phi) = \psi(t) = e^{-\tau t} \phi(t) \in M_{f,h-c,a,a}.$$

Proof: Let $\phi(t) \in M_{f,h-c,a,a}$.

$$\begin{aligned} \text{Consider, } \gamma_{h,c,a}\psi(t) &= \sup_i |\lambda_{h,c}(t) r^{q+1} D_i^q e^{-\tau t} \phi(t)| \\ &= \sup_i \left| \sum_{j=0}^i b_j e^{-\tau t} \lambda_{h,c}(t) r^{q+1} D_i^q \phi(t) \right| \\ &= \sup_i \left| \sum_{j=0}^i b_j e^{-\tau t} t^{j+q+1} D_i^q \phi(t) \right| \\ &\leq C A^q q^{q\alpha}. \end{aligned}$$

Thus $\psi(t) \in M_{f,h,c,a}$ if $\phi(t) \in M_{f,h-c,a,a}$.

Adjoint Operators of Fourier-Finite Mellin Transform

Proposition: The adjoint shifting operator is a continuous function from $M_{f,h,s,a}^*$ to $M_{f,h,s,a}^*$. The adjoint operator $f(t) \rightarrow f(t-\sigma)$ leads to the operation transform formula $M_f\{f(t-\sigma)\} = RM_f\{f(t)\}$.

Proof:

$$M_f\{f(t-\sigma)\} = \left\langle f(t-\sigma), \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) \right\rangle$$

$$= \left\langle f(t), \left(\frac{a^{2s+1}}{(t+\sigma)^{s+1}} - (t+\sigma)^s \right) \right\rangle$$

$$= \left\langle f(T-\sigma), \left(\frac{a^{2s+1}}{T^{s+1}} - T^s \right) \right\rangle \quad \text{where } t+\sigma = T, \quad t = T-\sigma$$

$$= R \left\langle f(t), \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) \right\rangle,$$

where, R is constant depending on σ .

$$= RM_f\{f(t)\}$$

$$\therefore M_f\{f(t-\sigma)\} = RM_f\{f(t)\}.$$

Proposition: The adjoint scaling operator is a continuous function from $M_{f,h,s,a}^*$ to $M_{f,h,s,a}^*$. The adjoint operator $f(t) \rightarrow \frac{1}{q}f\left(\frac{t}{q}\right)$ corresponding transform formula is

$$M_f\left\{\frac{1}{q}f\left(\frac{t}{q}\right)\right\} = QM_f\{f(t)\}.$$

$$M_f\left\{\frac{1}{q}f\left(\frac{t}{q}\right)\right\} = QM_f\{f(t)\}.$$

Proof:

$$M_f\left\{\frac{1}{q}f\left(\frac{t}{q}\right)\right\} = \left\langle \frac{1}{q}f\left(\frac{t}{q}\right), \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) \right\rangle$$

$$= \left\langle f(t), \left(\frac{a^{2s+1}}{(tq)^{s+1}} - (tq)^s \right) \right\rangle$$

$$= \left\langle f\left(\frac{T}{q}\right), \left(\frac{a^{2s+1}}{T^{s+1}} - T^s \right) \right\rangle \quad \text{where } tq = T, \quad t = \frac{T}{q}$$

$$= Q \left\langle f(t), \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) \right\rangle \quad \text{where, } Q \text{ is a constant depending on } q.$$

$$= QM_f\{f(t)\}.$$

$$\therefore M_f\left\{\frac{1}{q}f\left(\frac{t}{q}\right)\right\} = QM_f\{f(t)\}$$

Properties of Generalized Finite Mellin Transform

Linearity Property: If $M_f\{f(t)\}$ is generalized finite mellin transform of $f(t)$ and $M_f\{g(t)\}$ is generalized finite mellin transform of $g(t)$ then

$$M_f\{C_1f(t) + C_2g(t)\}(s) = C_1M_f\{f(t)\}(s) + C_2M_f\{g(t)\}(s)$$

Proof: Consider,

$$M_f\{C_1f(t) + C_2g(t)\}(s) = \int_0^a \left[C_1f(t) + C_2g(t) \right] \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) dt$$

$$= C_1 \int_0^a \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) f(t) dt + C_2 \int_0^a \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) g(t) dt$$

$$= C_1M_f\{f(t)\}(s) + C_2M_f\{g(t)\}(s)$$

$$\text{Result: } M_f\{f(at)\}(s) = a^{-s-1}M_{f,as}\{f(t)\}(s)$$

$$\text{Proof: } M_f\{f(at)\}(s) = \int_0^a \left(\frac{a^{2s+1}}{t^{s+1}} - t^s \right) f(at) dt = \int_0^a \left(a^{2s+1} t^{-s-1} - t^s \right) f(at) dt$$

$$\text{Put } at = T \quad \therefore t = \frac{T}{a} \quad \Rightarrow a dt = dT \quad \therefore dt = \frac{dT}{a}$$

$$= \int_0^a \left(a^{2s+1} \left(\frac{T}{a} \right)^{-s-1} - \left(\frac{T}{a} \right)^s \right) f(T) \frac{dT}{a}$$

$$= \frac{1}{a} \int_0^a \left(a^{2s+1} \frac{T^{-s-1}}{a^{-s-1}} - \frac{T^s}{a^s} \right) f(T) dT$$

$$= \frac{1}{a} \int_0^a \frac{1}{a^s} \left(a^{2s+1} a^{s+1} \frac{T^{-s-1}}{a^{-s-1}} - a^s \frac{T^s}{a^s} \right) f(T) dT$$

$$= \frac{1}{a^{s+1}} \int_0^a \left(a^{2s+1} a^{s+1} T^{-s-1} - T^s \right) f(T) dT$$

$$= a^{-s-1} \int_0^a \left(a^{2s+1} T^{-s-1} - T^s \right) f(T) dT$$

$$= \alpha^{-s-1} \int_0^{\infty} \left((a\alpha)^{2s+1} T^{-s-1} - T^s \right) f(T) dT$$

$$= \alpha^{-s-1} \int_0^{\infty} \left(\frac{(a\alpha)^{2s+1}}{T^{s+1}} - T^s \right) f(T) dT$$

$$= \alpha^{-s-1} M_{f,aa} \{f(t)\}(s).$$

$$\therefore M_f \{f(a\alpha)\}(s) = \alpha^{-s-1} M_{f,aa} \{f(t)\}(s)$$

CONCLUSIONS

In the present work generalized Finite Mellin transform is introduced in the distributional sense. Some operators and Adjoint operators for Finite Mellin transform is obtained which will be useful for solving Partial differential equations.

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ANALYTICAL BEHAVIOUR OF DISTRIBUTIONAL TWO DIMENSIONAL FOURIER-LAPLACE TRANSFORM

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ABSTRACT

In the literature there are several integral transforms and each are suitable for different type of differential equations. The two dimensional Fourier Transform is one of the most fundamental advances in scientific computing, Mathematical and Statistics problems. On the same lines two dimensional Laplace transform is applied as a frequency-domain tool for analysis and synthesis of communication systems.

In this paper, the aim is to study the possible extension of two dimensional Fourier-Laplace transform in the distributional sense. For this purpose, some testing function spaces by using Gelfand-Shilov technique are developed in order to discuss analytical behaviour of distributional two dimensional Fourier-Laplace transform.

Keywords: Fourier Transform, Laplace Transform, Two Dimensional Fourier-Laplace Transform, Generalized function.

INTRODUCTION

Integral transforms are widely used in various fields. The application of the Fourier transform (FT) to nuclear magnetic resonance spectroscopy and its extension to multiple dimensions has radically altered modern chemistry and medical science; NMR imaging and the determination of the structure of proteins in solution are two important advances that would be either impossible or orders of magnitude more difficult without the Fourier transform [1].

Similarly, Laplace transform method was used to obtain the explicit solution of a certain kind of ordinary differential equations with fractional derivatives and to construct the solution of linear non-homogeneous fractional order differential equations. By using Laplace transform, one can

also find the exact solution of time fractional partial differential equation and some fractional order integral and integro-differential equations [2]. Moreover system of partial differential equations related to telegraph equation is solved by using the double transformation [3].

On studying the vast applications of these Fourier transform and Laplace transform we developed new transform i.e. two dimensional Fourier-Laplace transform and this may give remarkable and extra ordinary applications in various fields of science and engineering. The main aim of this paper is to discuss the analytical behavior of distributional two dimensional Fourier-Laplace transform for this purpose here is the necessity to describe some testing function spaces and which are as follows:

1.1. The Space $FL_{\alpha, \beta, \gamma, \delta, \epsilon}$ (S_α -type space):

Let I be the open set in $R_s \times R_n$ and E_s denotes the class of infinitely differentiable function defined on I , the space $FL_{\alpha, \beta, \gamma, \delta, \epsilon}$ is given by,

$$FL_{\alpha, \beta, \gamma, \delta, \epsilon} = \left\{ \phi : \phi \in E_s / \gamma_{\alpha, \beta, \gamma, \delta, \epsilon}[\phi(t, x, y)] \right. \\ \left. = \sup_{I_1} \left| t^\alpha x^\beta K_{\alpha, \beta}(x) R_{\gamma, \delta}(y) D_t^\alpha D_x^\beta D_y^\gamma [\phi(t, x, y)] \right| \leq C_{1, \alpha, \beta, \gamma, \delta, \epsilon} A^\alpha B^\beta C^\gamma \right\}$$

where the constants A, B and $C_{1, \alpha, \beta, \gamma, \delta, \epsilon}$ depend on the testing function ϕ .

1.1. The Space $FL_{\alpha, \beta, \gamma, \delta, \epsilon}^p$ (S_α^p -type space):

It is given by,

$$FL_{a,b,c,d,e}^B = \left\{ \phi : \phi \in E_a / \rho_{a,b,c,d,e,f,g,h,i,j,k} \left[\phi(t, z, x, y) \right] \right. \\ \left. = \sup_{I_1} \left| t^a z^b K_{a,b}(x) R_{c,d}(y) D_1^f D_2^g D_3^h D_4^i D_5^j D_6^k \left[\phi(t, z, x, y) \right] \right| \leq C A^a k^{b\alpha} B^c r^{\alpha\beta} G^d t^{\beta\gamma} H^e n^{\alpha\delta} \right\}$$

where the constants A, B, G, H and C depend on the testing function ϕ .

1.3. The space $FL_{a,b,c,d,e,f} (S_r$ -type space):

This space is given by,

$$FL_{a,b,c,d,e,f} = \left\{ \phi : \phi \in E_a / \xi_{a,b,c,d,e,f,g,h,i,j,k} \left[\phi(t, z, x, y) \right] \right. \\ \left. = \sup_{I_1} \left| t^a z^b K_{a,b}(x) R_{c,d}(y) D_1^f D_2^g D_3^h D_4^i D_5^j D_6^k \left[\phi(t, z, x, y) \right] \right| \leq C_{a,b,c,d,e,f} A^a q^{\alpha\gamma} B^b m^{\alpha\delta} \right\}$$

where the constants A, B and $C_{a,b,c,d,e,f}$ depend on the testing function ϕ .

In the present paper, two dimensional Fourier-Laplace transform is generalized in distributional sense and discuss its analytical behaviour as given. The plan of the paper is as follows: In section 2, definition of two dimensional Fourier-Laplace transform is given. Definition of Distributional generalized two dimensional Fourier-Laplace transform is given in section 3. In section 4 we have proved Analyticity theorem. Lastly we conclude the paper.

The notations and terminology as per A. H. Zemanian [4], [5].

2. Definitions

The Two Dimensional Fourier-Laplace transform with parameters s, u, p, v , of $f(t, z, x, y)$ is denoted by $FL\{f(t, z, x, y)\} = F(s, u, p, v)$ performs a linear operation, given by the integral transform,

$$FL\{f(t, z, x, y)\} = F(s, u, p, v) = \int_{-\infty}^{\infty} \int_{-\infty}^{\infty} \int_0^{\infty} \int_0^{\infty} f(t, z, x, y) K(s, u, p, v) dt dz dx dy \quad (2.1)$$

where the kernel $K(s, u, p, v) = e^{-i[(u+u\tau)-i(p\sigma+v\eta)]}$

3. Distributional Generalized Two Dimensional Fourier-Laplace Transform (2DGLT)

For $f(t, z, x, y) \in FL_{a,b,c,d,e}^*$, where $FL_{a,b,c,d,e}^*$ is the dual space of $FL_{a,b,c,d,e}$. It contains all distributions of compact support. The distributional two dimensional Fourier-Laplace transform is a function of $f(t, z, x, y)$ is defined as,

$$FL\{f(t, z, x, y)\} = F(s, u, p, v) = \langle f(t, z, x, y), \phi(t, z, x, y, s, u, p, v) \rangle, \quad (3.1)$$

where $\phi(t, z, x, y, s, u, p, v) = e^{-i[(u+u\tau)-i(p\sigma+v\eta)]}$ and for each fixed $t(0 < t < \infty)$, $z(0 < z < \infty)$, $x(0 < x < \infty)$ and $y(0 < y < \infty)$. Also $s > 0$, $u > 0$, $p > 0$ and $v > 0$. The right hand side of (3.1) has a sense as an application of $f(t, z, x, y) \in FL_{a,b,c,d,e}^*$ to $\phi(t, z, x, y, s, u, p, v) \in FL_{a,b,c,d,e}$.

4. Analyticity Theorem

Let $f(t, z, x, y) \in FL_{a,b,c,d,e}^*$ and its two dimensional Fourier-Laplace transform $F(s, u, p, v)$ is defined by $FL\{f(t, z, x, y)\} = F(s, u, p, v) = \langle f(t, z, x, y), e^{-i[(u+u\tau)-i(p\sigma+v\eta)]} \rangle$.

Then $F(s, u, p, v)$ is analytic for some fixed $s > 0$, $u > 0$, $p > 0$, $v > 0$,

$$\frac{\partial}{\partial s} \frac{\partial}{\partial u} F(s, u, p, v) = \left\langle f(t, z, x, y), \frac{\partial}{\partial s} \frac{\partial}{\partial u} \phi(t, z, x, y, s, u, p, v) \right\rangle \quad (4.1)$$

$$\text{and } \frac{\partial}{\partial p} \frac{\partial}{\partial v} F(s, u, p, v) = \left\langle f(t, z, x, y), \frac{\partial}{\partial p} \frac{\partial}{\partial v} \phi(t, z, x, y, s, u, p, v) \right\rangle \quad (4.2)$$

$$\text{where } \phi(t, z, x, y, s, u, p, v) = e^{-i\| (st+uz) - i(px+vy) \|}.$$

Proof: Let $s, u,$ and p, v be an arbitrary but fixed. Choose the real positive numbers a_1, b_1 and r such that $\sigma_1 < a_1 < s-r < s+r < b_1 < \sigma_2$. Also let Δs be a complex increment such that $0 < |\Delta s| < r$.

For $\Delta s \neq 0$, we write

$$\begin{aligned} \frac{F(s+\Delta s, u, p, v) - F(s, u, p, v)}{\Delta s} &= \left\langle f(t, z, x, y), \frac{\partial}{\partial s} e^{-i\| (st+uz) - i(px+vy) \|} \right\rangle \\ &= \frac{1}{\Delta s} \left\langle \left\langle f(t, z, x, y), e^{-i\| (s+\Delta s)t + iuz - i(px+vy) \|} \right\rangle - \left\langle f(t, z, x, y), e^{-i\| st + iuz - i(px+vy) \|} \right\rangle \right\rangle \\ &\quad - \left\langle f(t, z, x, y), \frac{\partial}{\partial s} e^{-i\| st + iuz - i(px+vy) \|} \right\rangle \\ &= \frac{1}{\Delta s} \left\langle \left\langle f(t, z, x, y), e^{-i\| uz - i(px+vy) \|} \left[e^{-i\| (s+\Delta s)t - iux \|} - e^{-i\| st - iux \|} \right] \right\rangle - \left\langle f(t, z, x, y), \frac{\partial}{\partial s} e^{-i\| st + iuz - i(px+vy) \|} \right\rangle \right\rangle \\ &= \left\langle f(t, z, x, y), \frac{e^{-i\| uz - i(px+vy) \|}}{\Delta s} \left[e^{-i\| (s+\Delta s)t - iux \|} - e^{-i\| st - iux \|} \right] - \frac{\partial}{\partial s} e^{-i\| st + iuz - i(px+vy) \|} \right\rangle \\ &= \left\langle f(t, z, x, y), \psi_{\Delta s}(t, z, x, y) \right\rangle \quad (4.3) \end{aligned}$$

$$\text{where } \psi_{\Delta s}(t, z, x, y) = \frac{e^{-i\| uz - i(px+vy) \|}}{\Delta s} \left[e^{-i\| (s+\Delta s)t - iux \|} - e^{-i\| st - iux \|} \right] - \frac{\partial}{\partial s} e^{-i\| st + iuz - i(px+vy) \|}$$

To prove $\psi_{\Delta s}(t, z, x, y) \in FL_{s, b, c, d, e}$, we shall show that as $|\Delta s| \rightarrow 0$, $\psi_{\Delta s}(t, z, x, y)$ converges in $FL_{s, b, c, d, e}$ to zero.

To proceed, let C denotes the circle with centre at s and radius r_1 , where $0 < r < r_1 < \min\{s-a_1, b_1-s\}$.

We may interchange differentiation on s with differentiation on t and by using Cauchy's integral formula,

$$\begin{aligned} (-D_t)^j \psi_{\Delta s}(t, z, x, y) &= \frac{e^{-i\| uz - i(px+vy) \|}}{\Delta s} \left[(-i)^j (s+\Delta s)^j e^{-i\| (s+\Delta s)t - iux \|} - (-i)^j s^j e^{-i\| st - iux \|} \right] \\ &\quad - \frac{\partial}{\partial s} (-is)^j e^{-i\| st + iuz - i(px+vy) \|} \end{aligned}$$

Now applying Cauchy's integral formula,

$$\begin{aligned} (-D_t)^j \psi_{\Delta s}(t, z, x, y) &= \frac{(-i)^j e^{-i\| uz - i(px+vy) \|}}{\Delta s} \left\{ \frac{1}{2\pi i} \int_C \left[\frac{\xi^j e^{-i\| \xi t - iux \|}}{\xi - s - \Delta s} - \frac{\xi^j e^{-i\| \xi t - iux \|}}{\xi - s} \right] d\xi \right\} \\ &\quad - (-i)^j e^{-i\| uz - i(px+vy) \|} \cdot \frac{1}{2\pi i} \int_C \frac{\xi^j e^{-i\| \xi t - iux \|}}{(\xi - s)^2} d\xi \\ &= \frac{e^{-i\| uz - i(px+vy) \|}}{2\pi i \Delta s} \int_C \left[\frac{1}{\xi - s - \Delta s} - \frac{1}{\xi - s} \right] (-i)^j \xi^j e^{-i\| \xi t - iux \|} d\xi - (-i)^j e^{-i\| uz - i(px+vy) \|} \cdot \frac{1}{2\pi i} \int_C \frac{\xi^j e^{-i\| \xi t - iux \|}}{(\xi - s)^2} d\xi \end{aligned}$$

(34)

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$$\begin{aligned} &= \frac{e^{-i[\omega t - i(\mu x + \nu y)]}}{2\pi i \Delta s} \int_C \left[\frac{\xi - s - \xi + s + \Delta s}{(\xi - s - \Delta s)(\xi - s)} \right] (-i)^j \xi^j e^{-i\phi_j} d\xi - (-i)^j e^{-i[\omega t - i(\mu x + \nu y)]} \cdot \frac{1}{2\pi i} \int_C \frac{\xi^j e^{-i\phi_j}}{(\xi - s)^2} d\xi \\ &= \frac{e^{-i[\omega t - i(\mu x + \nu y)]}}{2\pi i} \int_C \left[\frac{1}{(\xi - s - \Delta s)(\xi - s)} - \frac{1}{(\xi - s)^2} \right] (-i\xi)^j e^{-i\phi_j} d\xi \\ &= \frac{e^{-i[\omega t - i(\mu x + \nu y)]}}{2\pi i} \int_C \left[\frac{\xi - s - \xi + s + \Delta s}{(\xi - s - \Delta s)(\xi - s)^2} \right] (-i\xi)^j e^{-i\phi_j} d\xi \\ &= \frac{e^{-i[\omega t - i(\mu x + \nu y)]}}{2\pi i} \Delta s \int_C \frac{(-i\xi)^j e^{-i\phi_j}}{(\xi - s - \Delta s)(\xi - s)^2} d\xi \end{aligned}$$

$$D_t^j D_z^k \psi_{\Delta s, \Delta s}(t, z, x, y) = \frac{(-i\mu)^n e^{-i[\omega t - i(\mu x + \nu y)]} \Delta s}{2\pi i} \int_C \frac{(-i\xi)^j e^{-i\phi_j}}{(\xi - s - \Delta s)(\xi - s)^2} d\xi$$

Now for all $\xi \in C$ and $-\infty < t < \infty$, $\sup_{I_1} \left| t^k z^r K_{\alpha, \beta}(x) R_{\gamma, \delta}(y) u^n e^{-i[\omega t - i(\mu x + \nu y)]} \right| \leq K$,

Where K is a constant independent of ξ and t . Moreover, $|\xi - s - \Delta s| > r_1 - r > 0$ and $|\xi - s| = r_1$,

$C_1 = \max \{ |\xi|^j, \xi \in C \}$, consequently

$$\begin{aligned} &\sup_{I_1} \left| t^k z^r K_{\alpha, \beta}(x) R_{\gamma, \delta}(y) D_t^j D_z^k \psi_{\Delta s, \Delta s}(t, z, x, y) \right| \\ &= \sup_{I_1} \left| t^k z^r K_{\alpha, \beta}(x) R_{\gamma, \delta}(y) \frac{(-i\mu)^n e^{-i[\omega t - i(\mu x + \nu y)]} \Delta s}{2\pi i} \int_C \left[\frac{(-i\xi)^j e^{-i\phi_j}}{(\xi - s - \Delta s)(\xi - s)^2} \right] d\xi \right| \\ &\leq \sup_{I_1} \left| t^k z^r K_{\alpha, \beta}(x) R_{\gamma, \delta}(y) \frac{(u)^n e^{-i[\omega t - i(\mu x + \nu y)]}}{2\pi} \left| \Delta s \int_C \frac{|\xi|^j |e^{-i\phi_j}|}{|\xi - s - \Delta s| |\xi - s|^2} |d\xi| \right| \right| \\ &\leq \frac{|\Delta s|}{2\pi} \int_C \frac{KC_1}{(r_1 - r)r_1^2} |d\xi| \\ &\leq \frac{|\Delta s|}{2\pi} \int_C \frac{C_2}{(r_1 - r)r_1^2} |d\xi|, \text{ where } C_2 = KC_1 \\ &\leq \frac{|\Delta s| C_2}{2\pi(r_1 - r)r_1^2} \cdot 2\pi r_1, \\ &\leq \frac{|\Delta s| C_2}{(r_1 - r)r_1}. \end{aligned}$$

The right hand side is independent of t and converges to zero as $|\Delta s| \rightarrow 0$. This shows that $\psi_{\Delta s, \Delta s}(t, z, x, y)$ converges to zero.

Now, let s, u , and p, v be an arbitrary but fixed points. Choose the real positive numbers a_2, b_2 and h such that $\sigma_2 < a_2 < \operatorname{Re} p - h < \operatorname{Re} p + h < b_2 < \sigma_2$. Also let Δp be a complex increment such that $0 < |\Delta p| < h$.

For $\Delta p \neq 0$, we write

$$\begin{aligned} \frac{F(s, u, p + \Delta p, v) - F(s, u, p, v)}{\Delta p} &= \left\langle f(t, z, x, y), \frac{\partial}{\partial p} e^{-i[(st+u\tau)-i(px+vy)]} \right\rangle \\ &= \frac{1}{\Delta p} \left\langle f(t, z, x, y), e^{-i[(st+u\tau)-i(p+\Delta p)x+vy]} \right\rangle - \left\langle f(t, z, x, y), e^{-i[(st+u\tau)-i(px+vy)]} \right\rangle \\ &\quad - \left\langle f(t, z, x, y), \frac{\partial}{\partial p} e^{-i[(st+u\tau)-i(px+vy)]} \right\rangle \\ &= \frac{1}{\Delta p} \left\langle f(t, z, x, y), e^{-i[(st+u\tau)-i\tau y]} \left[e^{-(p+\Delta p)x} - e^{-px} \right] \right\rangle - \left\langle f(t, z, x, y), \frac{\partial}{\partial p} e^{-i[(st+u\tau)-i(px+vy)]} \right\rangle \\ &= \left\langle f(t, z, x, y), \frac{e^{-i[(st+u\tau)-i\tau y]}}{\Delta p} \left[e^{-(p+\Delta p)x} - e^{-px} \right] - \frac{\partial}{\partial p} e^{-i[(st+u\tau)-i(px+vy)]} \right\rangle \\ &= \langle f(t, z, x, y), \psi_{\Delta p}(t, z, x, y) \rangle \end{aligned} \quad (4.4)$$

$$\text{where } \psi_{\Delta p}(t, z, x, y) = \frac{e^{-i[(st+u\tau)-i\tau y]}}{\Delta p} \left[e^{-(p+\Delta p)x} - e^{-px} \right] - \frac{\partial}{\partial p} e^{-i[(st+u\tau)-i(px+vy)]}$$

To prove $\psi_{\Delta p}(t, z, x, y) \in FL_{\sigma, h, \delta, \delta, \delta}$, we shall show that as $|\Delta p| \rightarrow 0$, $\psi_{\Delta p}(t, z, x, y)$ converges in $FL_{\sigma, h, \delta, \delta, \delta}$ to zero.

To proceed, let C_1 denotes the circle with centre at p and radius h_1 , where $0 < r < h_1 < \min(p - a_2, b_2 - p)$. We may interchange differentiation on p with differentiation on x and by using Cauchy's integral formula,

$$\begin{aligned} (-D_x)^q \psi_{\Delta p}(t, z, x, y) &= \frac{e^{-i[(st+u\tau)-i\tau y]}}{\Delta p} \left[(p + \Delta p)^q e^{-(p+\Delta p)x} - (p)^q e^{-px} \right] \\ &\quad - \frac{\partial}{\partial p} (p)^q e^{-i[(st+u\tau)-i(px+vy)]} \end{aligned}$$

Now applying Cauchy's integral formula, we get

$$\begin{aligned} (-D_x)^q \psi_{\Delta p}(t, z, x, y) &= \frac{e^{-i[(st+u\tau)-i\tau y]}}{\Delta p} \left\{ \frac{1}{2\pi i} \int_C \left[\frac{\xi^q e^{-\xi x}}{\xi - p - \Delta p} - \frac{\xi^q e^{-\xi x}}{\xi - p} \right] d\xi \right\} \\ &\quad - e^{-i[(st+u\tau)-i\tau y]} \cdot \frac{1}{2\pi i} \int_C \frac{\xi^q e^{-\xi x}}{(\xi - p)^2} d\xi \\ &= \frac{e^{-i[(st+u\tau)-i\tau y]}}{2\pi i \Delta p} \int_C \left[\frac{1}{\xi - p - \Delta p} - \frac{1}{\xi - p} \right] \xi^q e^{-\xi x} d\xi - e^{-i[(st+u\tau)-i\tau y]} \cdot \frac{1}{2\pi i} \int_C \frac{\xi^q e^{-\xi x}}{(\xi - p)^2} d\xi \end{aligned}$$

$$\begin{aligned}
 &= \frac{e^{-i[(x+u)-iy]}}{2\pi i \Delta p} \int_c \left[\frac{\xi - p - \xi + p + \Delta p}{(\xi - p - \Delta p)(\xi - p)} \right] \xi^q e^{-\xi x} d\xi - e^{-i[(x+u)-iy]} \cdot \frac{1}{2\pi i} \int_c \frac{\xi^q e^{-\xi x}}{(\xi - p)^2} d\xi \\
 &= \frac{e^{-i[(x+u)-iy]}}{2\pi i} \int_c \left[\frac{1}{(\xi - p - \Delta p)(\xi - p)} - \frac{1}{(\xi - p)^2} \right] (\xi)^q e^{-\xi x} d\xi \\
 &= \frac{e^{-i[(x+u)-iy]}}{2\pi i} \int_c \left[\frac{\xi - p - \xi + p + \Delta p}{(\xi - p - \Delta p)(\xi - p)^2} \right] (\xi)^q e^{-\xi x} d\xi \\
 &= \frac{e^{-i[(x+u)-iy]}}{2\pi i} \Delta p \int_c \frac{(\xi)^q e^{-\xi x}}{(\xi - p - \Delta p)(\xi - p)^2} d\xi \\
 D_x^q D_y^m \psi_{\Delta p, \Delta v}(t, z, x, y) &= \frac{(-v)^m e^{-i[(x+u)-iy]}}{2\pi i} \Delta p \int_c \frac{(\xi)^q e^{-\xi x}}{(\xi - p - \Delta p)(\xi - p)^2} d\xi
 \end{aligned}$$

Now for all $\xi \in C$ and $0 < x < \infty$, $\sup_{I_1} \left| t^k z^r K_{a,b}(x) R_{c,d}(y) (v)^m e^{-i[(x+u)-iy]} \right| \leq K$, where K is a constant independent of ξ and x . Moreover, $|\xi - p - \Delta p| > h_1 - h > 0$ and $|\xi - p| = h_1$,

$C_1 = \max \{ |\xi|^q, \xi \in C \}$, consequently

$$\begin{aligned}
 &\sup_{I_1} \left| t^k z^r K_{a,b}(x) R_{c,d}(y) D_x^q D_y^m \psi_{\Delta p, \Delta v}(t, z, x, y) \right| \\
 &= \sup_{I_1} \left| t^k z^r K_{a,b}(x) R_{c,d}(y) \frac{(-v)^m e^{-i[(x+u)-iy]}}{2\pi i} \Delta p \int_c \left[\frac{(\xi)^q e^{-\xi x}}{(\xi - p - \Delta p)(\xi - p)^2} \right] d\xi \right| \\
 &\leq \sup_{I_1} \left| t^k z^r K_{a,b}(x) R_{c,d}(y) \frac{(v)^m e^{-i[(x+u)-iy]}}{2\pi} \right| |\Delta p| \int_c \frac{|\xi|^q |e^{-\xi x}|}{|\xi - p - \Delta p| |\xi - p|^2} |d\xi| \\
 &\leq \frac{|\Delta p|}{2\pi} \int_c \frac{KC_1}{(h_1 - h) h_1^2} |d\xi| \\
 &\leq \frac{|\Delta p|}{2\pi} \int_c \frac{C_2}{(h_1 - h) h_1^2} |d\xi|, \text{ where } C_2 = KC_1 \\
 &\leq \frac{|\Delta p| C_2}{2\pi (h_1 - h) h_1^2} 2\pi h_1, \\
 &\leq \frac{|\Delta p| C_2}{(h_1 - h) h_1},
 \end{aligned}$$

The right hand side is independent of x and converges to zero as $|\Delta p| \rightarrow 0$. This shows that $\psi_{\Delta p, \Delta v}(t, z, x, y)$ converges to zero in $FL_{a,b,c,d,n}$ as $|\Delta p| \rightarrow 0$, which end the proof.

CONCLUSIONS

In the present work Distributional Fourier-Laplace Transform is extended in two dimensions. Some

testing function spaces are developed. Also, Analytical behaviour of two dimensional Fourier-Laplace transform is presented.

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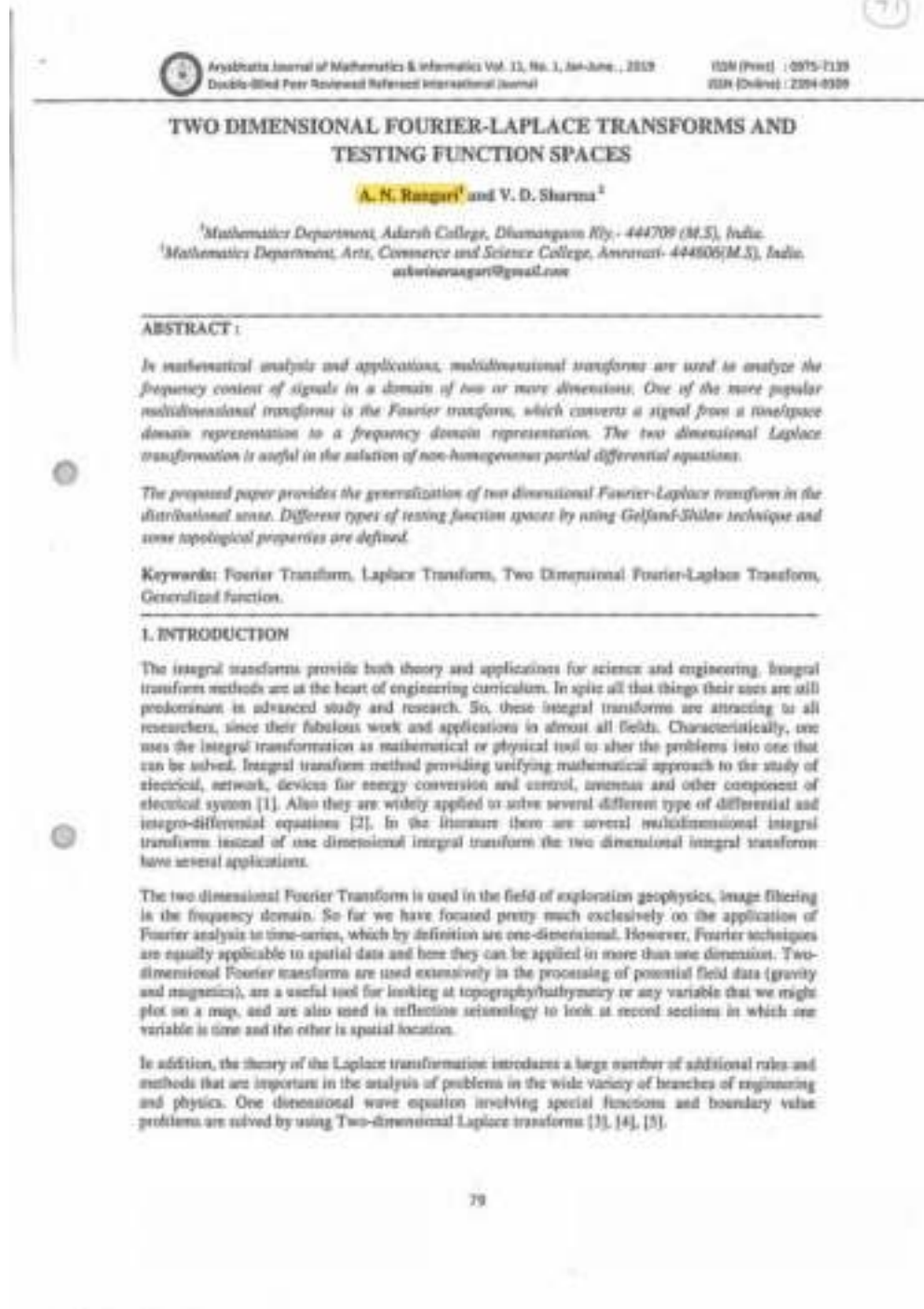
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Two Dimensional Fourier-Laplace Transform and Testing Functional Spaces: Ashwina Nilkanth Rangari



(72)

In the present paper, Two dimensional Fourier-Laplace transform is extended in the distributional sense. The plan of the paper is as follows:- The definitions are given in section 2. In section 3, twelve testing function space are defined by Gelfand-Shilov technique. Section 4 describes the distributional generalized two dimensional Fourier-Laplace transforms (2DFLT). In section 5 some topological properties are discussed. Lastly we conclude the paper.

The notations and terminology are as per Zemanian [7], [8].

2. DEFINITIONS

The Two Dimensional Laplace transform with the parameters p, v , of function $f(x, y)$ denoted by $L[f(x, y)] = F(p, v)$ and is given by,

$$L[f(x, y)] = F(p, v) = \int_0^{\infty} \int_0^{\infty} e^{-px-vy} f(x, y) dx dy \quad (2.1)$$

The Two Dimensional Fourier transform with the parameters s, u , of function $f(t, z)$ denoted by $F[f(t, z)] = F(s, u)$ and is given by,

$$F[f(t, z)] = F(s, u) = \int_{-\infty}^{\infty} \int_{-\infty}^{\infty} e^{-i(s+u\tau)} f(t, z) dt dz \quad (2.2)$$

The Two Dimensional Fourier-Laplace transform with parameters s, u, p, v , of $f(t, z, x, y)$ is defined as,

$$FL[f(t, z, x, y)] = F(s, u, p, v) = \int_{-\infty}^{\infty} \int_{-\infty}^{\infty} \int_0^{\infty} \int_0^{\infty} f(t, z, x, y) e^{-i(s+u\tau)-i(p+vy)} dt dz dx dy \quad (2.3)$$

where the kernel $K(s, u, p, v) = e^{-i(s+u\tau)-i(p+vy)}$

3. VARIOUS TESTING FUNCTION SPACES:-

3.1. The Space $FL_{s,h,c,d,B}(S_s)$ -type space:

Let I be the open set in $R_s \times R_s$ and E_s denotes the class of infinitely differentiable function defined on I , the space $FL_{s,h,c,d,B}$ is given by

$$FL_{s,h,c,d,B} = \left\{ \phi : \phi \in E_s / \gamma_{s,h,c,d,B}[\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} |t^h z^c K_{s,h}(x) R_{c,d}(y) D_s^h D_x^c D_y^d [\phi(t, z, x, y)]| \leq C_{s,h,c,d,B} A^h k^{4a} B^c r^{4a} \right\}$$

Where, the constants A, B and $C_{s,h,c,d,B}$ depend on the testing function ϕ .

3.2. The Space $FL_{a,b,c,d}^{\beta} (S^{\beta}\text{-type space})$:

This space is given by,

$$FL_{a,b,c,d}^{\beta} = \left\{ \phi : \phi \in E_a / \sigma_{a,b,c,d,h,r,q,m,l,n} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} |t^k z^l K_{a,b}(x) R_{c,d}(y) D_t^i D_x^j D_z^q D_y^m [\phi(t, z, x, y)]| \leq C_{a,b,c,d} G^i l^q H^m n^q \right\}$$

Where, the constants G, H and $C_{a,b,c,d}$ depend on the testing function ϕ .

3.3. The Space $FL_{a,b,c,d,e}^{\beta} (S_e^{\beta}\text{-type space})$:

This space is formed by combining the conditions (3.1) and (3.2)

$$FL_{a,b,c,d,e}^{\beta} = \left\{ \phi : \phi \in E_a / \rho_{a,b,c,d,h,r,q,m,l,n} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} |t^k z^l K_{a,b}(x) R_{c,d}(y) D_t^i D_x^j D_z^q D_y^m [\phi(t, z, x, y)]| \leq CA^k k^{l\alpha} B^r r^{\alpha} G^i l^q H^m n^q \right\}$$

where, the constants A, B, G, H and C depend on the testing function ϕ .

3.4. The space $FL_{a,b,c,d,f} (S_f\text{-type space})$:

This space is given by,

$$FL_{a,b,c,d,f} = \left\{ \phi : \phi \in E_a / \xi_{a,b,c,d,h,r,q,m,l,n} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} |t^k z^l K_{a,b}(x) R_{c,d}(y) D_t^i D_x^j D_z^q D_y^m [\phi(t, z, x, y)]| \leq C_{a,b,c,d} A^q q^{\alpha} B^m m^{\alpha} \right\}$$

where, the constants A, B and $C_{a,b,c,d}$ depend on the testing function ϕ .

3.5. The Space $FL_{a,b,c,d,n,g,h}^{\beta}$:

It is defined as,

$$FL_{a,b,c,d,n,g,h}^{\beta} = \left\{ \phi : \phi \in E_a / \gamma'_{a,b,c,d,h,r,q,m,l,n} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} |t^k z^l K_{a,b}(x) R_{c,d}(y) D_t^i D_x^j D_z^q D_y^m [\phi(t, z, x, y)]| \leq C_{a,b,c,d,n} (g + \delta)^k k^{l\alpha} (h + \mu)^r r^{\alpha} \right\}$$

For any $\delta > 0$, $\mu > 0$ and g, h are the constants depending on the function ϕ .

3.6. The space $FL_{a,b,c,d}^{\beta,i,j}$:

It is defined as,

$$FL_{a,b,c,d}^{\beta,i,j} = \left\{ \phi : \phi \in E_a / \sigma'_{a,b,c,d,k,r,q,m,j,n} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} |t^i z^j K_{a,b}(x) R_{c,d}(y) D_t^\beta D_x^\alpha D_z^\alpha D_y^\alpha [\phi(t, z, x, y)]| \leq C_{i,r,q,m,j,n} (i+\varepsilon)^j t^{i\beta} (j+\eta)^\alpha n^{\alpha\beta} \right\}$$

For any $\varepsilon > 0$, $\eta > 0$ and i, j are the constants depending on the function ϕ .

3.7. The Space $FL_{a,b,c,d,g,h}^{\beta,i,j}$

This space is defined by combining (3.5) and (3.6) as,

$$FL_{a,b,c,d,g,h}^{\beta,i,j} = \left\{ \phi : \phi \in E_a / \rho'_{a,b,c,d,k,r,q,m,j,n} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} |t^i z^j K_{a,b}(x) R_{c,d}(y) D_t^\beta D_x^\alpha D_z^\alpha D_y^\alpha [\phi(t, z, x, y)]| \right. \\ \left. \leq C_{i,r,q,m,j,n} (g+\delta)^i k^{\alpha\beta} (h+\mu)^j r^\alpha (i+\varepsilon)^j t^{i\beta} (j+\eta)^\alpha n^{\alpha\beta} \right\}$$

For any $\delta > 0$, $\mu > 0$, $\varepsilon > 0$, $\eta > 0$ and for given g, h, i, j are the constants depending on the function ϕ .

3.8. The Space $FL_{a,b,c,d,f,\omega}^{\beta,i,j}$

This space is given by,

$$FL_{a,b,c,d,f,\omega}^{\beta,i,j} = \left\{ \phi : \phi \in E_a / \xi'_{a,b,c,d,k,r,q,m,j,n} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{I_1} |t^i z^j K_{a,b}(x) R_{c,d}(y) D_t^\beta D_x^\alpha D_z^\alpha D_y^\alpha [\phi(t, z, x, y)]| \leq C_{i,r,q,m,j,n} (e+\tau)^i q^{\alpha\beta} (f+\omega)^\alpha m^{\alpha\beta} \right\}$$

For any $\tau > 0$, $\omega > 0$ and e, f are the constants depending on the function ϕ .

3.9. The Space $F^r L_{a,b,c,d,\omega}$

It is given by,

$$F^r L_{a,b,c,d,\omega} = \left\{ \phi : \phi \in E_a / \zeta'_{a,b,c,d,k,r,q,m,j,n} [\phi(t, z, x, y)] \right. \\ \left. = \sup_{\substack{-\infty < t, z < 0 \\ 0 < x, y < \infty}} |(-t)^i (-z)^j K_{a,b}(x) R_{c,d}(y) D_t^\beta D_x^\alpha D_z^\alpha D_y^\alpha [\phi(t, z, x, y)]| \leq C_{i,r,q,m,j,n} A^i k^{\alpha\beta} B^j r^{\alpha\beta} \right\}$$

The smooth function $\phi(t, z, x, y)$ defined on I_2 is in $F^r L_{a,b,c,d,\omega}$ if $\phi^*(t, z, x, y) = \phi(-t, -z, x, y)$ is in $FL_{a,b,c,d,\omega}$.

76

These topologies are respectively generated by the total families of semi-norms,

$$\begin{aligned} & \{\gamma_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\sigma_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\rho_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\xi_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\gamma'_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \\ & \{\sigma'_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\rho'_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\xi'_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\zeta_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\lambda_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \\ & \{\kappa_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}, \{\psi_{a,b,c,d,e,f,g,h,i,j,k,l,m,n}\}. \end{aligned}$$

4. DISTRIBUTIONAL GENERALIZED TWO DIMENSIONAL FOURIER-LAPLACE TRANSFORM (2DFLT):-

For $f(t, z, x, y) \in FL'_{a,b,c,d,e}$, where $FL'_{a,b,c,d,e}$ is the dual space of $FL_{a,b,c,d,e}$. It contains all distributions of compact support. The distributional two dimensional Fourier-Laplace transform is a function of $f(t, z, x, y)$ is defined as,

$$FL\{f(t, z, x, y)\} = F(s, u, p, v) = \langle f(t, z, x, y), \phi(t, z, x, y, s, u, p, v) \rangle, \quad (4.1)$$

Where $\phi(t, z, x, y, s, u, p, v) = e^{-i[su + ut + i(pv + vy)]}$ and for each fixed $t (0 < t < \infty)$, $z (0 < z < \infty)$, $x (0 < x < \infty)$ and $y (0 < y < \infty)$. Also $s > 0$, $u > 0$, $p > 0$ and $v > 0$. The right hand side of (4.1) has a sense as an application of $f(t, z, x, y) \in FL'_{a,b,c,d,e}$ to $\phi(t, z, x, y, s, u, p, v) \in FL_{a,b,c,d,e}$.

5. TOPOLOGICAL PROPERTIES:-

5.1. Theorem: For a real numbers $a_1, a_2, b_1, b_2, c_1, c_2, d_1$ and d_2 such that $a_1 \leq a_2, b_1 \leq b_2, c_1 \leq c_2$ and $d_1 \leq d_2$ then $2DFL_{a_1,b_1,c_1,d_1,e} \subset 2DFL_{a_2,b_2,c_2,d_2,e}$ and the induced topology on $2DFL_{a_2,b_2,c_2,d_2,e}$ is weaker than the original topology that is $T_{a_1,b_1,c_1,d_1,e} / 2DFL_{a_2,b_2,c_2,d_2,e} \subset T_{a_2,b_2,c_2,d_2,e}$.

Proof:- Consider,

$$\begin{aligned} \gamma_{a_1,b_1,c_1,d_1,e,f,g,h,i,j,k,l,m,n}[\phi(t, z, x, y)] &= \sup_{I_1} |t^s z^u K_{a_1,b_1}(x) R_{c_1,d_1}(y) D_1^f D_2^g D_3^h D_4^i D_5^j [\phi(t, z, x, y)]| \\ &\leq \sup_{I_1} |t^s z^u K_{a_2,b_2}(x) R_{c_2,d_2}(y) D_1^f D_2^g D_3^h D_4^i D_5^j [\phi(t, z, x, y)]| \\ &= \gamma_{a_2,b_2,c_2,d_2,e,f,g,h,i,j,k,l,m,n}[\phi(t, z, x, y)] \end{aligned}$$

Hence $2DFL_{a_1,b_1,c_1,d_1,e} \subset 2DFL_{a_2,b_2,c_2,d_2,e}$ if $a_1 \leq a_2, b_1 \leq b_2, c_1 \leq c_2$ and $d_1 \leq d_2$.

Second part of the proof is simple and hence omitted. This completes the proof.

5.2. Theorem: If $\alpha_1 < \alpha_2$ and $\beta_1 < \beta_2$ then $FL_{a,b,c,d,\alpha_1}^{\beta_1} \subset FL_{a,b,c,d,\alpha_2}^{\beta_2}$ and the topology of $FL_{a,b,c,d,\alpha_2}^{\beta_2}$ is equivalent to the topology induced on $FL_{a,b,c,d,\alpha_1}^{\beta_1}$ by $FL_{a,b,c,d,\alpha_2}^{\beta_2}$.

Proof:- Let $\phi \in FL_{a,b,c,d,\alpha_1}^{\beta_1}$ therefore

77

$$\begin{aligned}\rho_{a,b,d,k,r,q,m,l,n}(\phi) &= \sup_{I_1} |t^k z^r K_{a,b}(x) R_{c,d}(y) D_t^l D_x^q D_y^m [\phi(t, z, x, y)]| \\ &\leq CA^k k^{ka} B^r r^{rb} G^l l^{lq} H^m m^{mq} \\ &\leq CA^k k^{ka} B^r r^{rb} G^l l^{lq} H^m m^{mq} \text{ where } k, r, q, m, l, n = 0, 1, 2, 3, \dots\end{aligned}$$

Hence $\phi \in FL_{a,b,d}^k$.

Consequently, $FL_{a,b,d}^k \subset FL_{a,b,d}^k$. The topology of $FL_{a,b,d}^k$ is equivalent to the topology $T_{a,b,d}^k / FL_{a,b,d}^k$. It is clear from the definition of the topologies of these spaces.

5.3. Theorem: $FL_{a,b,d} = \bigcup_{k=1}^{\infty} FL_{a,b,d}^k$ and if the space $FL_{a,b,d}$ is equipped with the strict $FL_{a,b,d}$ inductive limit topology defined by the injection maps from $FL_{a,b,d}^k$ to $FL_{a,b,d}$ then the sequence $\{\phi_s\}$ in $FL_{a,b,d}$ converges to zero iff $\{\phi_s\}$ is contained in some $FL_{a,b,d}^k$ and converges to zero.

Proof: Once we show that $FL_{a,b,d} = \bigcup_{k=1}^{\infty} FL_{a,b,d}^k$.

Clearly $\bigcup_{k=1}^{\infty} FL_{a,b,d}^k \subset FL_{a,b,d}$.

For proving other inclusion, let $\phi \in FL_{a,b,d}$ then

$\rho_{a,b,d,k,r,q,m,l,n}(\phi) = \sup_{I_1} |t^k z^r K_{a,b}(x) R_{c,d}(y) D_t^l D_x^q D_y^m [\phi(t, z, x, y)]|$ is bounded by some number. We can choose the integers α_m and β_n such that

$$\rho_{a,b,d,k,r,q,m,l,n}(\phi) \leq CA^k k^{ka} B^r r^{rb} G^l l^{lq} H^m m^{mq}$$

$\therefore \phi \in FL_{a,b,d}^k$ for some integers α_m and β_n .

Hence $FL_{a,b,d} \subset \bigcup_{k=1}^{\infty} FL_{a,b,d}^k$.

Thus, $FL_{a,b,d} = \bigcup_{k=1}^{\infty} FL_{a,b,d}^k$.

5.4. Definition:

Let $\{a_s\}$, $\{b_s\}$, $\{c_s\}$ and $\{d_s\}$ be monotonic sequence, converging to $w+$, $z-$, $\theta+$, $\pi-$ respectively.

Now we define countable union space. $FL[w, z, \theta, \pi, \alpha] = \bigcup_{s=1}^{\infty} FL_{a_s, b_s, c_s, d_s}$

(5.4) Concerning this space we prove next theorem.

5.5. Theorem: The space $FL\{w, z, \theta, \pi, \alpha\}$ is independent of the choice of the sequence $\{a_n\}$, $\{b_n\}$, $\{c_n\}$ and $\{d_n\}$. If $FL\{w, z, \theta, \pi, \alpha\}$ is equipped with the strict inductive limit topology defined by $FL_{b_n, c_n, d_n, \pi, \alpha}$ then the sequence $\{\phi_n\}$ in $FL\{w, z, \theta, \pi, \alpha\}$ converges to zero iff $\{\phi_n\}$ belongs to some $FL_{b_n, c_n, d_n, \pi, \alpha}$ and converges to zero in that space. Moreover $FL\{w, z, \theta, \pi, \alpha\}$ is complete.

Proof: Proof is easy and hence omitted.

6. CONCLUSIONS

The proposed paper provides the generalization of two dimensional Fourier-Laplace transform in the distributional sense. Different types of testing function spaces by using Gelfand-Shilov technique and some topological properties are defined.

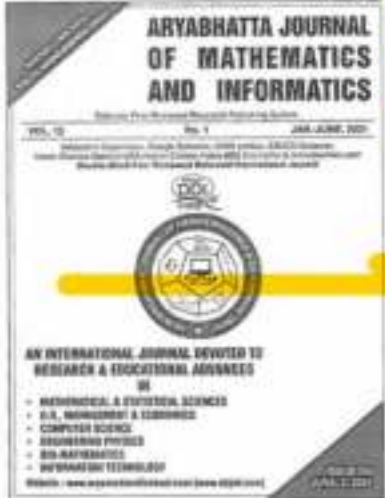
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Exigobacterium aurantiacum virulent pigment producing a novel pathogenic bacteria associated with cases of corneal ulcers : Dr. Vilas Adinath Kamble

Original Research Article

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Exigobacterium aurantiacum virulent pigment producing a novel pathogenic bacteria associated with cases of corneal ulcers

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Abstract

Blindness is a major public health problem in most developing countries. Corneal ulcer (Keratitis) is a major cause of blindness throughout the world. About 10% cases of blindness are due to corneal ulcer. "Corneal ulcer means loss of corneal substances as a result of infection and formation of raw, excavated area." Bacterial keratitis is an acute or chronic, transient or recurrent infection of the cornea with varying predilection for anatomical and topographical parts of the cornea like marginal or central. This study demonstrates the Novel pathogenic bacteria *Exigobacterium aurantiacum* with had pigment production, which are causing corneal ulcer and determine the best possible empirical therapy and specific therapy. Particular attention should be given to this condition as it can progress very rapidly with complete corneal destruction can be occur within 24-48 hours.

Keywords: Corneal Ulcer, *Exigobacterium aurantiacum*, Molecular characterization, Antibiotic treatment, Pigment.

Introduction

Vision is an important part of our everyday life. Eyes are the organs of vision. The eye which is the window to the soul, it's mainly depends on the clarity of the cornea. Corneal ulceration is major cause of monocular blindness in developing countries. Surveys in Asia and Africa have confirmed these findings.^{1,2} Corneal ulcer is a major cause of blindness throughout the world. About 10% cases of blindness are due to Corneal ulcer.^{3,4} "Corneal ulcer means loss of corneal substances as a result of infection and formation of raw, excavated area".⁵

Blindness is major public health problem in most developing countries. The World Health Organization (WHO) estimates 45 million blind cases in the world and another 135 million that have severely impaired vision in both eyes. Out of which 5.4 million blind people are from India.^{4,6,7} In the total disabled population of the Maharashtra state, the proportion of in seeing disabled persons is ranked first.^{1,8} It is important to note that corneal diseases including corneal ulcers are among the major cause of vision loss and blindness. It is projected that corneal blindness in India will reach up to 10.6 million by 2020.^{9,10}

The importance of corneal disease as a major cause of blindness in the world today remains second only to cataract, but its epidemiology is complicated and encompasses a wide variety of infectious and inflammatory eye diseases.^{11,4}

Corneal ulcers can be caused by exogenous infections i.e. by bacteria, fungi, viruses or parasites and sometimes it is allergic in nature or it can be due to endogenous infections. Almost any organism can invade the corneal stroma if the normal corneal defence

mechanisms, i.e., lids, tear film and corneal epithelium are compromised.^{12,6}

Bacterial keratitis accounts for approximately 65% to 90% of all microbial corneal infections. Tiny tears or scratches on the cornea can make it easier for bacteria to enter the eye and cause infection. A severe ulcer without treatment can lead to blindness in the affected eye. Bacterial keratitis is caused by *Staphylococcus aureus*, *Streptococcus pneumoniae*, *Escherichia coli*, *Enterococci*, *Pseudomonas*, *Klebsiella*, *Proteus*, *Bacillus cereus*, *N. gonorrhoeae* and many other bacteria.^{13,15,6}

Alteration in the components of tear fluid or the aqueous humour may result in pathological changes in cornea. This study demonstrates The Novel Study for Keratitis and the isolation of Novel Bacteria which are causing Keratitis. Prompt diagnosis of corneal ulcers and virulence factors and treatment with appropriate antibiotics prevent blindness and devastating visual disability.

Materials and Methods

The present work was undertaken to characterize the unknown novel pathogenic bacteria from corneal ulcer by performing its isolation, identification, molecular characterization and virulent factors.

Collection of Samples: Standard Methods were usually adopted for samples collection.^{16,17} Corneal scrapings were taken from patients with infectious keratitis under topical anesthesia using a sterile No. 11 and 15 Bard Parker Surgical blades and by swabbing the lid margins with sterile tooth-mounted cotton swabs by ophthalmologist. Scrapings were taken from the edges and base of the ulcer.^{13,15,18,6} Samples were collected in sterilized container containing Nutrient broth and

transferred immediately to laboratory for further processing. A total of 300 samples were collected from clinically diagnosed patients of corneal ulcers, admitted in Government Medical College and Hospital, Ophthalmic Hospitals and Clinical Laboratories of various districts and towns of Maharashtra state like Akola, Jalna, Buldhana, Chikhli etc. The hospitals were selected on the basis of geographical proximity, feasibility of conducting the study and availability of the samples.^{40,41}

Enrichment of Samples: Samples were collected in sterile container containing 0.5ml of Brain Heart Infusion Broth (BHI) as enrichment culture medium which supports the growth of bacteria and then transferred immediately to laboratory for further processing. Samples were then incubated at 37 °C for 24 hours for propagation.^{15,40,41}

Isolation and Identification of Pathogenic Bacteria by Phenotypic Methods: After 24 hours of incubation, a loopful of each enriched culture was taken and streaked on CLED agar and Nutrient agar plates in aseptic conditions and were incubated at 37 °C for 24 hours. Colonies with different morphological characters (Size, Shape, Margin, Texture, Opacity, Elevation, Colour, Motility and Gram's (Positive) characters were selected and inoculated on respective selective media viz. Milk agar, Mannitol salt agar. All the plates were incubated at 37 °C for 24 hours. All the suspicious screened colonies of the isolates were then analyzed for

Table 1: Primer details

Primer Name	Sequence Details	Number of Base
27F	AGAGTTTGATCMTGGCTCAG	20
1492R	TACGGYTACCTTGTTACGACTT	22

Add 1µL of template DNA in 20µL of PCR reaction solution. Use 27F/1492R primers used for bacteria, and then PCR reaction performed with below conditions: Initial denaturation 94°C for 2 min and then 35 amplification cycles at 94°C for 45 sec, 55°C for 60 sec, and 72°C for 60 sec. Final extension at 72°C for 10 min. DNA fragments are amplified about 1,400bp in the case of bacteria. Include a positive control (*E.coli* genomic DNA) and a negative control in the PCR.⁴⁰

Purification of PCR Products: Removed unincorporated PCR primers and dNTPs from PCR products by using Montage PCR Clean up kit

their conventional biochemical tests such as Carbohydrate fermentation, IMViC, Enzyme tests etc.^{19,40,41}

Identification of Pathogenic Bacteria by Genotypic

Methods: Molecular studies were carried out by using 16S rRNA analysis at Yaazh Xenomics (Crack the life code), DNA sequencing service, Madurai (Chennai Branch), Tamil Nadu, (India).⁴⁰ The molecular characterization of different pathogenic bacteria was done in following steps.⁴⁰

DNA Extraction: Bacterial Genomic DNA was isolated using the InstaGene™ Matrix Genomic DNA isolation kit – As per the kit instruction below procedure followed. An isolated bacterial colony was picked and suspend in 1ml of sterile water in a micro centrifuge tube. Centrifuge it for 1 minute at 10,000–12,000 rpm to remove the supernatant. Add 200 µl of Insta Gene matrix to the pellet and incubate at 56°C for 15 minutes. 5. Vortex at high speed for 10 seconds and place the tube in a 100°C in heat block or boiling water bath for 8 minutes. 6. Finally, vortex the content at high speed for 10 seconds and Spin at 10,000–12,000 rpm for 2 minutes. 7. In result, 20µl of the supernatant was used per 50 µl PCR reaction.⁴⁰

PCR Protocol: Using below 16S rRNA Universal primers gene fragment was amplified using MJ Research Peltier Thermal Cycler.

(Millipore). The PCR product was sequenced using the 518F/800R primers. Sequencing reactions were performed using a ABI PRISM® BigDye™ Terminator Cycle Sequencing Kits with AmpliTaq® DNA polymerase (FS enzyme).⁴⁰

Sequencing Protocol: Single-pass sequencing was performed on each template using below 16s rRNA universal primers. The fluorescent-labelled fragments were purified from the unincorporated terminators with an ethanol precipitation protocol. The samples were resuspended in distilled water and subjected to electrophoresis in an ABI 3730xl sequencer.⁴⁰

Table 2: Sequencing primer details

Primer Name	Sequence Details	Number of Base
785F	GGATTAGATACCCTGGTA	18
907R	CCGTCAATTCMTTTRAGTTT	20

Sequence data was aligned and analyzed for Identifying the Sample.⁴⁰

16S rRNA Sequence Analysis: Database homology searching was performed with BLAST on the NCBI (National Center for Biotechnology Information).

Antimicrobial Susceptibility Testing: After molecular characterization, the pathogenic bacteria were subjected for antibiogram. The antimicrobial susceptibility testing was done by the agar Disk Diffusion Method.^{26,31,40} Antibiotic disc were placed on a lawn culture of the

isolate under test on Mueller Hinton Agar (MHA).⁴⁰ The Antibiotics used were:

β-Lactam Antibiotics: β-Lactamase production in all the isolates was detected by Disc Diffusion Method and Well Diffusion Method by using three groups of antibiotics.⁴⁰ **1) Penicillins:** Amoxicillin (10 mcg), Amoxycylav (20µg Amoxicillin and 10µg Clavulanic acid) (30 mcg), Ampicillin (10 mcg), Methicillin (10 mcg), Penicillin G (10 mcg), Cephalosporins: Cefalexin (30 mcg), Cefixime (30 mcg), Cefpodoxime (30 mcg), Cefazidime (30 mcg), Cefotaxime (30 mcg), Cephalosporin (5%), Cefadroxil (30 mcg), Ceftriaxone (30 mcg) 3) Carbapenems: Imipenem (10mcg), Meropenem (10mcg).

Non β-Lactam antibiotics: **1) Tetracycline:** Tetracycline (30 mcg), Chloramphenicol (30 mcg). **2) Aminoglycosides:** Bacitracin (30 mcg), Kanamycin (20 mcg), Gentamicin (10 mcg), Erythromycin (15 mcg), Tobramycin (1.33%), Vancomycin (30mcg). **3) Fluoroquinolones:** Ofloxacin (5 mcg), Moxifloxacin (0.5%). **4) Quinolones:** Ciprofloxacin (10 mcg), Norfloxacin (10 mcg).

Combination Therapy or Dual Therapy

1. Cefazidime + Ciprofloxacin
2. Cephazolin + Vancomycin
3. Cephazolin+ Tobramycin
4. Gentamicin + Cefuroxime

Pigment Extraction and its Assessment as Virulent

Factor: Seed cultures were prepared by inoculating 100 ml Erlenmeyer flask containing 30 ml nutrient broth by a loopful from a single pigmented colony and shaken at 150 rpm at 30 °C till bacterial growth reached OD 550 nm = 1. The seed culture was introduced into 1000 ml Erlenmeyer flasks containing 500 ml of sterile nutrient broth, a growth medium with pH adjusted to 7. Inoculated flask was incubated in a rotary shaker at 180rpm, 37°C for three days. After three days of incubation, cells were harvested by centrifugation at 8,000 rpm for 15 minutes. Then the pellet was washed with sterile distilled water and again centrifuged for 15 minutes at 4,000rpm. Then 5 ml of methanol was added to the pellet and suspended it. Then it was incubated in a water bath at 60°C for 15 minutes until all visible pigments were extracted and centrifuged at 4,000 rpm for 15 minutes. The process was repeated till the cell pellets became colourless. The coloured supernatant was separated and then it was filtered through Whatman filter paper no. 1.^{21,22}

Pigment Identification by FTIR: The qualitative identification of pigments was done by Infrared spectroscopy.

Assay of Nitric Oxide Scavenging Activity: According to the method of^{24,25,42} the assay of scavenging activity was done by taking reaction mixture (5.0 ml) containing sodium nitroprusside (10mM) in phosphate buffered saline (pH 7.3), was mixed with different concentrations (25, 50, 100µg/ml) of methanol extract of each pigment were dissolved in

methanol and incubated at 30°C for 3 hours. The same reaction mixture without the extract but the equivalent amount of methanol served as the control. The NO radical thus generated interacted with oxygen to produce the nitrite ion (NO⁻) which was assayed at 30 min intervals by mixing 1.0 ml of incubation mixture with an equal amount of Griess reagent (1% sulfanilamide in 5% phosphoric acid and 0.1% naphthyl ethylenediamine dihydrochloride). The absorbance was measured at 546 nm. The nitrite generated in the presence of the pigment extract was estimated using a standard curve based on sodium nitrite solutions of known concentrations. Each experiment was carried out at least three times and the data presented as an average of three independent determinations.

Antioxidant Activity Assay: The percentage inhibition of the extract was calculated and recorded.^{34,25,43} The percentage nitrite radical scavenging activity of the methanol extracts was calculated using the following formula:

Percentage nitrite oxide radical scavenging activity:

$$\% \text{ Inhibition} = \frac{A_{\text{control}} - A_{\text{test}}}{A_{\text{control}}} \times 100$$

Where, A_{control} = absorbance of control sample and A_{test} = absorbance in the presence of the samples of extracts.

Statistical Analysis

Data analysis was performed using SPSS software (Statistical Package for the Social Sciences). By using ANOVA (Analysis of variance) test (using the number of independent variables in one way and two way) levels of significance were determined. $P \leq 0.05$ values were considered to be statistically significant difference.

Results

In present research study 300 samples were collected during period of January 2014 to January 2017 from clinically diagnosed patients of corneal ulcers, admitted in government medical college, ophthalmic hospitals and clinical laboratories of various cities and towns.⁴⁰ Out of 300 samples, bacteria were isolated from 212 samples. The patients selected were belonging to both genders and age groups varying from 11 to 80 years from rural and urban areas. Highest prevalence of bacterial corneal ulcers was found to be in the patients belonging to the age group of 31-40 years. Male patients were noted to be at an increased risk of corneal ulcers as compared to female patients. The distribution of patients as per the place of resident reveals that 61% of cases were found from urban areas and 39% cases were obtained from rural area. As per the etiology of corneal ulcer cases, noted in these investigations, the significant number (32%) of isolates were obtained from the patients with contact lens users, followed by 21% isolates from traumatic patients. The severity distribution of bacterial corneal ulcers reveals

34.57% cases of mild, 29.25% cases of moderate and 36.17% cases of severe.

From 212 total positive clinical samples 278 different kinds of bacteria were isolated. Out of 278 isolates, 07 isolates were identified as *Exiguobacterium aurantiacum* (coded as PBCUUn05). PBCUUn05 isolates were Gram positive, motile and bacilli in shape. They showed yellow to orange coloured colonies on nutrient agar and mannitol fermenting yellow coloured colonies on mannitol salt agar.⁴¹ PBCUUn05 fermented Mannitol, maltose and sucrose sugars with acid and gas production. These isolates were indole positive, amylase, Catalase, gelatinase and nitrate reduction positive. These isolates have ability to hydrolyze DNA and utilize it as a source of carbon and energy for growth by showing DNase enzyme test positive.

We have confirmed our findings again and again. To acquire the accuracy in the assignment of genus and species, these 07 Isolates of *Exiguobacterium aurantiacum* were further confirmed by 16SrRNA molecular analysis.⁴⁰ The 16s rRNA sequence was blast using NCBI blast similarity search tool. The phylogeny analysis of our sequence with the closely related sequence of blast results was performed followed by multiple sequence alignment. By using the program MUSCLE 3.7, the sequence was compared with sequences of closely related species in GenBank by multiple sequence alignment.^{36,40} Phylogenetic relationships were determined by using the neighbour joining method.

Sequences producing significant alignments:

Name of isolate	Maximum score	Total score	Query cover	E value	Total Identity	Accession No.
<i>Exiguobacterium aurantiacum</i>	2760	2760	87%	0.0	99%	KJ722475.1

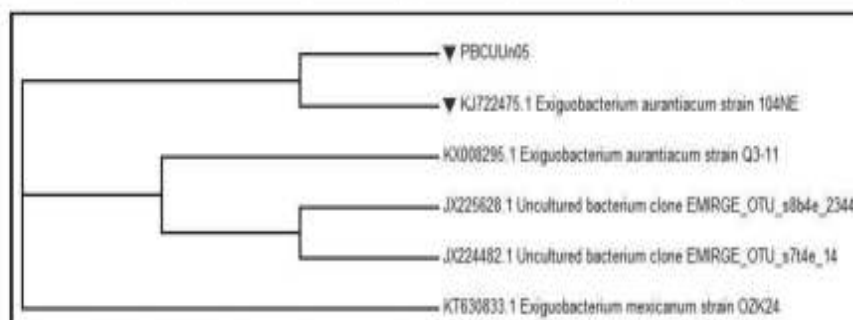


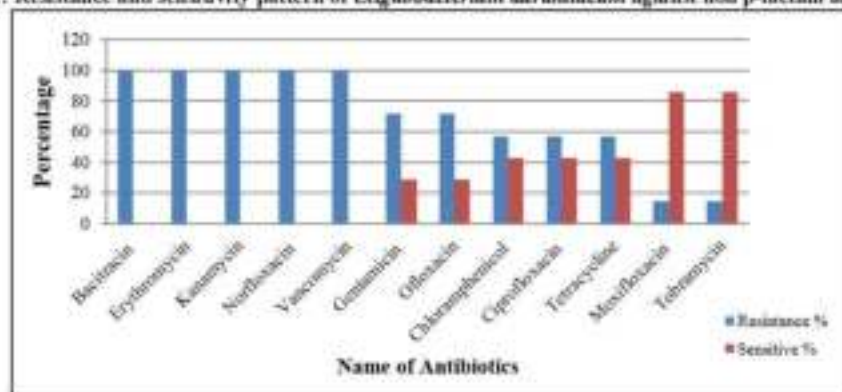
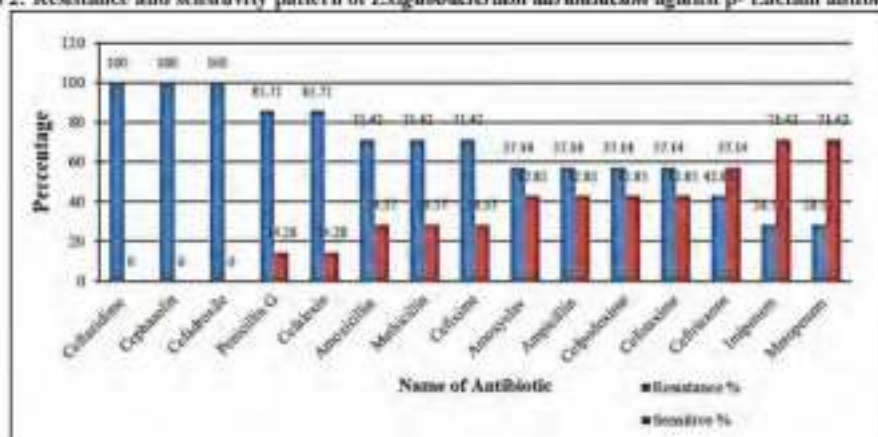
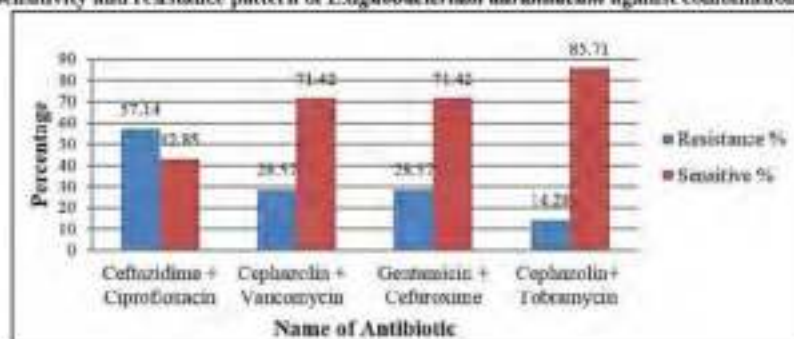
Fig. 1: Phylogenetic tree of *Exiguobacterium aurantiacum* isolate (PBCUUn05) and related species

Sequencing of the 16S rRNA of the isolates showed that there was 99% similarity between the 16S rRNA sequences of PBCUUn05 isolates of present study and *Exiguobacterium aurantiacum* strain 104NE (GenBank accession no. KJ722475.1). This provided us taxonomy report with 99% identity in BLAST analysis. The 16S rRNA was made up of 1706 base pairs.

In present study, antibiotic susceptibility studied for *Exiguobacterium aurantiacum* isolated from corneal ulcers and examined towards a panel of 27 antibiotics of several group (viz., 12 non β -lactam, 15 β -lactam). 04 combination of antibiotics using Kirby Bauer's disc diffusion method on Mueller Hinton Agar. The results of antibiotic susceptibility testing were categorised as sensitive, intermediately sensitive and resistant on the basis of zone of inhibition (Scale: <10: Resistant, 10-12: Intermediate, >12: Sensitive).

Exiguobacterium aurantiacum showed 100% resistance to Bacitracin, Erythromycin, Kanamycin, Norfloxacin and Vancomycin antibiotics. They had

shown sensitivity towards Moxifloxacin, Tobramycin (Graph 1). Cefazidime, Cephazolin, Cefadroxile showed 0% effectiveness on *E. aurantiacum* isolates while Ceftriaxone, Imipenem, Meropenem showed 57 to 72% sensitivity (Graph 2). These isolates have shown 57.14% resistance towards Cefazidime + Ciprofloxacin, intermediate resistances towards Cephazolin + Vancomycin and Gentamicin + Cefuroxime combinations but 85% isolates were sensitive to Cephazolin+ Tobramycin combinations (Graph 3).

Graph 1: Resistance and sensitivity pattern of *exiguobacterium aurantiacum* against non β -lactam antibioticsGraph 2: Resistance and sensitivity pattern of *Exiguobacterium aurantiacum* against β -Lactam antibioticsGraph 3: Sensitivity and resistance pattern of *Exiguobacterium aurantiacum* against combination therapy

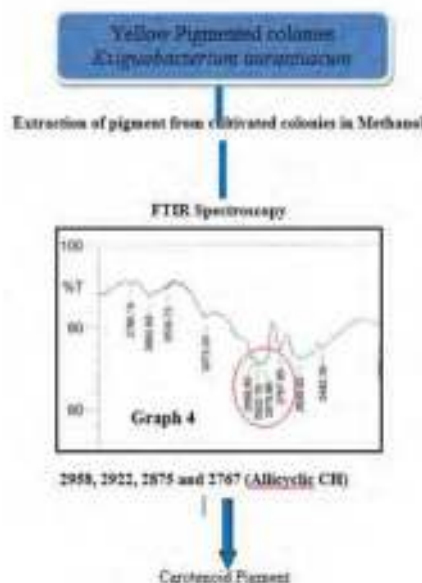
In present study, *Exiguobacterium aurantiacum* was found to be the novel bacterium and not reported earlier from the cases of corneal ulcers. As a matter of inquisitiveness, these novel isolates were

further subjected to virulence studies, which constitute one of the mechanisms for survival.

The pigment production is also one of the mechanisms among bacterial pathogens to increase their virulence. In the present studies, Pigment

identification was done by studying characteristic peaks in FTIR spectrum, which confirms the presence of structural functional groups (Graph 4).

FTIR spectrum of dried powder of isolated pigments confirmed that *Exiguobacterium aurantiacum* produces Carotenoid pigment.



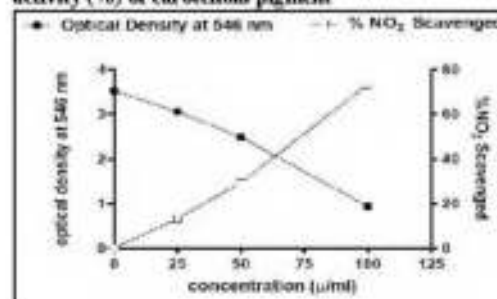
Scavenging effect of Carotenoid on NO production: Nitric oxide released from sodium nitropruside has a strong NO⁺ character which can alter the structure and function of many cellular components.⁴² The methanol extract of Carotenoid pigments isolated from *Exiguobacterium aurantiacum* exhibited well NO scavenging activity leading to the reduction of the nitrite concentration in the assay medium. The NO scavenging capacity was concentration dependent.⁴² In this study, with 100 µg/ml scavenging was found most efficient. The present study showed a potent nitric oxide scavenging activity of Carotenoid pigments. The absorbance of the chromophore (purple azo dye) formed during the diazotisation of nitrite ions with sulphanilamide and subsequent coupling with naphthyl ethylenediamine dihydrochloride was measured at 546 nm (Table 1).⁴²

Antioxidant Activity: The percentage inhibition of the Carotenoid pigment extract was calculated and recorded. The Carotenoid pigments showed a very good concentration-dependent inhibition of nitric oxide. Graph 5 is showing that, as concentration of pigments is increasing, % of inhibition is increasing and absorbance decreasing. These pigments are showing virulent factor with an antioxidant action because these pigments neutralize the oxidants.

Table 1: Absorbance and Nitric oxide scavenging activity (%) of Carotenoid pigment

Concentration (µg/ml)	Optical density at 546nm	% NO ₂ Scavenged
0	3.53	0
25	3.06	13.31
50	2.49	29.46
100	0.94	73.37

Graph 5: Absorbance and nitric oxide scavenging activity (%) of carotenoid pigment



Discussion

Corneal ulceration has only recently been recognized as a 'silent epidemic' in developing countries. Corneal scarring and vision loss are possible consequences. Bacterial corneal ulcer is a therapeutic challenge and vision threatening serious ocular condition.

Bacterial Corneal infection can affect people of all ages. The age groups with a higher prevalence of disease are likely tied to risk factors, in the first group (<40 years) who are more likely to be contact lenses wearers and/or sustain ocular trauma, and those in the second group (>50 years) who are more likely to undergo eye surgery. In this study, majority of the patients (49%) were in 3rd and 4th decade which is the crucial stage of the life. In a study by Panda et al.,²⁷ performed on thousand eyes of thousand patients, 50% of the patients with corneal ulcer were aged between 36 and 65 years. The present study showed similar age distribution.

In present study, the incidence of bacterial keratitis was higher in males (54%) than in females (46%) with Male to Female Ratio 1.18:1. This ratio was very close to the ratio 1.6:1 of previous study reported by Srinivasan et al., in 1997;²⁸ Maske et al., 1986²⁹ and 1.9:1 by Narsani et al., 2009.³⁰ The outcome of the study according to male female distribution was found in male due to predisposing factors like traumatic condition and use of contact lens.

Earlier several bacteria viz. *Staphylococcus aureus*, *Pseudomonas aeruginosa*, *Escherichia coli*, *Klebsiella pneumoniae* were isolated and identified in the cases of corneal ulcers.⁴¹ *Exiguobacterium aurantiacum* was not reported earlier from the cases of corneal ulcers as per

the literature. In present studies, same organism was isolated from the patients associated with the cases of corneal ulcers with the isolation rate of 02.51%.

The genus *Exiguobacterium* was first described in 1983 by Collins *et al.*, who characterized the species *E. aurantiacum*. It is facultative anaerobic and varied in shape from rods to almost coccoid forms. In some respects it resembled certain coryneform bacteria but its true taxonomic position was not determined. *Exiguobacterium aurantiacum* founds from potato-processing effluent.³¹

Exiguobacterium spp. are alkaliphilic, halotolerant, non-spore-forming Gram-positive bacilli, hitherto uncharacterised from human infections. Six isolates of *Exiguobacterium aurantiacum* were obtained from patients with bacteraemia.³²

In present study, the 16S rRNA sequencing of the isolates (PBCUUn05) confirmed the presence of *Exiguobacterium aurantiacum*, which is a new bacterium for corneal ulcers.

Shatila *et al.*,³³ have characterized *Exiguobacterium aurantiacum* but its 16S rRNA sequence was made up of 1500 base pairs. These estimates were slightly different from present study.

In present study comparison of different classes of antibiotics resistance was done by calculating % antibiotics resistance pattern. *E. aurantiacum* showed higher resistance to Aminoglycoside followed by Quinolones and Cephalosporin and higher sensitive to Cephalosporin+ Tobramycin combinations.

Microbial pigments have numerous beneficial properties. On the other hand, Pigment production may increase virulence in bacterial pathogens. These pigments have been shown to increase resistance to oxidative stress, killing by immune cells, and mutagenesis. Pigments seem to increase virulence of pathogens by increasing survival in immune cells, invasiveness and size of local abscesses.^{34,35}

In present study, *Exiguobacterium aurantiacum* was found to be Novel Bacteria and not reported earlier for the corneal ulcers were subjected to virulence study, which was done by testing pigment extraction. As per the literature, no researcher has reported about this bacterium from the cases of corneal ulcers.

Production of pigments appeared to be more significantly associated with multi-drug resistance, presence of virulence-associated genes, and expression of certain virulence factors, most notably elastase, protease, siderophore and DNase activity. Since pigment production is easy to determine, this might to be a good starting point to identify the virulence status of an isolate.³⁶

The FTIR analysis of methanolic extracts of *Exiguobacterium aurantiacum* confirmed that *Exiguobacterium aurantiacum* produced Carotenoid. Carotenoid may have evolved to reinforce bacterial membranes and when it inserted into a membrane, conjugated double bonds provide rigidity, serve to

stabilize it. Carotenoids are serving mechanical functions into pathogenic bacteria.

Several researchers have reported that some pigmented bacteria, which are rich in carotenoids, have been resistant to radiation when subjected to sub-lethal and lethal doses of ionizing radiations, due to the accumulation of the radio-protective pigments in the outer membrane.^{37,38}

Antioxidants play an important role in inhibiting and scavenging free radicals, thus providing protection to bacteria against various innate and adaptive immune defence mechanisms as well as antibiotics.

The present study showed that Carotenoid pigments have a potent nitric oxide scavenging activity. Carotenoid pigments showed a very good concentration-dependent inhibition of nitric oxide. These pigments showed virulent factor with an antioxidant action because these pigments neutralize the oxidants. These carotenoid pigments may be responsible for virulence of *Exiguobacterium aurantiacum* associated with the cases of corneal ulcers. Bacteria pigments protect themselves from oxidative immune attack.

In present study, with respect to Carotenoid pigment, the higher radical scavenging activity was found with 100 µg/ml concentrations with 73.37 values. Result of current study showed that, as the concentration of pigments increases, % inhibition increases with fall in absorbance. According to Balraj *et al.*, [39], radical scavenging activity increases with concentrations of carotenoid pigment.

In present study, Data analysis was performed by using SPSS software (Statistical Package for the Social Sciences). By using ANOVA (Analysis of variance) test, the levels of significance were determined using the number of independent variables in one way and two way.

To show there is significance difference among antibiotic resistance of different organism we have used two way anova, the p value was 0.005 which was less than 0.01 which shows that different antibiotics having different resistance on different organisms. Comparing the resistance of the drug except the Meropenem & Imipenem having the lesser resistance than the other drugs.

In present study we checked the association between the Severity of the Bacterial Ulcers and number of organisms. The p value was found 0.115 which was greater than 0.05 which shows that there is no association between severity and number of organism. So it revealed that the severity is due to type of bacteria.

Conclusion

The corneal ulcer is an ocular emergency that raises high-stakes questions about diagnosis and management. Rapid bacterial isolation and treatment with intensive ocular antibiotics represent decisive steps

in the management of such pathologies. Fluoroquinolones appeared to be the therapy of choice for bacterial keratitis but based upon these *in vivo* studies, some strains were resistant. Proper diagnosis and antibiotic selection requires for successful treatment of a corneal ulcer. For certain cases of bacterial keratitis fluoroquinolone antibiotics may be appropriate as empirical monotherapy treatment. As per the present studies observations, these days antibiotic resistance has been increased therefore combination therapies are suggested for the initial treatment of corneal ulcers. In present study, as per the literature referred, the bacteria *Exiguobacterium aurantiacum* was found to be Novel Bacteria which was not reported earlier for the cases of corneal ulcers and produced BAD colour.

Accession Number of Nucleotide Sequence: The complete genome sequence of *Exiguobacterium aurantiacum* deposited at the NCBI GenBank sequence database and got accessed as LC217383.

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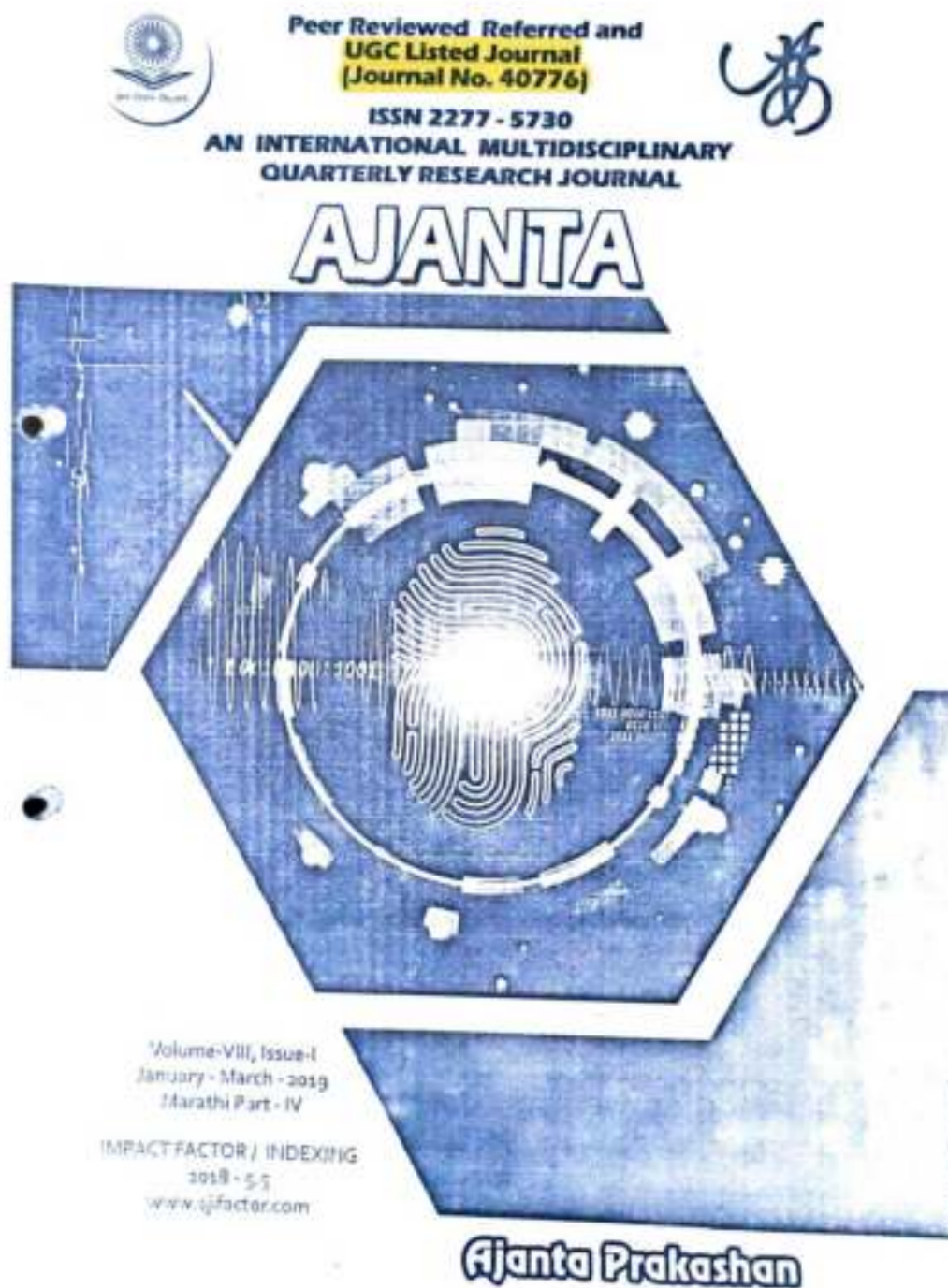
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❧ CONTENTS OF MARATHI PART - IV ❧

अ.क्र.	लेख आणि लेखकाचे नाव	पृष्ठ क्र.
१६	महात्मा ज्योतीराव फुले यांच्या शिक्षणविषयक विचारांची प्रामाणिकता आकाश शेखराव खांगर	६४-६६
१७	डॉ. बाळासाहेब आंबेडकरांचे राजकीय विचार अमोल भाऊराव बंड	६७-७०
१८	सामाजिक न्यायव्यवस्था विचारवंतांचे योगदान प्रा. दिगंबरकुमार प्रल्हादराव लांडगे	७१-७५
१९	पंडित जवाहरलाल नेहरू यांचे आदर्शावादी विचार कु. अलकाया ई. खानावाले प्रा. डॉ. जीवन पवार	७६-८०
२०	समाजसुधारणा राज्यव्यवस्था तुरुंगी ऐतिहासिक योगदान प्रा. डॉ. किशोर एच. बारापाडे	८१-८३
२१	मध्यमवर्गीय प्रमुख महिला संस्थांचे स्त्री कल्याणसंबंधी सामाजिक प्रयत्न प्रा. डॉ. एम. पी. खेडेकर	८४-८६
२२	महात्मा फुलेच्या विचारांतील सामाजिक व आर्थिक क्रांती प्रा. डॉ. मधुकर श्रीराम ताकतोडे	८७-९०
२३	डॉ. बाळासाहेब आंबेडकर कृषी अर्थव्यवस्थेसंबंधी विचार डॉ. उत्पल ना. पेडशीकर	९१-९२
२४	सामाजिक कामांमध्ये गो. व. आगरकरांचे योगदान डॉ. नाना वानखडे	९३-९६
२५	पं. नेहरुंचे लोकशाही विषयक विचार प्रा. नीता पांडे	९७-१००
२६	दलित व समाज परिवर्तन प्रा. विनय पैकिने	१०१-१०२
२७	बंडारा महिलांची आर्थिक व सामाजिक स्थिती प्रेमसिंह प्रल्हाद जाधव	१०३-१०५
२८	सामाजिक न्यायव्यवस्था विचारवंतांचे ऐतिहासिक योगदान राजेंद्र बापूराव वाकडे	१०६-११०
२९	महोत्तरीचा अधिष्ठाता आणि त्याचे महत्त्व प्रा. रमेश गुलाबराव मुरडकर	१११-११३
३०	महात्मा गांधी आणि शास्त्र विज्ञान डॉ. सवित्रा ज. वेले	११८-१२१

VOLUME - VIII, ISSUE - I, JANUARY - MARCH 2019
AJANTA - ISSN 2277 - 5738 - IMPACT FACTOR - 5.5 (www.ajfactor.com)

१७. डॉ. बाबासाहेब आंबेडकरांचे राजकीय विचार

अशोक भाऊराव शेंडे

सहायक प्राध्यापक, राज्यशास्त्र विभाग, आदर्श विद्यालय, बसवसगाव, अमरावती जिल्हा व विरला वडीलान्य महाविद्यालय,
भायलगाव रोड, जि. अमरावती.

सारांश

डॉ. भीमराव रामजी आंबेडकर विसाव्या शतकात होऊन गेलेले सुपरगुरु. डॉ. आंबेडकरांची संपूर्ण आयुष्यभर मानवीय दुर्घटनेला तोंड देत असतानाही ते हिंदू धर्मातील असलेल्या वाईट कलौडींमधील कठोर प्रहार केले. डॉ. आंबेडकरांनी स्वतः आपल्या जीवनामध्ये ज्या वाईट कलौडींमधील अनुभव घेतले, त्यामुळे त्या वाईट कलौडींमधील अविश्वसनीय संयुक्त नष्ट जाण्या याकरीता आपले विचार व कार्य कल्याणाच्या प्रयत्न सोडताना कल्याणात केला. समाजातील असुस्थता माझी, मुलभूत अधिकार तसले, सामाजिक दळी न देणे, हे दोष नष्ट करण्यासाठी त्यांनी आपले राजकीय विचार वेगवेगळ्या प्रमाणात व भाषणातून मांडले आहेत. जे की आजही उपयुक्त आहेत. आंबेडकरांनी घटना समितीकडे मनुष्य समितीचे अध्यक्ष म्हणून कार्य केले व इतर देशातील घटनांचा सूत्र अन्वय करून भारतीय परिस्थितीनुसार भारतीय जनतेकडे राजकीय संतोषबोधन सामाजिक व आर्थिक समता कधी निर्माण होईल यासाठी सर्व भारतीय नागरिकांना स्वातंत्र्य, समता, संयुक्त व सामाजिक न्याय देण्याचा प्रयत्न केला. त्याचप्रमाणे त्यांनी लोकशाहीसंबंधी सुद्धा सर्वोपरक असे विचार मांडले आहेत. की ज्यांचा वापर आजही केला तर भारतातील ज्या कठी समस्या आहेत-धर्मभेद, जातीवाद, प्रोत्साहन त्या सोडवण्या जाऊ शकतात व भारता महासत्ता बनू शकते.

प्रस्तावना

डॉ. भीमराव रामजी आंबेडकर विसाव्या शतकात होऊन गेलेले और समाजसुधारक, दलितसुधारक आणि राजकीय विचारवंत होते. आंबेडकरांना आपल्या संपूर्ण जीवनामध्ये जातीय असुस्थतेने चढके जास्वीर बसले होते. त्यामुळे बाबासाहेब आंबेडकरांनी आपल्या कार्य व विचारांमधून असुस्थत निवारण, समता, सामाजिक न्याय देण्यासाठी संपूर्ण आयुष्य खर्ची घातले. हे कार्य कल्याणासाठी त्यांनी राज्यघटना, तसेच वेगवेगळे संवसृष्ट कल्याण कार्य केले. आंबेडकरांनी जे आपले राजकीय विचार मांडले ते त्यांच्या संपूर्ण आयुष्याच्या अनुभवानुसार निर्माण झाले होते. त्यांच्या मते जोपर्यंत सामाजिक समता निर्माण होणार नाही, तोपर्यंत राजकीय समतास काही अर्थ उगणार नाही. त्यामुळे सामाजिक न्याय, स्वातंत्र्य, समता आणि संयुक्त ह्या गोष्टींचे महत्त्व त्यांनी आपल्या राजकीय विचारातून स्पष्ट केले आहे.

सौमन्यवाचे परिदृष्टी

- १) डॉ. बी. आर. आंबेडकरांच्या राजकीय विचारांचे अन्वयन.
- २) आंबेडकरांच्या राजकीय विचारांचे महत्त्व घटून देण्यासाठी.

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

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डॉ. अंबेडकरांचे राष्ट्र व राष्ट्रवादाविषयी विचार

डॉ. अंबेडकरांनी घोटस् ऑन पाकिस्तान, स्टेटस अँड मायनॅसिटिज, अँबिहिलेशन ऑफ कास्ट, रानडे, गांधी अँड जीना इत्यादी आपल्या ग्रंथांमधून राष्ट्रवादाविषयी विचार व्यक्त केले आहेत. त्यांच्या विचारांचे स्पष्टीकरण आपण खालीलप्रमाणे करू शकतो.

1) सामाजिक एकोपाची भावना म्हणजे राष्ट्रवाद

डॉ. अंबेडकरांच्या मते, देशातील संपूर्ण जनतेमध्ये सामाजिक एकोपा निर्माण होणे आवश्यक आहे. राष्ट्रातील संपूर्ण जनतेत संवाद, सहभाग आणि भागीदारी यातून सामाजिक एकोपा निर्माण होत असतो. तोच कोणत्याही प्रकारचा भेदभाव, भेद कनिष्ठत्वाच्या भावनेत वास नसतो.

2) राष्ट्रवाद ही किंवासक शक्ती नसून ती विधायक व रचनात्मक शक्तीही आहे

डॉ. अंबेडकरांच्या मते, राष्ट्रवादामुळे ज्वापरमाणे मोठमोठ्या राजवटी व साम्राज्य उत्पन्न पडू शकतात त्याचप्रमाणे, नवीन राष्ट्र निर्माण होऊ शकतात. राष्ट्र जमानीसाठी व राष्ट्रनिर्मितीसाठी राष्ट्रवाद हा चांगलाच प्रकारे कार्य करतो.

3) राष्ट्रवाद म्हणजे राजकीय ऐक्य नव्हे तर आर्थिक आणि सांस्कृतिक स्वरूपाचे ऐक्य

डॉ. अंबेडकरांनी राष्ट्रवादाविषयी विचार व्यक्त करताना असे म्हटले की, राष्ट्रवाद म्हणजे केवळ राजकीय ऐक्य नव्हे तर आर्थिक व सांस्कृतिक स्वरूपाचे ऐक्य म्हणजे सर्व भारतीयांच्या मनामध्ये सर्वांगीण सारखेपणाची भावना निर्माण झाली पाहिजे. सर्व व्यक्ती मग तो कोणत्याही जाती, धर्माचा, वंशाचा असो त्याला समतेची वागणूक मिळाली पाहिजे. प्रत्येकाचे समाजातील स्थान त्याच्या जातीधर्मानुसार न ठरता गुण कर्तृत्वावर ठरले पाहिजे. समाजातील प्रत्येक व्यक्तीच्या सुखदुखाविषयी इतरांना जाणीव असली पाहिजे.

4) राष्ट्रान्वये चळवळी व आंदोलनाच्या महर्ना विरोध

डॉ. अंबेडकरांना आंतरराष्ट्रीय क्षेत्रात तर सोळाव परंतु अंतर्गत राजकारणातही कोणत्याही समस्यांच्या विरोधात आंदोलन व चळवळीचे मार्ग मान्य नव्हते. कोणत्याही समस्यांचे निराकरण हे सनदशीर व योग्य मार्गानेच कायबास पाहिजे असे त्यांचे मत होते. कारण जेव्हा आंदोलन व चळवळीमुळे राष्ट्राची प्रगती खुटते आणि तसेच पायडे राष्ट्रान्वये पडतात.

अशाप्रकारे डॉ. अंबेडकरांना आक्रमक, अस्मिन्, अमानुष राष्ट्रवाद मान्य नव्हता. त्यांना धर्मेतिरपेक्ष कुटुंबाभावावादी व पुरोगामी राष्ट्रवाद मान्य होता. सर्व धर्मांना समान स्थान असायला पाहिजे असे त्यांचे मत होते.

डॉ. अंबेडकरांचे लोकशाहीसंबंधी विचार

डॉ. बबानाथेव अंबेडकर यांचे लोकशाही विषयी विचार अत्यंत स्पष्ट, रखडले आणि मानवावादी आहेत. डॉ. अंबेडकर हे भारतीय संविधान सभेतील बहुदा समितीचे अध्यक्ष होते व भारताची राज्यघटना त्यांच्या अधिक प्रदान व परिश्रमातूनच निर्माण झाली आहे. त्यामुळेच त्यांच्या विचारांचा प्रभाव हा भारतीय घटनेवर सुद्धा पडलेला आहे.

VOLUME - VIII, ISSUE - 1 - JANUARY - MARCH - 2019
AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.ajfactor.com)

1) डॉ. अंबेडकरांच्या मते भारताने लोकशाही निर्माण करून इतर लोकशाही राष्ट्रांशी सहकार्य करावे.

डॉ. अंबेडकरांच्या मते भारताने भारतात लोकशाहीची उभारणी करून इतर लोकशाही राष्ट्रांशी सहकार्य करावे आणि लोकशाही मूल्ये संदर्भित करणे म्हणजे समाजात स्वातंत्र्य, समता, बंधुता, सामाजिक न्याय, व्यक्ती व राष्ट्रांशी प्रतिष्ठा यांची जपवणूक करावी.

2) लोकशाहीमध्ये सत्ता ज्यांना कधी सत्तेचा स्पर्श झाला नाही अशा लोकांना मिळावला पाहिजे

डॉ. अंबेडकरांच्या मते प्रत्येक देशात एक सत्ताधारी वर्ग असतो, परंतु स्वातंत्र्योत्तर काळातही सत्ता त्यांच्याच ताब्यात येवी तर इतर जनतेवर अन्याय होईल. म्हणून खरी लोकशाही निर्माण करायची असेल तर ज्यांना कधीही सत्तेचा स्पर्श झाला नाही अशा समाजाच्या खात्याचा बळत खिंतवत पडलेल्या पडदस्तित्त वर्गास सत्ता प्राप्त झाली पाहिजे.

3) लोकशाही म्हणजे एक व्यक्ती एक मूल्य

डॉ. अंबेडकरांच्या मते खरी लोकशाही जर निर्माण करायची असेल तर एक व्यक्ती एक मत तर आर्सेक, परंतु एक व्यक्ती एक मूल्य अशी परिस्थिती निर्माण झाली पाहिजे म्हणजे प्रत्येक व्यक्तीला सन्मान व समान स्थान प्राप्त झाले पाहिजे समाजात ज्ञाती, धर्म पंथ यांच्यामध्ये कोणत्याही प्रकारचा भेदभाव असायला नको व समाजात कोणत्याही विभेदाधिकार मिळावला नको.

4) लोकशाहीमध्ये विरोधी पक्ष व विरोधी मूल्यजांना फार महत्वाचे स्थान

डॉ. बाबासाहेब अंबेडकरांच्या मते, संसदीय लोकशाहीमध्ये दोन पक्ष असतात, एक म्हणजे सत्ताधारी पक्ष व दुसरा म्हणजे विरोधी पक्ष ज्याकडतीने सत्ताधारी पक्षाला महाव असतो तेवढेच महत्त्व खंबीर व प्रबळ विरोधी पक्षाला सुद्धा असते. विरोधी पक्ष जर शक्तीशाली असेल व तो प्रामाणिकपणे आपली जबाबदारी पार पाडत असेल तर सत्ताधारी पक्ष हुकुमचाह प्रमाणे कार्य करणार नाही.

5) दारिद्र्य, जातीयता व निस्वसत्ता ही भारतीय लोकशाहीच्या वाटपांतील अडथळे.

डॉ. अंबेडकरांच्या मते भारतीय लोकशाहीतील अडथळे म्हणजे दारिद्र्य, जातीयता व निस्वसत्ता, दारिद्र्य व्यक्ती भुर्केल्या पोटी आपली जबाबदारी पार पाडू शकणार नाही, त्याप्रमाणे जातीयता, की ज्यामुळे योग्य व्यक्तीची निवड न होता बहुसंख्याक आपल्याच बहुसंख्याक व्यक्तीची निवड करतील म्हणजे अल्पसंख्याक व्यक्तींचा अनी गुण कर्तुत्व असूनही त्याला महत्त्व उरणार नाही व शेवटी निस्वसत्ता जो व्यक्ती सासरच नसेल त्याला काहीही समजणार नाही, म्हणून तो योग्य निर्णय घेऊ शकणार नाही, या सर्व समस्यांचे निर्मूलन आपण जनतेला मोक्ष व योग्य शिक्षणाच्या माध्यमातून करू शकतो.

6) सत्तांतर करण्यासाठी मुक्त व न्याय्य निवडणुका

डॉ. अंबेडकरांच्या मते संसदीय तारान पद्धतीमध्ये सत्तांतर करण्यासाठी मुक्त व न्याय्य निवडणुकांना फार महत्त्व आहे. निवडणुका ह्या मुक्त वातावरणात जात्या पाहिजे. जनतेवर कोणत्याही प्रकारचा दबाव असायला काय नये व ह्या न्याय्य पद्धतीनुसार पार पडल्या पाहिजेत म्हणजे त्यामध्ये कोणत्याही प्रकारचा पक्षपात होत काय नये.

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.ajifactor.com)

७) लोकशाहीच्या यथाकशिता कायद्याबरोबरच घटनात्मक नीतिमतेलाही फार महत्त्व.

श्री. अंबेडकरांच्या यथे लोकशाही दिकानायची असेल तर लोकशाहीमध्ये कायद्याला तर मान्य असतेच. कायद्यानुसारच सरकार यमनवीरित्य चालू असावी. परंतु त्याचबरोबर घटनात्मक नीतिमतेलाही फार महत्त्वाचे स्थान असते. लोकशाही सदसद्विवेकबुद्धी हा लोकशाहीचा फार महत्त्वाचा घटक आहे.

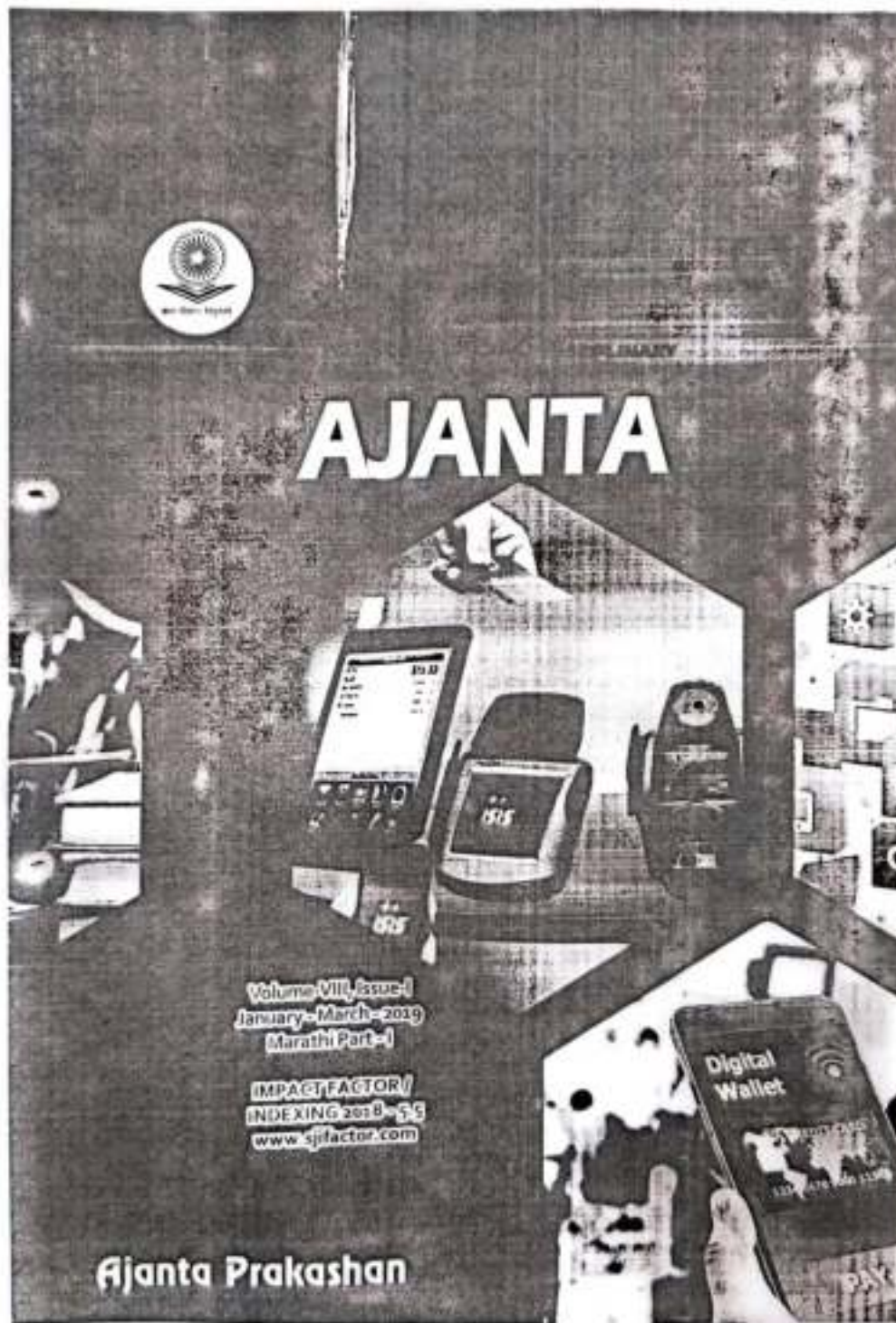
निष्कर्ष

श्री. बाबासाहेब अंबेडकर हे थोर मान्यतावादी विचारवंत व समाजसुधारक होते. त्यांनी आपल्या विचार आणि कर्तव्याद्वारे भारतीय समाजातील अनिष्ट प्रथा, वाईट चालीरिती मोठ्या प्रमाणात नष्ट केल्या. श्री. अंबेडकरांचे विचार राष्ट्रनिर्माण व राष्ट्रउपासणीसाठी आजही मोठ्या प्रमाणात उपयुक्त आहेत. श्री. अंबेडकरांच्या राजकीय विचारांचा निष्कर्ष आपण काढला तर आपल्याला असे लक्षात येईल की त्यांच्यामार्गे सर्व जाती, धर्म, पंथ, पंथ यांना समान स्थान असण्याला पाहिजे त्याचप्रकारे कोणत्याही प्रकारचा भेदभाव त्यांना मान्य नाही. सर्व जंतुमध्ये सामुष्टिक एकीय व आर्थिक एकता निर्माण व्हायला पाहिजे. प्रत्येक व्यक्तीमध्ये समान राजकीय लोकशाहीबरोबर सामाजिक लोकशाही निर्माण व्हायला पाहिजे. समाजातील प्रत्येक व्यक्तीला सन्मानाची व आदराची वागणूक मिळायला पाहिजे. तेव्हाच कोणते समाजाची प्रगती होऊ शकते.

संदर्भ

१. राजकीय सिध्दांत आणि राजकीय विचारक – प्रा. शेख हादुम, प्रा. जोगेंद्र रावई
२. यांनी घडविला भारत – गो. द. पहिनकर
३. आधुनिक भारतातील राजकीय विचार – श्री. भास्कर लक्ष्मण भोळे
४. समाजशास्त्र – इकता बारावी
५. राष्ट्र उपासणीत श्री. अंबेडकरांचे योगदान लोकांसाठी
६. अंबेडकरवादसमोरील प्रतिक्रितीचे आग्रहान लोकमत (६ डिसेंबर 2018)
७. श्री. बाबासाहेब अंबेडकर यांचे राजकीय विचार – संकरराय खरत

Bhartiya Sansdiya shasan Paddhatil Dosh ani Upay : Amol Bhauraoji Band



VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

❧ CONTENTS OF MARATHI PART - I ❧

अ.क्र.	लेख आणि लेखकाचे नाव	पृष्ठ क्र.
१	कविता : आकलन व आस्वादाची काही सुत्रे प्रा. विजय वाघमारे	१-३
२	भारतातील कर सुधारणा - एक विविक्षितक विश्लेषण : जी. एस. टी. संदर्भ प्रा. डॉ. उषा एन. पाटील	४-९
३	मानवी जीवनात पोषणचे महत्त्व आणि पुरक पोषण आहार विषयक शासकीय योजना - अध्ययन प्रा. डॉ. रश्मी प्रविण मजरे	१०-१३
४	आधुनिक शिक्षण प्रणालीत बुद्धीच्या विचारांची गरज प्रा. डॉ. विष्णूजीत कांबळे	१४-२३
५	समाज परिवर्तनात सामाजिक माध्यमांची भूमिका समाजशास्त्रीय अध्ययन प्रा. डॉ. सुधा म. खडके (काबु)	२४-२९
६	अभिेडकरवादी कवितेचे सौंदर्यशास्त्र प्रा. डॉ. अशोक कांबळे	३०-३२
७	ऑनलाईन मार्केटींगमुळे भारतीय अर्थव्यवस्थेची बदललेली परिस्थिती प्रा. रुपेश म. कुन्नेकर	३३-३७
८	हरित प्रचलने कु. मिनल व्ही. गाडवे प्रा. डॉ. के. ओ. मांडगावकर	३८-४१
९	भारतीय अर्थव्यवस्थेत कृषिचे स्थान प्रा. खुशाल विश्वनाथ डवळे	४२-४४
१०	काचनकौशल्य सर्वांगीण समृद्धी डॉ. एकता अ. मेनकुदळे	४५-४९
११	भारतीय सामंतीय शासन पद्धतीमधील दोष आणि उपाय अमोल भाऊराव बोंड	५०-५४
१२	मानवी जीवनात आहाराचे महत्त्व प्रा. डॉ. विभा छ. घोडखाने	५५-६०
१३	भारतातील रस्ते विकास योजनेचे ग्रामीण विकासातील योगदान दीपक दत्तात्रय निलावार	६१-६६

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

११. भारतीय सांसदीय शासन पद्धतीमधील दोष आणि उपाय

अमोल भाऊराव बंड

सहाय्यक प्राध्यापक, राज्यशास्त्र, आदर्श महाविद्यालय, धामणगाव रेल्वे, जि. अमरावती.

सारांश

भारताने इंग्रजांच्या गुलामगिरीतून मुक्त झाल्यानंतर लोकशाही शासन पद्धतीचा स्वीकार केला. अब्राहम लिंकनच्या मते, लोकशाही म्हणजे "लोकांचे, लोकांनी आणि लोकांकरिता चालविलेले शासन". असे भारतीयांच्याद्वारे भारतीयांसाठी चालविलेले शासन. परंतु या लोकशाहीचे सुद्धा दोन प्रकार पडतात. १) प्रत्यक्ष लोकशाही व २) अप्रत्यक्ष लोकशाही यातही भारताने अप्रत्यक्ष म्हणजे प्रातिनिधीक लोकशाहीचा स्वीकार केला. प्रातिनिधीक लोकशाहीचेही दोन प्रकार पडतात. एक सांसदीय लोकशाही व अभ्यक्षीय लोकशाही. भारताने सांसदीय लोकशाहीचा स्वीकार केला. सांसदीय लोकशाहीचा स्वीकार केल्यामुळे भारतीय कायदेमंडळाला म्हणजेच संसदेला एक विशेष महत्त्व प्राप्त झाले. भारतीय संसद ही द्विगुही आहे. एक म्हणजे लोकसभा व दुसरे सभागृह राज्यसभा. लोकसभा हे जनतेचे प्रतिनिधीत्व करते तर राज्यसभा घटकराज्यांचे प्रतिनिधीत्व करते. ही दोन्ही सभागृह मिळून संसद तयार होते. संसद म्हणजे जनतेच्या समस्या सोडवणारी लोकशाहीचे मंदिर. लोकशाहीमध्ये कायदेमंडळाला मध्यवर्ती भूमिका पार पाडावयाची असते. जनतेच्या समस्या, अडचणी, विकास कार्य यावर चर्चा करून, प्रसंगी वादविवाद करून सुसंबाद पडवून आणावयाचा असतो. परंतु आपण मागील दोन दशकांपासून जर संसदेचे चित्र पाहिले तर ही संसदीय लोकशाही आहे की गुंडशाही हे समजतच नाही. संसदेचे कामकाज बंद पाडल्या जाते. संसदेचे कामकाज हे जनतेच्या कराच्या पैशांवर चालते. त्यामुळे तेथे जनतेच्या प्रश्नावर चर्चा झालीच पाहिजे. त्याकरिता संसद सदस्य म्हणजे खासदारांमध्ये जोपर्यंत जबाबदारी व कर्तव्याची भावना निर्माण होत नाही, कामकाजावर करोडो रुपये खर्च होतो याची जाणीव त्याचप्रमाणे राष्ट्रप्रेम व राष्ट्रहीत व संसद चालावी याकरिता नवीन कायदे जोपर्यंत केले जात नाही, तोपर्यंत संसदेचे कामकाज योग्य पद्धतीने चालू शकत नाही. व संसदेला लोकशाहीतील मंदिराचे स्वरूप प्राप्त होऊ शकत नाही.

प्रस्तावना

भारत इंग्रजांच्या गुलामगिरीतून मुक्त झाल्यानंतर लोकशाही शासन पद्धतीचा स्वीकार केला. लोकशाहीमध्ये प्रत्यक्ष लोकशाही व अप्रत्यक्ष लोकशाही असे दोन प्रकार आहेत. अप्रत्यक्ष लोकशाहीचे म्हणजेच प्रातिनिधीक लोकशाहीचे सांसदीय व अभ्यक्षीय असे दोन प्रकार पडतात. भारताने सांसदीय लोकशाहीचा स्वीकार केला. सांसदीय शासन पद्धतीमध्ये कायदेमंडळ, कार्यकारी मंडळ या शासन शाखामध्ये कोणत्या प्रकारचा संबंध असावा या विषयी निश्चित तरतूद पुरविते. सांसदीय शासन पद्धतीमध्ये कायदेमंडळ व कार्यकारी मंडळ यांमध्ये घनिष्ठ संबंध असतो. या शासनपद्धतीमध्ये दोन प्रमुख असतात. त्याचप्रमाणे सामुदायिक जबाबदारीचे तत्त्व येथे पाळले जाते. अशा वैशिष्ट्यपूर्ण सांसदीय शासन पद्धतीचा स्वीकार भारताने

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

केला. त्यामुळे भारताच्या कायदेमंडळाचा महत्त्वपूर्ण स्थान प्राप्त झाले, परंतु आपण स्वतंत्र मिळाल्यापासून काही वर्षां आणि मागील दोन दशके याची तुलना जर कायदेमंडळाच्या बाबतीत केली तर सुरुवातीच्या काळात म्हणजे पंडित जवाहरलाल नेहरू, लालबहादूर शास्त्री, इंदिरा गांधी, राजीव गांधी, अटलबिहारी वाजपेयी यांच्याकाळात आणि वर्तमान काळात जर आपण कायदेमंडळाची तुलना केली तर सुरुवातीचा काळ हा कायदेमंडळाचा सुवर्ण काळ होता. त्यावेळेस जनतेच्या समस्यांचे, अडचणीचे, विदेश नीती आणि कायद्यांची निर्मिती यांच्या संदर्भात संसद सदस्यांमध्ये मोठ्या प्रमाणात चर्चा, वादविवाद होत होते. आणि त्यामधून सुसंवाद होऊन योग्य मार्ग काढल्या जात होता. परंतु वर्तमान काळात जनतेच्या समस्याविषयी कोणतीच चर्चा न होता, फक्त घोषणा गोंधळ, कामकाज बंद पाडणे इतकेच कार्य केले जाते. म्हणून वर्तमान काळात भारतातील सांसदीय शासन पद्धती ढासाळलेली आहे हे स्पष्ट होते.

शोधनिबंधाची उद्दिष्टे

- १) कायदेमंडळाचे महत्त्व स्पष्ट करण्यासाठी
- २) कायदेमंडळातील प्रतिनिधी मार्फतच जनतेच्या समस्या सुटू शकतात हे पटवून देण्यासाठी.
- ३) कायदेमंडळाचे कामकाज बंद पाडणे ही किती गंभीर समस्या आहे याचे अध्ययन करण्यासाठी.

भारतीय संसदीय पद्धती

भारताने संसदीय पद्धतीचा स्वीकार केला असून भारतीय कायदेमंडळाचा संसद असे म्हणतात. भारतीय संविधानाच्या पाचव्या भागात प्रकरण २ मध्ये कलम ७९ ते १२२ मध्ये भारतीय संसदेची तरतुद करण्यात आलेली आहे. भारतीय संविधानामध्ये कलम ७९ नुसार संघराज्यासाठी एक संसद असेल आणि राष्ट्रपती राज्यसभा व लोकसभा मिळून ती बनलेली असेल. भारतीय संसद ही ब्रिगुडी अगुन लोकसभा हे कनिष्ठ व प्रथम सभागृह आणि राज्यसभा हे बरिष्ठ व द्वितीय सभागृह आहे. लोकसभा हे जनतेचे सभागृह व राज्यसभा हे घटक राज्यांचे सभागृह आहे.

संसदेच्या कामकाजाची पद्धती

संसदेच्या कामकाजाचे नियम संविधानाच्या ११८ व्या कलमानुसार तयार केलेले आहेत. संविधानानुसार संसदेच्या कामकाजाचे प्रस्तावना, लक्ष्येची सूचना, स्वयं प्रस्ताव, शुद्धप्रहर असे भाग आहेत. त्याचप्रमाणे संसदेचे वर्षातून तीन अधिवेशन होतात. आवश्यकता वाटल्यास अतिरिक्त अधिवेशन बोलावण्याची तरतुद आहे. या तीन अधिवेशनांमध्ये,

- १) अर्थसंलयीय अधिवेशन – फेब्रुवारी ते मे
- २) पावसाळी अधिवेशन – जुलै ते सप्टेंबर
- ३) हिवाळी अधिवेशन – नोव्हेंबर ते डिसेंबर

संसद ही कायदा करणारी अंतिम संस्था आहे. जनतेच्या इच्छा, आकांक्षा, समस्या या सर्वांचे प्रतिबिंब संसदेत उमटत असते. संसद म्हणजे नवीन कायदे निर्माण करणारी आणि जुन्या कायद्यांमध्ये दुरुस्ती करणारी संस्था आहे परंतु संसदेची संख्ये संख्या मोठ्या प्रमाणात असल्यामुळे सर्वत्र सदस्यांना आपले मत मांडता येणार नाही. त्याकरीता नेमलेल्या सांसदीय समित्या निर्माण करण्यात येतात. त्यामुळे सभागृहांना वेळ वाचून कार्यक्षमतागृह वाढीस लागते.

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

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भारतीय सांसदीय शासन पद्धतीतील दोष**१) कायदेमंडळांमध्ये घोषणाबाजी व गोंधळ**

भारतीय कायदेमंडळ म्हणजे संसदेच्या कामकाजाचा अभ्यास आपण केला तर आपल्या असे निदर्शनास येईल की, गेल्या दोन दशकांपासून संसदेचे कामकाज फारच कमी झाले आहे. सभागृहाचा जास्तीत जास्त वेळ गोंधळ व घोषणाबाजीमध्येच जातो. युपीए सरकारच्या कारकीर्दीत दोन्ही वेळा भाजपाने संसदेत भरपूर गोंधळ घालून कामकाज बंद पाडले. सन २००५ मध्ये १६ टक्के, २००६ मध्ये १६ टक्के, २००७ मध्ये ११ टक्के, २००८ मध्ये १७ टक्के, २००९ मध्ये ८ टक्के, २०१० मध्ये १२ टक्के, २०१२ मध्ये १४ टक्के, २०१३ मध्ये १२ टक्के, २०१४ मध्ये १३ टक्के, २०१५ मध्ये ३० टक्के २०१६ मध्ये १७ टक्के २०१७ मध्ये ३४ टक्के २०१८ मध्ये १ टक्का लोकसभेत कामकाज झाले.

राज्यसभेत सन २००४ मध्ये ३ टक्के २००५ मध्ये १६ टक्के, २००६ मध्ये ५ टक्के २००९ मध्ये ८ टक्के, २०१० मध्ये ११ टक्के, २०१६ मध्ये १६ टक्के २०१३ मध्ये १२ टक्के २०१४ मध्ये १६ टक्के, २०१५ मध्ये २२ टक्के, २०१६ मध्ये १५ टक्के आणि २०१७ मध्ये १९ टक्के कामकाज झाले. सन २०१८ मध्ये ६ टक्के.

लोकसभेत किमान वर्षातून १०० दिवस कामकाज व्हावे असे अपेक्षित आहे. पूर्वी १२७ दिवस कामकाज चाललेले आहे. आता वर्षातून ३५ दिवसही काम होत नाही. हे दुर्दैव आहे. पूर्वी संसदेत वर्षाला ७० ते ८० विधेयक संमत होत असत. आता जवळजवळ पंधरा ते बीस होतात. पूर्वी संसदेत हुल्लडबाजी करणाऱ्या सदस्यांना मार्शलकडून सभागृहाच्या बाहेर काढले जात असे. आता संसदेत रोजच गोंधळ घातल्या जातो. त्यांच्यावर काहीच कारवाई होत नाही.

२) सांसदीय कामकाज मंत्र्याची भुमिका

सांसदीय कामकाज मंत्र्याचे पद हे फार महत्वाचे पद आहे. कारण सांसदीय कामकाजमंत्री हे सत्ताधारी पक्ष व विरोधी पक्ष यांच्यामधील दुवा असतो. सांसदीय कामकाज मंत्र्यांना विरोधी पक्षाला विश्वासात घ्यायचे असते. किंबहुना आपला जास्तीत जास्त वेळ हा विरोधी पक्षासोबत घालवायचा असतो. जेणेकरून कामकाज बंद पडणार नाही व जनतेच्या कष्टाचा पैसा वाया जाणार नाही.

३) संसद सदस्यामध्ये जबाबदारीच्या व कर्तव्याच्या भावनेचा अभाव

संसद सदस्यामध्ये आपल्या कर्तव्याप्रती आणि जबाबदारीच्या भावनेचा अभाव दिसून येतो. आपल्याला जनतेने जनतेच्या समस्या सोडविण्यासाठी आपल्या आशा, आकांक्षा पूर्ण करण्यासाठी पाठविलेले आहे. आपल्या मतदार संघातील समस्या, देशातील समस्या, बेरोजगारी, कुपोषण, पर्यावरण प्रदूषण स्त्रियांची सुरक्षितता याविषयी जाण प्रतिनिधीमध्ये नाही. त्याचप्रमाणे, संसदेच्या एका दिवसाच्या कामकाजावर रोज किमान अडीच कोटी रुपये खर्च होतो. प्रत्येक मिनिटाच्या कामकाजावर दिड लाख रुपये खर्च होतो. आणि हा सर्व पैसा जनतेच्या कराच्या स्वरूपात आहे. याची जाणीव संसद सदस्यांना असायला हवी.

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.ajifactor.com)

४) गरिब देशाचे करोडपती लोकप्रतिनिधी

भारतीय संसदीय शासन पध्दतीतील महत्वाची उणीव म्हणजे भारतीयामध्ये गरिब लोकांचे प्रमाण जास्त आहे. गरिब लोकांच्या समस्या मांडण्यासाठी करोडपती प्रतिनिधीची निवड केली जाते. गरिब जनतेच्या समस्या करोडपती लोकांना काय कळणार ही फार मोठी उणीव आहे.

५) तज्ञ व बुध्दीजीवी सदस्यांचा अभाव

भारतीय संसदीय शासन पध्दतीतील एक महत्वाचा दोष म्हणजे तज्ञ व बुध्दीजीवी सदस्यांचा अभाव सभागृहात दिसून येतो. पूर्वीच्या काळी म्हणजे १९५२ पासून २००० पर्यंत पंडित नेहरू, राममनोहर लोहिया, अटलबिहारी वाजपेयी यांसारखे प्रतिनिधी होते. यांसारखे अभ्यासू आणि लोककल्याण हाच ज्याच्या प्रतिनिधीत्वाचा उद्देश होता तसे प्रतिनिधी आता दिसत नाहीत.

१९६३ मध्ये राममनोहर लोहिया यांनी संसदेमध्ये एक महत्त्वपूर्ण खुलासा केला की, देशामधील जास्तीत जास्त लोकसंख्या रोज तीन आण्यामध्ये आपला उदरनिर्वाह करते. परंतु प्रधानमंत्री जवाहरलाल नेहरू यांच्यावर रोज २५,००० रु. खर्च होत होता. जवाहरलाल नेहरूनी उत्तर दिले की, योजना आयोगाच्या मते ही रक्कम १५ आणे म्हणजे आजच्या रूपरुपापेक्षा कमी आहे. लोहियांनी या मुद्द्यावर विरोध घेवैची मान्यता केली आणि जेव्हा ते बोलतयला उठले तेव्हा एका मागेमाग एक अनेक संसद सदस्यांनी आपल्या बोलण्याचा वेळ लोहियांना दिला. आणि लोहियांना योजना आयोगाच्या अकड्यांचे पितळ उपळे फाडले. अशा संसद सदस्यांची भारताला आता गरज आहे.

६) कास्टिंग काऊच

भारतीय संसदीय शासन पध्दतीतील आणि एक उणीव म्हणजे आतापर्यंत आपल्याला चित्रपट सुद्धीत जे प्रकार दिसत होता, तो म्हणजे कास्टिंग काऊच हा प्रकार काँग्रेसच्या माजी खासदार रणुका चौधरी यांनी तर संसदेतही चालतो असा खळबळजनक आरोप केला आहे. यामुळे जनतेच्या समस्या कशा सुटतील असा प्रश्न पडतो.

संसदीय शासन पध्दतीतील उणिवा दूर करण्यासाठी पुढील उपाय करता येईल.

१) गोंधळ करणाऱ्यावर कठोर कारवाई

भारतीय संसदेमध्ये सकाळी ११ वाजता कामकाज चालू झाले की, दोन्ही सभागृहांमध्ये गोंधळ, बॅरर्स भेकून सभागृहाच्या आसनासमोर जाणे, अशा प्रतिनिधीवर कठोर कारवाई करायला हवी. त्यांचे सदस्यत्व रद्द करणे, पुढील निवडणुकीमध्ये पक्षाने टिकिट न देणे त्यांचे वेतन व निवृत्तीवेतन बंद करणे अशाप्रकारची कारवाई करायला हवी.

२) उपस्थिती अनिवार्य

भारतीय संसदीय शासन पध्दतीतील महत्वाची उणीव म्हणजे सदस्यांची अल्प उपस्थिती १९९१ मध्ये ५२२ पैकी केवळ १४६ सदस्य हजर होते. २०१२ मध्ये संसदेच्या हीरक सभोसबाविमिता संसदेची विशेष बैठक बोलाविण्यात आली. यावेळी सभागृहात सर्वां सुरू असतांना सत्ताधारी आणि विरोधी सदस्यांच्या बहुतांश खुर्चा रिकाम्याच होत्या. अशा सभागृहातील उपस्थिती अनिवार्य करायला पाहिजे.

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
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३) उपस्थिती आणि वेतन यांचा संबंध

कायदेमंडळातील सदस्यांची उपस्थिती आणि वेतन यांचा संबंध असावा. कारण बरेचशे सदस्य सभागृहामध्ये उपस्थित नसतात. तरीसुद्धा त्यांना वेतन मिळते. म्हणून जे सदस्य सभागृहात हजर राहत नाहीत त्यांना वेतन सुद्धा मिळायला नको.

निष्कर्ष

भारतीय सांसदीय शासन पद्धतीतील कायदेमंडळाची भूमिका ही महत्वाची असते. कारण कायदेमंडळ हे जनतेच्या आशा आकांक्षांचे, समस्यांचे प्रतिबिंब असते. देशाची संसद ही सव्वाशे कोटी जनतेच्या भावभावनांचे प्रतीक आहे. संसदेच्या सभागृहात जनतेच्या आशाआकांक्षांचे प्रतिबिंब पडले पाहिजे. परंतु प्रत्यक्षात तसे न होता संसद सदस्य सभागृहाचे कामकाज चालू झाले म्हणजे अकरा वाजता कामकाज सुरू होते. बरोबर अकरा वाजता राज्यसभेत 'अध्यक्ष महोदय' व लोकसभेत 'सभापती महोदय' असा पुकार झाला की, दहा पंधरा खासदार आपआपल्या जागा सोडून पुढे येत व बॅनर्स फडकावून घोषणाबाजी सुरू करतात. यापद्धतीने संसदेचे कामकाज बंद पाडल्या जाते.

याचा अर्थ असा नक्कीच नव्हे की, संसदेत विरोधी पक्षाचे खासदार असाच गोंधळ घालतात व पूर्वी अगदी चांगले दिवस होते. १९५२ साली पहिल्या लोकसभेत सुद्धा गोंधळ होत असे व त्याकाळी सुद्धा गोंधळामुळे संसदेचा बहुमूल्य वेळ वाया गेलेला आहे. मात्र तेव्हा त्याचे प्रमाण फक्त ५ टक्के होते. आज गोंधळाचे प्रमाण ९५ टक्के आहे. अर्थसंकल्पीय अधिवेशनात संसदेचे कामकाज २४ दिवस ठप्प राहिले म्हणून संसदेचे कामकाज योग्य पद्धतीने पार पाडले पाहिजे, जनतेच्या समस्यांचे समाधान झाले पाहिजे, लोककल्याणकारी योजना आखल्या गेल्या पाहिजे. त्याकरीता संसद सदस्यांची मानसिकता निर्माण झाली पाहिजे. त्याचप्रमाणे, संसद सदस्यांमध्ये जनतेच्या पैशाबद्दल जाणीव निर्माण झाली पाहिजे. कारण संसदेचे कामकाज जर ठप्प झाले तर जनतेच्या पैशाचा अपव्यय होतो. ही भावना जोपर्यंत संसद सदस्यांमध्ये निर्माण होत नाहीत तो पर्यंत संसद योग्य मार्गाने कामकाज पार पाडणार नाही.

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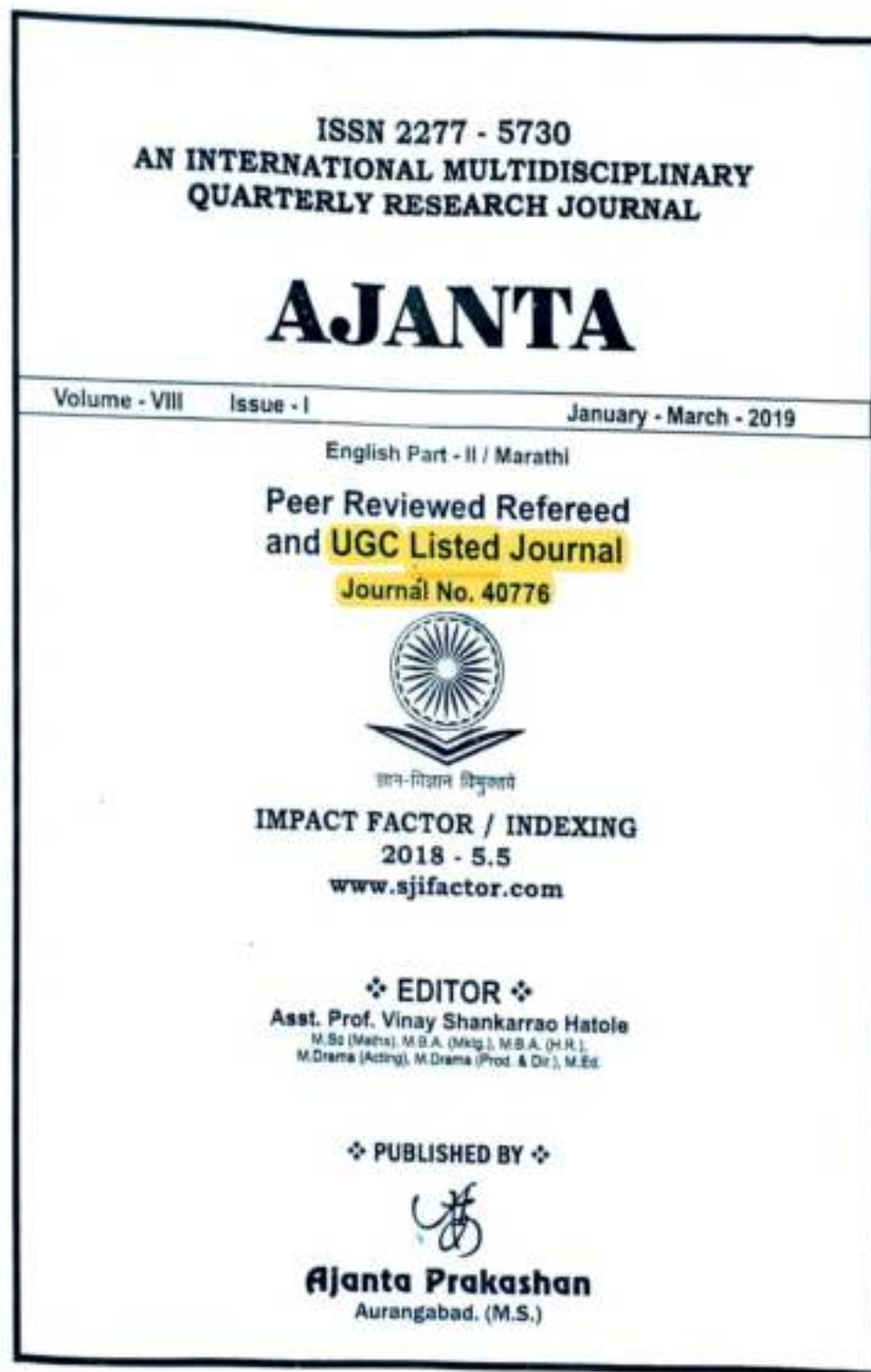
Sl.No.	Journal No.	Title	Publisher	ISSN	E-ISSN
920	64714	Air, Water and Soil Pollution	Springer	0049697X	1573293X
921	3164	Aircraft Engineering and Aerospace Technology	Emerald Group Publishing Ltd	17488842	17584213
922	3165	Airline Business	Reed Business	2687615	
923	63597	AIS Transactions on Human-Computer Interaction	Association for Information Systems	19443900	
924	3167	AIST Today (International Edition)	National Institute of Advanced Industrial Science and Technology	1346602X	
925	3168	Aisthesis	Pontificia Universidad Catolica De Chile, Facultad De Filosofia, Instituto De Estetica	5683939	7187181
926	41067	Alwan-E- Urdu	Delhi Urdu Academi, Delhi		
927	3169	Ajalooline Ajakiri	Tartu University Press	14063859	22283897
928	40776	Ajanta	AJANTA PRAKASHAN, AURANGABAD	22775730	
929	3171	Ajatus	Philosophical Society of Finland	3551725	
930	40742	Ajker Jodhan	Kolkata	9715819	
931	3173	AJOB Empirical Bioethics	Taylor and Francis Ltd	23294515	23294523
932	3174	AJOB Neuroscience	Taylor and Francis	21507740	21507759
933	3176	AJP Reports	Thieme Medical Publishers	21576998	21577005
934	3177	AJS Review	Association for Jewish Studies	3640094	14754541
935	42274	Akadmi			
936	3179	AKCE International Journal of Graphs and Combinatorics	Kalasalingam University	9728600	
937	3180	Akkadica	Assyriological Cent Georges Dossin	13785087	7797842
938	42266	Aks-E-Adab' (Quarterly)		23206519	
939	40702	Akshargatha	Dr.M.M.Jadhav Nanded		9762957
940	3182	Aktuality v Nefrologii	Tigis Sro	1210955X	
941	3183	Aktualnosci Neurologiczne	Malgorzata Pachecka	16419227	
942	3186	Aktuelle Dermatologie	Georg Thieme Verlag	3402541	
943	3189	Aktuelle Ernährungsmedizin Klinik und Praxis	Georg Thieme Verlag	3410501	14389916
944	3193	Aktuelle Neurologie, Supplement	Georg Thieme Verlag	14372754	
945	3197	Aktuelle Rheumatologie	Georg Thieme Verlag Kg	0341051X	14389940
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Argo Entrepreneurship: A vital tool for sustainable agriculture: Anand Gangadhar Naranje





VOLUME - VIII, ISSUE - I - January - March - 2019

AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

CONTENTS OF ENGLISH PART - II

S. No.	Title & Author	Page No.
1	Emerging Trends in E Commerce: An Outline Dr. Yogesh L. Patinge	1-4
2	Changing Trends of Indirect Taxes in India Swati A. Saurangpate	5-12
3	Skill Development: Challenges and Opportunities Prof. Dr. Sunil N. Dhere	13-16
4	Comparative Study of VAT and GST in Indian Pharmaceutical Sector Industry Dr. Anil Marotrao Raut	17-21
5	A Skill Development - A Step towards Kaushal Bharat Dr. P. S. Vishwakarma	22-26
6	Business Ethics: An Integrated Approach Dr. Naglaxmi Narsingrao Tirmanwar	27-30
7	Role of Skill India in Economic Development Dr. Ravindra Bhaurao Tembhurne	31-35
8	Advertising World with Augmented Reality Technology Prof. Dr. Aruna R. Chudasama	36-42
9	Emerging Trends in Indian Agricultural Dr. Ulhas N. Medshikar	43-48
10	Impact of Demonetization on Retail Sector Prof. Kishor Narayanrao Tayade	49-53
11	Impact of Goods and Services Tax on Manufacturing Sector with Reference to Amravati City Dr. Nitin Wamanrao Jaswante	54-58
✓ 12	Agro Entrepreneurship: A Vital Tool for Sustainable Agriculture Dr. A. G. Naranje	59-61
13	E-Business - Challenges and Opportunities Dr. Narendra K. Nagpure	62-65

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.ajifactor.com)

12. Agro Entrepreneurship: A Vital Tool for Sustainable Agriculture

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Abstract

The rural development in India is an important segment of economic development. Although, thousands of crores of rupees have been spent on rural development during the last 65 years, the problem is still severe. Yet, the importance of this subject has enhanced. Present study reveals that the status of agro entrepreneurship for sustainable agriculture. 72% students from rural background are interested in job while only 28% students prefer business profession and 62% students think finance as a major problem to start any new business. Therefore, it is necessary to develop an entrepreneurial culture among rural areas. Without entrepreneurship, rural areas will remain cut off from the benefits of the structural adjustment, which has been already going on for sometime in urban and industrially developed regions. The agriculture becomes sustainable if application of bio-input increases. Our findings reveal that more than 60% of entrepreneurs do not sale bio- inoculate as demand is less and 18% do not sale due to any assurance of guaranty.

Key Word: Entrepreneurship/Sustainable agriculture/ Rural development)

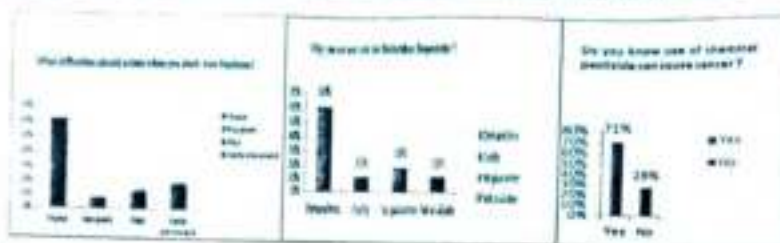
Introduction

Agriculture in India, the prominent sector of the economy, is the source of livelihood of almost two thirds of the workforce in the country. The contribution of agriculture and allied activities to India's economic growth in recent years has been no less significant than that of industry and services (Economic Survey 2009-10). Therefore it is necessary to develop rural entrepreneurship effectively, so that an environment conducive to sustainable development of rural areas, based on rural entrepreneurship, will be achieved. The modern agriculture cannot be sustainable in long run because of the adverse changes being caused to the environment and the ecosystem due to extensive use of chemical fertilizers and pesticides (Kaiser, 2004; Ghosh, 2004; Worthington M K., 1980). Due to the rising input costs involved in modern farming and its

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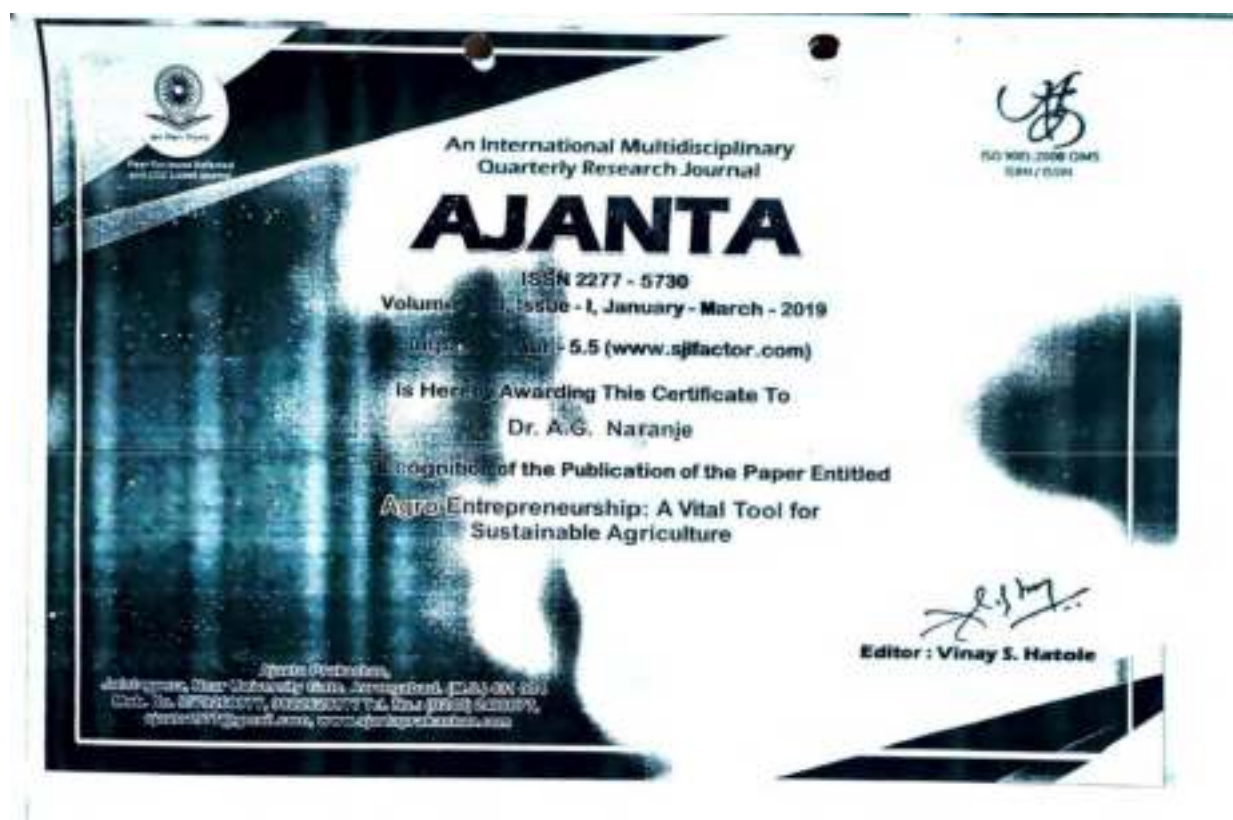
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SYNTHESIS OF SCHIFF BASES BY CONDENSATION OF AROMATIC AMINES WITH 3-(5-BROMOTHIOPHEN-2-YL)-1-PHENYL-1-H-PYRAZOLE-4-CARBALDEHYDE. : Sachin V. Manohare

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SYNTHESIS OF SCHIFF BASES BY CONDENSATION OF AROMATIC AMINES WITH 3-(5-BROMOTHIOPHEN-2-YL)-1-PHENYL-1-H-PYRAZOLE-4-CARBALDEHYDE.

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ABSTRACT

In the present study, novel Schiff bases were synthesized by condensation of 3-(5-bromothiophen-2-yl)-1-phenyl-1-H-pyrazole-4-carbaldehyde with different aromatic amines. 3-(5-bromothiophen-2-yl)-1-phenyl-1-H-pyrazole-4-carbaldehyde was synthesized from 2-acetyl-5-bromothiophene via 1-(3-(5-bromothiophen-2-yl)ethynyl)-2-phenylhydrazine. The progress of the reaction and purity of the synthesized compounds was monitored by thin layer chromatography with F252 silica gel precoated aluminium sheets using petroleum ether – ethyl acetate (8:1) as a developing solvent and spots were visualized with near UV light and iodine vapour. The structures of newly synthesized compounds were confirmed on the basis of IR, ¹H-NMR spectroscopy and Mass spectrometry.

Keywords: Hydrazine, pyrazole, Schiff bases and spectral characterization.

INTRODUCTION

Heterocyclic systems containing mainly nitrogen, sulfur and oxygen atom constitute a large class of compounds of biological and medicinal interest. A huge number of heterocyclic systems which include mainly five and six membered compounds represent a diverse group of molecular scaffolds. Several such heterocyclic scaffolds have been successfully incorporated into novel drug leads and therapeutic agents¹. In recent years Schiff bases are frequently used and studied for the betterment of human welfare, attracting much attention in organic and inorganic synthesis. Schiff bases are the important compounds owing to their wide range of biological activities and industrial

applications, they have been found to possess the different pharmacological activities such as antibacterial^{2,3}, antifungal^{4,5}, anticancer^{6,7}, antiviral^{8,9}, anti-inflammatory¹⁰, anti-HIV¹¹ and antitubercular¹². Further more from the literature survey it is observed that Schiff bases with sulfur atom are a class of important compounds in medicinal and pharmaceutical field. Therefore, it seemed interesting to consider the synthesis of new Schiff bases by condensing different aromatic amines with 3-(5-bromothiophen-2-yl)-1-phenyl-1-H-pyrazole-4-carbaldehyde. In this paper we report the synthesis and characterization of Schiff bases derived from 3-(5-bromothiophen-2-yl)-1-phenyl-1-H-pyrazole-4-carbaldehyde.



2. Experimental

MATERIALS AND METHOD

All the chemicals were purchased from Sigma Aldrich and SDFINE chemicals and used further without purification. Melting points were determined in open capillary tube and are uncorrected. The progress of the reactions as well as purity of the compounds was monitored by thin layer chromatography with F252 silica gel precoated aluminium sheets using petroleum ether – ethyl acetate (9:1) as a developing solvent and spots were visualized with near UV light and iodine vapors. The IR spectra were recorded on FTIR SHIMADZU SPECTROPHOTOMETER using KBr pellets and expressed in cm^{-1} . The NMR spectra were recorded on BRUKER ADVANCE (400 FT-NMR) spectrophotometer in DMSO using tetramethyl silane as an internal standard and chemical shift values are expressed in δ ppm. The MASS spectra were recorded on Waters UPLC-TQC MASS Spectrometer.

2.2. Synthesis of 1-(1-(5-bromothiophen-2-yl)ethylidene)-2-phenylhydrazine 3.

0.01 mol of 2-acetyl-5-bromothiophene (1), 0.01 mol phenyl hydrazine (2) and 2ml glacial acetic acid in 20 ml of ethanol. The reaction mixture was stirred on magnetic stirrer at room temperature for two hours. The yellow precipitate thus obtained was separated by filtration, washed with ethanol, dried and purified by recrystallization from ethanol. Yellow solid. Yield – 76.20%. M.p. 122°C . IR (KBr cm^{-1}): 3347 cm^{-1} (N—H), 3022 cm^{-1} , 3046 cm^{-1} (C—H), 1595 cm^{-1} (C=N), 1452 cm^{-1} (C=C). $^1\text{H-NMR}$ (DMSO, δ , ppm): 9.1 (s, 1H, NH), 7.1 (s, 3H, CH₂), 6.7 (m, 1H, Ar—H), 7.1 (m, 4H, Ar—H), 6.8 (d, 1H, Ar—H), 6.9 (d, 1H, Ar—H). Mass: 293 (M^+), 204, 133, 109. Anal. (%) for $\text{C}_{12}\text{H}_{11}\text{BrN}_2\text{S}$, calcd., C, 48.83; H, 3.76; Br, 27.07; N, 9.49; S, 10.86.

2.3. Synthesis of 3-(5-bromothiophen-2-yl)-1-phenyl-1H-pyrazole-4-carbaldehyde 4.

0.03 mol of POCl_3 was added drop wise to an ice-cold stirred solution of 1-(1-(5-bromothiophen-2-yl)ethylidene)-2-phenylhydrazine (3) (0.01 mol) in 5ml DMF. The reaction mixture was stirred for half hour at room temperature first and then at 70°C for about five hours on magnetic stirrer. The

resulting mixture was poured onto crushed ice and neutralized with saturated sodium bicarbonate solution. The pale green precipitate obtained was purified by recrystallization with ethanol to yield the product. Pale green solid. Yield- 89.27 %. M. p. 132°C . IR (KBr, cm^{-1}) 3102 cm^{-1} (Ar—H), 2740 cm^{-1} (CO—H), 1680 cm^{-1} (C=O), 1598 cm^{-1} (C=N), 1467 cm^{-1} (C=C). $^1\text{H-NMR}$ (DMSO, δ , ppm): 10 (s, 1H, CHO), 9.2 (s, 1H, C—H, Pyrazole), 8.0 (d, 1H, J=3.92 Hz, Ar—H) 7.1 (d, 1H, J=3.92 Hz, Ar—H), 7.3-7.9 (m, 5H, Ar—H). Mass: 333 (M^+). Anal. (%) for $\text{C}_{14}\text{H}_8\text{BrN}_2\text{OS}$, calcd., C, 50.47; H, 2.72; Br, 23.98; N, 8.41; O, 4.80; S, 9.62.

2.4. Synthesis of Sebüt bases 5a-5b.

0.01 mol of 3-(5-bromothiophen-2-yl)-1-phenyl-1H-pyrazole-4-carbaldehyde (4), 0.01 mol of aromatic aldehydes were dissolved in 20 ml of ethanol followed by addition of 2-3 drops of glacial acetic acid. The reaction mixture was refluxed for about 1-4 hours and then cooled at room temperature. The solid thus obtained separated by filtration, washed with ethanol, dried and purified by recrystallization from ethanol.

2.4.1. 1-(3-(5-bromothiophen-2-yl)-1-phenyl-1H-pyrazol-4-yl)-N-phenylmethanimine 5a.

Pale yellow solid. M. p. 134°C . Yield-69.48%. IR (KBr, cm^{-1}): 3138 cm^{-1} (Ar—H), 1616 cm^{-1} (C=N), 1484 cm^{-1} (C=C), 758 cm^{-1} (C—Br). $^1\text{H-NMR}$ (DMSO, δ , ppm): 9.0 (s, 1H, C—H, Pyrazole), 8.6 (s, 1H, C=N), 7.2-8.0 (m, 12 H, Ar—H). Mass: 408 (M^+). Anal. (%) for $\text{C}_{18}\text{H}_{14}\text{BrN}_3\text{S}$, calcd., C, 58.83; H, 3.46; Br, 19.57; N, 10.29; S, 7.85.

2.4.2. 1-(3-(5-bromothiophen-2-yl)-1-phenyl-1H-pyrazol-4-yl)-N-(4-chlorophenyl) methanimine 5b.

Pale yellow solid. M. p. 114°C . Yield-78.38%. IR (KBr, cm^{-1}): 3136 cm^{-1} (Ar—H), 1608 cm^{-1} (C=N), 1477 cm^{-1} (C=C), 758 cm^{-1} (C—Br). $^1\text{H-NMR}$ (DMSO, δ , ppm): 9.3 (s, 1H, C—H, Pyrazole), 8.5 (s, 1H, C=N), 6.5-7.9 (m, 11 H, Ar—H). Mass: 442 (M^+). Anal. (%) for $\text{C}_{20}\text{H}_{13}\text{BrClN}_3\text{S}$, calcd., C, 54.26; H, 2.96; Br, 18.05; Cl, 8.01; N, 9.49; S, 7.24.

Compound	Mol. Formula	Ar	Colour	Yield (%)	M.P. (°C)
3	C ₁₇ H ₁₁ BrN ₂ S	—	Yellow	76.20	122
4	C ₁₇ H ₁₁ BrN ₂ OS	—	Pale green	89.27	132
5a	C ₁₈ H ₁₃ BrN ₂ S	C ₆ H ₅	Pale yellow	69.48	124
5b	C ₁₈ H ₁₃ BrClN ₂ S	C ₆ H ₄ -Cl-p	Pale yellow	78.38	114
5c	C ₁₈ H ₁₃ BrClN ₂ S	C ₆ H ₄ -Cl-o	Pale yellow	84.19	102
5d	C ₁₈ H ₁₃ BrN ₂ S	C ₆ H ₄ -Br-p	Yellow	67.40	118
5e	C ₁₈ H ₁₃ BrN ₂ OS	C ₆ H ₄ -OH-p	Yellow	68.22	128
5f	C ₁₈ H ₁₃ BrN ₂ O ₂ S	C ₆ H ₄ -NO ₂ -p	Yellow	73.76	94
5g	C ₁₈ H ₁₃ BrN ₂ O ₂ S	C ₆ H ₄ -NO ₂ -m	Yellow	76.18	124
5h	C ₁₇ H ₁₁ BrN ₂ S	C ₆ H ₅ CH ₂ -p	Yellow	83.42	122

Table – Physical constant data of synthesized compounds.

RESULTS AND DISCUSSION

The synthetic route to synthesize title compounds is outline in scheme. 1-(1-(5-bromothiophen-2-yl)ethylidene)-2-phenylhydrazine (3) was prepared according to the method reported in literature, using 2-acetyl-5-bromothiophene as a starting material. 3-(5-bromothiophen-2-yl)-1-phenyl-1-H-pyrazole-4-carbaldehyde (4) was prepared by Vilsmeier Haack Reaction. The reaction of 3-(5-bromothiophen-2-yl)-1-phenyl-1-H-pyrazole-4-carbaldehyde (4) with different aromatic amines in ethanol using glacial acetic acid as a catalyst, afforded Schiff bases in good yield.

The structure of prepared compounds was confirmed by Infra Red spectroscopy, ¹H-NMR spectroscopy and mass spectrometry. The FTIR spectra of compound (3) showed the band at 3347 cm⁻¹ for NH group of hydrazone and then vanished. The bands at 1680 cm⁻¹ and 2740 cm⁻¹ corresponds to C=O and CO—H groups of pyrazole (4). The band of C=N imine stretching vibration was found in prepared Schiff bases at 1616 cm⁻¹ and 1591 cm⁻¹. The ¹H-NMR spectrum of compound (3) showed the following characteristic chemical shifts: the singlet signal at δ=9.1ppm suggested the attribution of NH proton, the singlet signal at δ=2.1 ppm suggested the attribution of the protons of CH₃ group. The ¹H-NMR spectrum of compound (4) showed the following characteristic chemical shifts: the singlet signal at δ= 10 ppm suggested the attribution of

proton of CHO group, the singlet signal at δ= 9.2 ppm suggested the attribution of CH proton of pyrazole ring. Also the ¹H-NMR spectrum of Schiff bases showed the following characteristic chemical shifts: the singlet at δ= 9.3 ppm corresponds to CH proton of pyrazole ring and the singlet signal at δ= 8.5 ppm suggested the attribution of H—C=N proton. In the mass spectra of prepared compounds, molecular ion peaks were observed at 295 (M⁺), for compound (3), 333 (M⁺) for compound (4), 408 (M⁺) for compound (5a) and 442 (M⁺) for compound (5b), which confirms the proposed structure of prepared compounds.

CONCLUSION

A new series of Schiff bases was prepared and the structure of newly synthesized Schiff bases was confirmed on the basis of IR spectroscopy, ¹H-NMR spectroscopy and Mass spectrometry. These newly prepared Schiff bases constitute a family of compounds, of great interest with regards to biological activity.

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Investigation of BMI-PF₆ ionic liquid/graphite interface using frequency modulation atomic force spectroscopy: Harshal P. Mungase



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Investigation of BMI-PF₆ Ionic Liquid/Graphite Interface Using Frequency Modulation Atomic Force Microscopy

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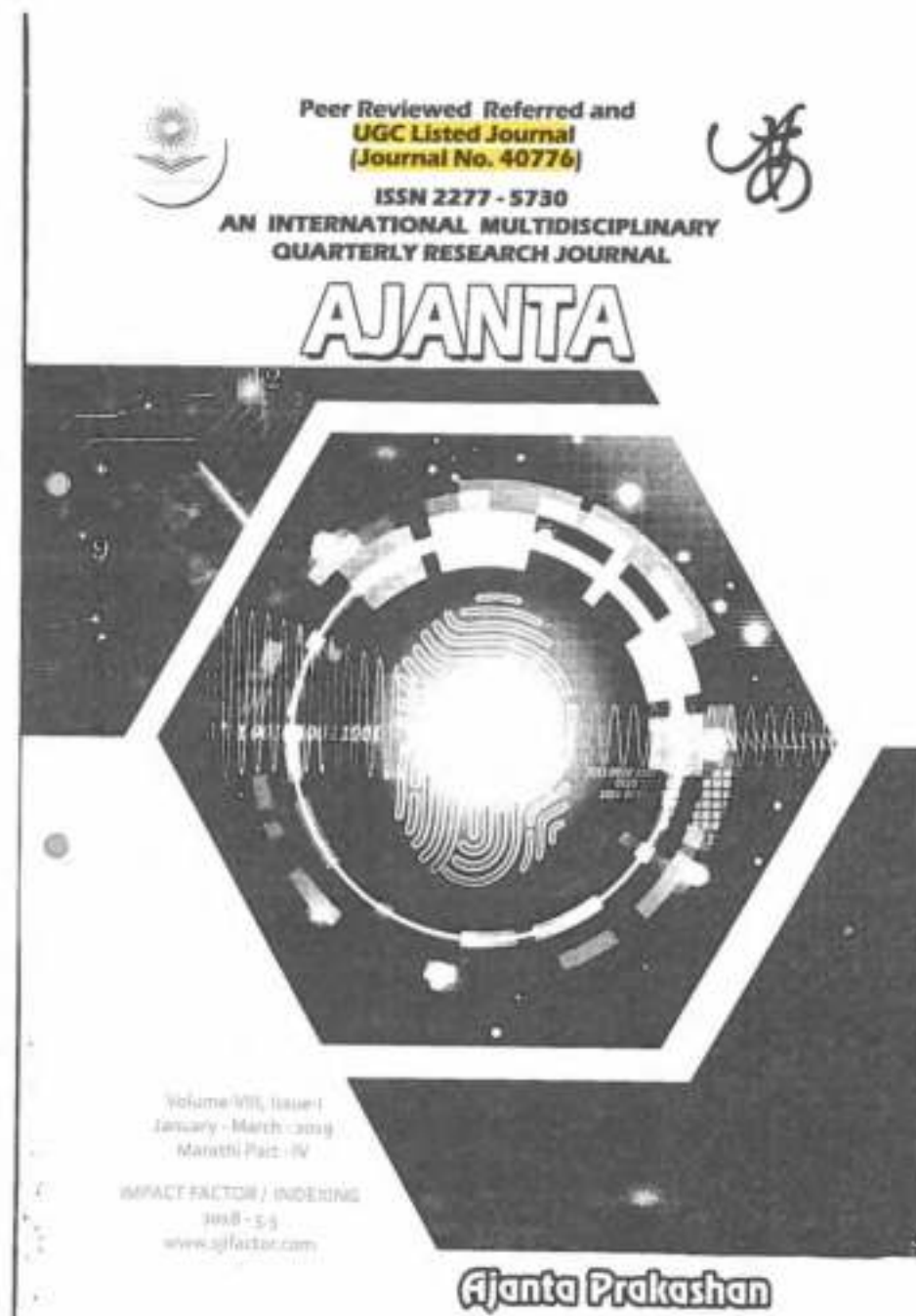
ABSTRACT

Structural analysis on interfaces between ionic liquids (ILs) and solid substrates is an important study for not only the basic fundamental aspects but also many technological processes. In the present work, we utilized frequency modulation atomic force microscopy (FM-AFM) based on a quartz tuning fork sensor to elucidate the structure of interface between 1-butyl-3-methylimidazolium hexafluorophosphate (BMI-PF₆) IL and highly ordered pyrolytic graphite (HOPG) surface. It was observed that this IL form solvation layers at their interface, with ~0.5–0.57 nm thickness of each layer. We have compared our experimental results with previously reported results from molecular dynamics simulation study, and combination of classical molecular dynamics and density functional theory calculations in order to understand the IL/HOPG interface.

INTRODUCTION

Room temperature ILs are new class of ionic salts which contains organic cations and organic/inorganic anions. In recent years, they attracted great attention of the various research communities due to their remarkable properties such as high ion conductivity, low vapor pressure, high thermal stability, and high electrochemical stability [1,2]. Due to these properties their demand increased in many technological applications such as catalysis, coating, electrolyte for batteries and solar cells, crystal growth processes, as a solvent in synthetic processes, in the field of water splitting to produce hydrogen, and neat or base lubricants in tribology [3–14]. However, for these applications, a detailed understanding of molecular-scale properties of ILs at the IL/solid interfaces is essential because important processes occur at their interface. The IL exhibit different behavior at the interface as compare to bulk. An organization of IL molecules depends not only on the structure of cations/anions but also on nature of substrate materials, on which they organized [15,16]. Overall the interfacial properties depend on various factors such as charge on the substrate surface, chemical nature, geometry of surface, and chemical

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CONTENTS OF MARATHI PART - IV

अ.क्र.	लेख आणि लेखकाचे नाव	पृष्ठ क्र.
१६	महात्मा जोतीराव फुले यांच्या शिक्षणविषयक विचारांची प्रामाणिकता आकाश शंभराव बांगर	६४-६६
१७	डॉ. बाबासाहेब आंबेडकरांचे राजकीय विचार अमोल भाऊराव बंड	६७-७०
१८	सामाजिक चळवळीत विचारवंतांचे योगदान प्रा. दिगंबरकुमार ब्रह्मादराव लांडगे	७१-७५
१९	पंडित जगज्जलाल नेहरू यांचे आदर्शवादी विचार कु. अलफाया ई. खाजावाले प्रा. डॉ. जीवन पवार	७६-८०
२०	समाजसुधारणा चळवळीत राष्ट्रसंत तुकडोजी महाराज ऐतिहासिक योगदान प्रा. डॉ. किशोर एच. बाराबाबे	८१-८३
२१	मध्ययुगातील प्रमुख महिला संतांचे स्त्री कल्याणासंबंधी सामाजिक प्रबोधन प्रा. डॉ. एम. पी. खेडकर	८४-८६
२२	महात्मा फुलेंच्या विचारांतील सामाजिक व आर्थिक प्रगती प्रा. डॉ. यशुकर श्रीराम ताकतोडे	८७-९०
२३	डॉ. बाबासाहेब आंबेडकर कृषी अर्थव्यवस्थेसंबंधी विचार डॉ. उत्पल ना. येडशीकर	९१-९२
२४	सामाजिक वर्गांमध्ये गो. म. आगरकरांचे योगदान डॉ. नाना वानखेडे	९३-९६
२५	पं. नेहरूंचे लोकशाही विषयक विचार प्रा. नीता पांडे	९७-१००
२६	दलित व समाज परिवर्तन प्रा. विनय पैकिने	१०१-१०२
२७	बंगाल महिलांची आर्थिक व सामाजिक स्थिती प्रेमसिंग ब्रह्माद जाधव	१०३-१०५
२८	सामाजिक चळवळीत विचारवंतांचे ऐतिहासिक योगदान राजेंद्र बापूराव काकडे	१०६-११०
२९	माहितीचा अधिकार आणि त्याचे महत्त्व प्रा. रमेश गुलाबराव सुरजकर	१११-११३
३०	महात्मा गांधी आणि शाश्वत विकास डॉ. सचिन ज. पैले	११८-१२१

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

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२०. समाजसुधारणा चळवळीत राष्ट्रसंत तुकडोजी ऐतिहासिक योगदान

प्र. डॉ. किशोर एच. बारापाडे

इतिहास विभाग प्रमुख, आदर्श महाविद्यालय, धामंगाव रेल्वे जि. अमरावती.

राष्ट्रसंत तुकडोजींचे समाजसुधारणा चळवळीत अमूल्य योगदान आहे. पारतंत्र्याच्या काळापासून ते स्वातंत्र्यानंतर सन्मत् २० वर्षे समाजोन्नतीच्या कार्यासहती त्यांनी संपूर्ण आयुष्य घालविले. या कार्यासहती विदर्भ, संपूर्ण महाराष्ट्र व महाराष्ट्राबाहेरही भटकंती केली त्यांना यावेळी समाजातील दोष जे दृष्टीपथात आले त्यावर त्यांनी सुधारणा करण्याचा ध्यास अंगी बानला. त्यांच्या कार्यकर्तृत्वाचा विचार केल्यास समाजमनावर संस्कार करण्याचा प्रयत्न करणा-या सामाजिक व राजकीय सुधारकात त्यांचे स्थान जागतिक महापुरुषात निश्चितच मोठे आहे त्यांच्या याणीतून निघणा-या एका एका शब्दात माणूस घडविण्याची महान क्षमता आहे.

राष्ट्रसंतांच्या विचारांची देवात आजच्या घडीला अत्यंत आवश्यकता आहे. तत्कालीन समाजात जातिभेद, उच्च-निम्नता जस्पृश्यता मोठ्या प्रमाणात प्रचलित होती. अंधश्रद्धाही शिगेला पोहोचली होती. भारतीय समाज यावेळी अज्ञान असून देव-धर्माच्या खोट्या कल्पनेत यावरून इच्छित फलप्राप्तीसाठी कनिष्ठ देवतेच्या पाठीमागे पळलेला आहे नवस व्रतदैकत्ये मोठ्या प्रमाणात होत असून जनतेचा कायदा पुजारी, बुवाबाजी लोकांनी घेतला. जनतेस नवसासाठी प्राण्यांचा वकी देण्याचीही प्रथा होती. तेव्हा जनतेस मार्गदर्शन करण्याचे काम राष्ट्रसंतांनी केले.

समाजात प्लेन, मलेरीया, देवी कौलरा इ. रोग मरिमाय व खंडोबाची अवकृपा समजत व औषधीपधाराशिवाय रोगी मृत्युमुखी पडत असे. तेव्हा महाराजांनी अंधश्रद्धेवर किरतून, मज्जन व खंजिरीच्या माध्यमाने प्रहार करून समाजात जनजागृती केली. आजही देशात यावेळा वेगळी स्थिती समाजात नाही. ग्रामीण भागात अशा पद्धतीचे आजार निर्माण झाले की, डॉक्टरचा सल्ला घेण्याऐवजी भोळी जनता मंत्रतंत्राचा व बुवाबाजीचा आश्रय घेते. अशावेळी राष्ट्रसंतांच्या वचनांची नक्कीच आठवण घेते. हे राष्ट्रसंतांचे विचार आजही समाजाला पुरक ठरतात. राष्ट्रसंताने खरा धर्म समाजाला सांगितला आहे. ते म्हणतात " धर्म म्हणजे चांगल्या विचारांचा समुदाय, जे विचार व्यक्ती, समाज व राष्ट्र उन्नीतीसाठी उपयुक्त ठरतात. त्या विचारांचा समुदाय म्हणजे धर्म. अशी धर्माकडल व्याख्या सांगून ते पुढे म्हणतात, " हिंदु, मुसलमान, ख्रिश्चन, सिख व जैन असे भेद नाही जेथे सर्व मानव सारखे आहेत आचार विचारांची शुध्दता ज्यात आहे, तोच खरा धर्म होय असा धर्म समाज व राष्ट्राला नेहमी पोषक असतो. पदधर्माकडल त्यांना प्रेम असून धर्मातराचा राष्ट्रसंतांनी विरोध केला धर्मातराऐवजी मानवी प्रकृती बदलविणे आवश्यक असल्याचे ते सांगतात. भक्तीतील अवडंबर राष्ट्रसंतांना आवडत नसे ते आचारात श्रेष्ठ स्थान देतात दीन-दुख्खांच्या सौधेत खरी परमेश्वर भक्ति आहे प्रत्येकाने सर्वांना समान लेखावे, येशमावना न ठेवता निस्वार्थपणे कार्य करावे हीच खरी परमेश्वर भक्ती आहे प्रत्येकाने सर्वांना समान लेखावे, येशमावना न ठेवता निस्वार्थपणे कार्य करावे हीच खरी परमेश्वर भक्ती आहे हे सर्व विचार तत्कालीन समाजात राष्ट्रसंत तुकडोजी महाराजांनी रुजविण्याचा प्रयत्न केला.

भारतात
संस्थां
लोक
जाला
प्रथा
संज्ञात
लोकतून
सौंदर्य
प्रथा
त्या
संदेश
गविले
त्यांनी

उच्च
संज्ञांनी
एका
जन्मून

प्राचा
एका
संज्ञा
प्राचा
जिन्हा
संज्ञा
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आहे. त्याची आज समाजात अत्यंत आवश्यकता आहे. जातीभेद, उच्चनिचता, स्त्री-पुरुष भेदभाव विचाराकडे पाहण्याचा दृष्टिकोन या सर्वांवर राष्ट्रसंतानी आवाज उठविला.

संदर्भ ग्रंथ सूची

१. बाबा मोहोड - राष्ट्रसंत तुकडोजी महाराज
२. प्रा. डॉ. बाळ पदमाड - राष्ट्रसंत वैचारिक क्रांती
३. बाबा आमारे - भगवान तुकडोजी
४. सुदाम सावरकर - राष्ट्रसंत तुकडोजी महाराज चरित्र
५. ए. भा. सरदार - संत यादवगयाची फलश्रुती
६. प्रा. सधुनाथ कडवे - मानवतेचे पुजारी राष्ट्रसंत तुकडोजी
७. बाबा मोहोड - राष्ट्रसंतांची भाषणे

Luminescence properties of near-uv excitable yellow-orange light emitting warm $\text{CaSrAl}_2\text{SiO}_7:\text{Sm}^{3+}$ phosphors: Dr. Shweta S Sharma



Scanned with CamScanner

10

B. Whorring et al. / *Journal of New Sports* 27 (2005) 209–219

excitation energy goes to the excited state and later returns to the bottom state, leading to emitting state within the regions, starting from ultraviolet light to infrared (IR). Lanthanum oxide (La_2O_3) is a crystal lanthanide chemical compound and has been studied wide because of its variable valency properties, thermal stability and chemical stability; it is appropriate for optics, glass, ceramic, catalytic applications, etc.

Trivalent samarium ion exhibits a $4f^5$ configuration and has complicated energy level. $5m^2$ ion shows distinct possible transitions between f -energy levels. The transitions are highly selective and give rise to sharp line spectra.

Thermoluminescence has been a recognized technique for radiation dosimetry for several decades primarily because of its convenient applications. TLDs have proven their applicability in varied fields like medical dosimetry, archaeological dating, personnel dosimetry, environmental monitoring and space dosimetry.¹ In the previous studies, many researchers have developed imaging radiation detectors using various kinds of material forms such as single crystals, ceramics, crystalline powder and glasses. Among the various material forms, glass materials have many advantages such as low cost, large productivities, easy handling and flexible chemical compositions. Glass is very suitable materials due to its considerable flexibility in size and shape, and can be doped at very high concentrations with active ions with excellent homogeneity. One of the important glass material belongs to meltable group having formula $M_2O \cdot M_2SiO_5$ with $M = Ca, Sr$. We selected $CaO \cdot Al_2O_3$ as a host from meltable group.

Inspired by the above knowledge, we have prepared $\text{CaAl}_2\text{Si}_2\text{O}_7$ phosphors doped with different concentrations of Sm^{3+} via solid state reaction route. Structural characterization was performed by powder X-ray diffraction (XRD) analysis. Detail investigations on photoluminescence (PL) properties of present phosphors was performed to find out potentiality of sample for use in LEDs, solid state lighting and display devices. Thermoluminescence (TL) properties of UV-irradiated $\text{CaAl}_2\text{Si}_2\text{O}_7:\text{Sm}^{3+}$ phosphors have been discussed in detail. To the best of our knowledge, PL and TL characterization of $\text{CaAl}_2\text{Si}_2\text{O}_7:\text{Sm}^{3+}$ phosphors have been rarely reported.

2. Experimental

Single phase $\text{In}_{1-x}\text{Ga}_x\text{-doped CaO}/\text{Al}_2\text{SiO}_5$ phosphors were successfully prepared through conventional high temperature solid-state reaction method. The employed raw materials were calcium carbonate (CaCO_3 , 98% purity), extra pure aluminum carbonate (Al_2CO_3 , 98% purity), silicon di-oxide (SiO_2 , 99.99% purity), aluminum oxide (Al_2O_3 , >98% purity) and samarium oxide (Sm_2O_3 , 99.99% purity). According to the nominal composition of $\text{Ca}_{1-x}\text{In}_x\text{Al}_2\text{SiO}_5$, Al_2SiO_5 with x ranging from 1 mol% to 0 mol%, these raw materials were precisely weighed without further purification. All the weighed powders were evenly mixed for 3 h by using mortar and pestle and then fired for 5 h in a programmable furnace maintained at 1300°C . On slowly cooling down to room temperature as prepared phosphors were recuperated from a crucible and then ground into powder for subsequent measurement.

For the confirmation of phosphorus on formed, X-ray diffraction analysis was done by using an Advance Bruker AXS-Diffractometer with Cu K α radiation ($\lambda = 0.154$ nm) and scanning rate 0.04°/s. The data were collected in the wide range of $2\theta = 2\theta \leq 30^\circ$. Surface morphology was characterized by field emission scanning electron microscopy (FESEM) S48000 (Hitachi). Photoluminescence (PL) properties of the phosphors were investigated by using an spectrofluorophotometer RF-S2012P (Shimadzu) equipped with 150 W xenon lamp as a light source. Powder samples were excited at single wavelength and emission spectra

were recorded over the range of visible wavelength. Excitation and emission slit widths were 1 mm for PL measurement. Thermoluminescence (TL) study was performed with the use of TLD reader, Nucletron TL1000 in the temperature range between 35 and 400 °C. Samples were placed on a small heating plate called as kanthal strip at the bottom of which a thermocouple is fixed to measure temperature and to sustain constant heating rate. A photomultiplier tube (PMT) was fixed above the kanthal strip to detect the TL signals. In this experiment, each time 7 mg of fine powder was exposed to 254 nm of UV light and then heated at a constant linear heating rate. Decay characteristic was performed with house-made indigenous setup and TL spectrum was recorded by using interference filters of various wavelengths.

3. Results and discussion

3.3. Strong and weak termination

Fig. 1 shows XRD patterns of $\text{CaFe}(\text{OH})_2\text{Ox}^{\text{H}}$ ($x = 1, 2, 3, 4, 5$ mol%, 2 mol%, 1 mol%, 4 mol%, 5 mol%, 5 mol%) phosphates prepared at 100°C together with Miller plane parameters (hkl). Sharp and narrow diffraction peaks show high crystalline quality and fine grain size of materials. All the diffraction patterns are found to synchronize well with standard pattern of pure $\text{CaFe}(\text{OH})_2\text{Ox}^{\text{H}}$ whose JCPDS card No. is 26-0327. It is suggested that doping of Sn^{4+} ions does not induce any significant influence on host crystal structure and no extra diffraction peaks were observed corresponding to any impurity. Therefore, no other impurity phase can be detected.

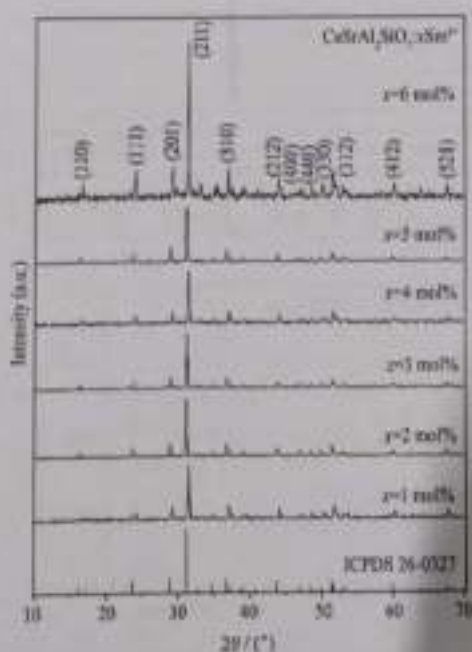


Fig. 1. NMR patterns of $\text{Ca}(\text{NO}_3)_2 \cdot x\text{H}_2\text{O}$ ($x = 1$ mol/L, 2 mol/L, 3 mol/L, 4 mol/L, 5 mol/L and 6 mol/L) at various temperatures.

which indicates that Sm^{3+} ions were completely dissolved in $\text{CaSrAl}_2\text{SiO}_7$ host. The XRD analysis indicated that powders crystallized in tetragonal phase and falls under the space group $P4_2/m$, as reported elsewhere.^{3,7} The observation results could confirm the formation of single phase $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ phosphor and substitution of Sm^{3+} ions for cation sites of Ca^{2+} and Sr^{2+} ions.

Average crystallite size of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor was evaluated by X-ray line broadening process using Scherrer equation stated as follows:

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

where D is crystallite size in nanometres, λ (≈ 0.154 nm for Cu K α radiation) wavelength of X-rays, β broadening of peak measured at half maximum intensity, θ diffraction peak position and K Scherrer constant generally equals to 0.94 for powder samples. According to Eq. (1), graph was plotted with $1/\beta$ along x-axis and $\cos \theta$ y-axis for $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor, each data point is attributed to different diffraction peaks. Here some intense planes such as (111), (201), (211), (100), (212) and (312) were taken into account. After a linear fitting to data, average crystallite size was estimated from the slope of the fitted line, see Fig. 2. The calculated crystallite size of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor from Scherrer method is 51 nm.

Crystal imperfections and distortion of strain-induced peak broadening are related by $\epsilon = \beta/\tan \theta$. There is an extraordinary property of Eq. (1) which has the dependency on the diffraction angle θ . Scherrer equation follows a $1/\cos \theta$ dependency but not $\tan \theta$ as W-H method. This basic difference was that both microstructural causes small crystallite size and microstrain occurs together from the reflection broadening. Depending on different θ positions the separation of size and strain broadening analysis is done using W-H method. The following results are the addition of the Scherrer equation and $\epsilon = \beta/\tan \theta$.

$$\beta = \frac{K\lambda}{D \cos \theta} + 4\epsilon \tan \theta \quad (2)$$

On rearranging Eq. (2):

$$\beta \cos \theta = \frac{K\lambda}{D} + 4\epsilon \sin \theta \quad (3)$$

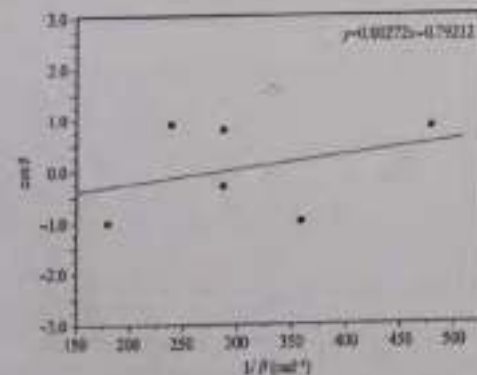


Fig. 2. Scherrer plot of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor. Crystallite size is extracted from the slope of linear fit line.

Eq. (3) is known as Williamson-Hall (W-H) equation. The W-H plot between $\sin \theta$ along x-axis and $\beta \cos \theta$ along y-axis was drawn as shown in Fig. 3. The crystallite size was determined from $K\lambda$ multiplied with reciprocal of intercept on $\sin \theta$ -axis and strain was found from slope of linear regression for the data in W-H plot. The obtained crystallite size and strain of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor from W-H method are 55 nm and -0.580×10^{-3} . The negative strain may be due to lattice shrinkage.²²

3.2. Morphological characterization

Morphology of the sample was investigated by FESEM tool. Fig. 4 represents different magnified FESEM micrographs of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor synthesized via high temperature solid state reaction method. $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor shows layered morphology with formation of small particles near layers.

3.3. Energy dispersive X-ray spectroscopy (EDS) characterization

Fig. 5 represents EDS spectrum of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor. It is a standard tool to definitive composition of material area as small as a few nanometres. EDS spectrum has emission peaks of calcium (Ca), strontium (Sr), aluminium (Al), silicon (Si) and samarium (Sm), no other extra emission peak was noticed that verified formation of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ phosphor.

3.4. Photoluminescence (PL) study

Concentration dependent photoluminescence excitation (PLE) spectra of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ ($x = 1$ mol%–6 mol%) phosphors were monitored at constant emission wavelength of 603 nm and are shown in Fig. 6. Intensity of all the excitation peaks increases as Sm^{3+} concentration increases and the highest intensity was found for 4 mol% of Sm^{3+} , afterwards excitation intensity decreases. Excitation spectra consist of several narrow bands between 300 and 500 nm which are ascribed due to well-known intra-configurational 4f–4f transitions of Sm^{3+} ions. Electronic transitions responsible for the occurrence of excitation bands are $^6\text{H}_{5/2} \rightarrow ^4\text{I}_{13/2}$ at 319 nm, $^6\text{H}_{5/2} \rightarrow ^6\text{H}_{9/2}$ at 345 nm, $^6\text{H}_{5/2} \rightarrow ^6\text{D}_{3/2}$ at 381 nm, $^6\text{H}_{5/2} \rightarrow ^6\text{D}_{5/2}$ at 376 nm, $^6\text{H}_{5/2} \rightarrow ^4\text{F}_{7/2}$ at 404 nm, $^6\text{H}_{5/2} \rightarrow ^6\text{F}_{5/2} + ^6\text{F}_7$ at 436 nm.

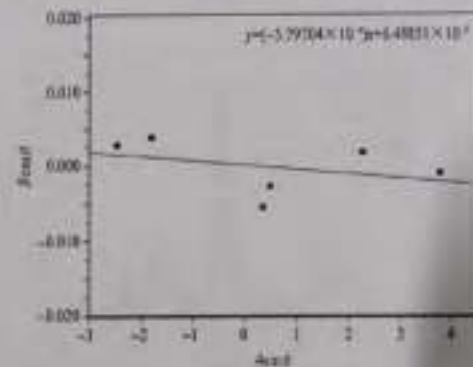


Fig. 3. Williamson-Hall (W-H) plot of $\text{CaSrAl}_2\text{SiO}_7\text{Sm}^{3+}$ (3 mol%) phosphor. Crystallite size D is estimated from the $\beta \cos \theta$ -intercept, and the strain ϵ extracted from the slope of the fit.

Fig. 4. FESEM micrograph of $\text{CaSrAl}_2\text{SiO}_6:\text{Sm}^{3+}$ (3 mol%) phosphor.

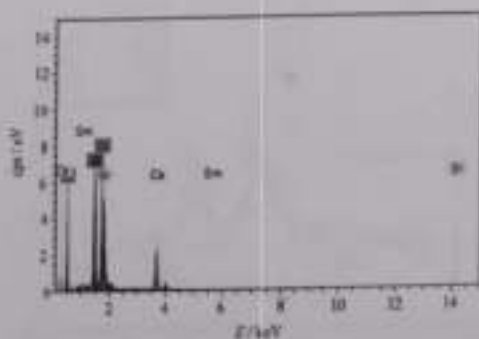
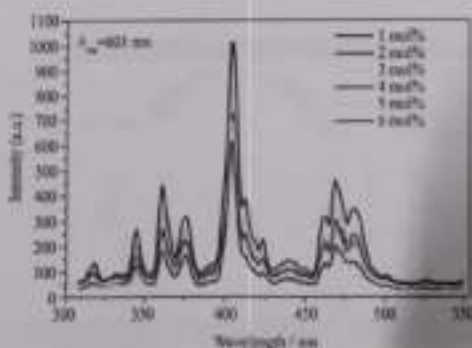
412 nm, $^4\text{H}_{11/2} \rightarrow ^4\text{F}_{7/2}$ at 424 nm, $^4\text{H}_{11/2} \rightarrow ^4\text{G}_{5/2}$ at 438 nm, $^4\text{H}_{11/2} \rightarrow ^4\text{F}_{9/2}$ at 463 and 469 nm, $^4\text{H}_{11/2} \rightarrow ^4\text{F}_{5/2} + ^4\text{M}_2$ at 481 nm.²²

In order to recognize optimum excitation wavelength, various excitation wavelengths (345, 363, 376, 404, 413, 463, 469 and 480 nm) have been excited to record PL emission spectra. Fig. 7 depicts comparison of emission spectra of $\text{CaSrAl}_2\text{SiO}_6:\text{Sm}^{3+}$ (4 mol%) phosphor measured at different excitation wavelengths. Under all excitation wavelengths, emission spectra have similar trend and containing four emission peaks in orange-red spectral region (550–700 nm). It is observed that the strongest emission intensity was obtained when phosphor is excited under 404 nm as compared to other excitation wavelengths. This implied that the present phosphors can be efficiently excited by UV (~400 nm) LED chip. Now the sample is excited with 404 nm to acquire concentration dependent PL emission spectra, this result is shown in Fig. 8.

Sharp emission features are typical for forbidden transitions of rare earth (Sm^{3+} and Sm^{2+}) ions also show such emissions in many optical materials. Moreover, these electronic transitions are shielded by the 5d outer orbitals, hence the electronic structures are more or less consistent regardless of the host matrix but the positions can be affected by the surrounding crystal fields to some extent.²³ In the PL spectra, the main emission peaks were located at 564, 570, 601 and 650 nm. The emission bands located at 564 and 570 nm are associated with transition $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$ while emission bands at 601 and 650 nm are due to $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$ and $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$, respectively. The general selection rules for magnetic-dipole (MD) transition and electric-dipole (ED) transition are $\Delta J = 0, \pm 1$ and $\Delta J \leq 6$ when $\Delta L = 2, 4, 6$, respectively. According to these selection rules the $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$ transition is purely a MD transition, the $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$ transition is a purely MD and partly ED transition and

the $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$ transition is purely an ED transition.²² Generally, the intensity ratio of electric dipole to magnetic dipole transitions is useful to understand the symmetry of local environment around the trivalent Sm^{3+} ions in the host matrix. The asymmetric nature is more prominent when the intensity of ED transition is stronger. In the present series of $\text{CaSrAl}_2\text{SiO}_6:\text{Sm}^{3+}$, the magnetic dipole transition $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$ is more intense than electric dipole transition $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$ for all concentrations of Sm^{3+} ions, it causes the result that there is no deviation from inversion center and more symmetric in nature.^{23–25}

Doping concentration significantly affects luminescence properties of the sample. Hence to get efficient luminescence output doping concentration should be optimized for which strongest emission intensity is obtained. Consequently we have used solid state reaction method to synthesize $\text{CaSrAl}_2\text{SiO}_6:\text{Sm}^{3+}$ phosphors with varying concentrations of Sm^{3+} keeping all other synthesis parameters constant. PL emission spectra (Fig. 8) for all the samples were recorded. Notably, there was no change in peak positions with variation in doping (Sm^{3+}) concentration while only emission intensity gets modified. From Fig. 8 it is seen that emission intensity of transition $^4\text{G}_{5/2} \rightarrow ^4\text{H}_{11/2}$ (601 nm) initially increases up to 4 mol% Sm^{3+} concentration (x) and then decreases. Hence 4 mol% of Sm^{3+} is the optimum doping concentration in the host matrix $\text{CaSrAl}_2\text{SiO}_6$. The decay in emission intensity over optimum doping concentration (4 mol%) is due to concentration quenching mechanism. As the Sm^{3+} concentration increases the probability of energy transfer between Sm^{3+} ions via non-radiative process becomes stronger. The non-radiative energy transfer generally originated from either exchange interaction or multipolar interaction within

Fig. 5. EDI spectrum of $\text{CaSrAl}_2\text{SiO}_6:\text{Sm}^{3+}$ (3 mol%) phosphor.Fig. 6. PL excitation spectra of $\text{CaSrAl}_2\text{SiO}_6:\text{Sm}^{3+}$ ($x = 1$ mol%, 2 mol%, 3 mol%, 4 mol%, 5 mol% and 6 mol%) phosphors.

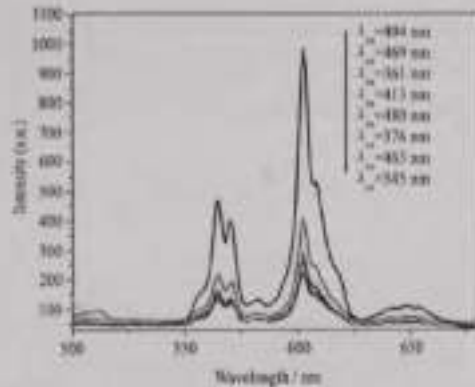


Fig. 3. PL emission spectra of $\text{Ca}_{0.95}\text{Al}_{0.05}\text{SiO}_3:\text{Sm}^{3+}$ (4 mol%) phosphor with different excitation wavelengths.

same sixth ions. The type of interaction can be determined according to the Dexter's theory. The type of interaction can be outcome from the following equation that gives emission intensity (I) per doping concentration (x).

$$I/x = K/[1 + \beta(x)^{\theta+1}]^{-1} \quad (4)$$

Where I is the emission intensity, x is dopant concentration over the quenching point, K and β are constants for given excitation wavelength and crystal structure, θ can be 3, 6, 8 and 10 for exchange interaction, dipole-dipole (d-d) interaction, dipole-quadrupole (d-q) interaction and quadrupole-quadrupole (q-q) interaction, respectively.

If $\beta(x)^{\theta+1} \gg 1$, Eq. (4) can be simplified as:

$$\lg(I/x) = K' - \theta/3 \lg x \quad (\text{with } K' = \lg K - \lg \beta) \quad (5)$$

Eq. (5) is a straight line equation, therefore θ can be obtained from the slope of $\lg(I/x)$ versus $\lg x$ curve, which is plotted as shown in Fig. 5, θ is found to be 7.344, which is nearly 8, hence

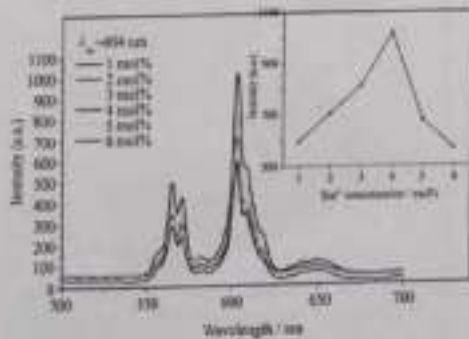


Fig. 4. PL emission spectra of $\text{Ca}_{0.95}\text{Al}_{0.05}\text{SiO}_3:\text{Sm}^{3+}$ ($x = 1$ mol%, 2 mol%, 3 mol%, 4 mol%, 5 mol% and 6 mol%) phosphor, inset shows emission intensity of ${}^6G_{7/2} \rightarrow {}^6H_{7/2}$ transition as a function of Sm^{3+} concentration.

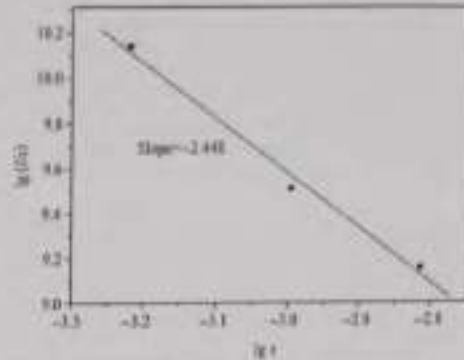


Fig. 5. Plot of $\lg(I/x)$ versus $\lg x$ for the ${}^6G_{7/2} \rightarrow {}^6H_{7/2}$ (603 nm) transition of Sm^{3+} ions in the host $\text{Ca}_{0.95}\text{Al}_{0.05}\text{SiO}_3$.

concentration quenching phenomenon of Sm^{3+} ions in $\text{Ca}_{0.95}\text{Al}_{0.05}\text{SiO}_3$ host lattice is due to the dipole-quadrupole (d-q) interaction.

The energy level scheme is shown in Fig. 10 to understand the mechanism involved in luminescence process in $\text{Ca}_{0.95}\text{Al}_{0.05}\text{SiO}_3:\text{Sm}^{3+}$ phosphor. When the sample is excited under 404 nm, Sm^{3+} ions are excited from ${}^6H_{5/2}$ ground state level to ${}^6P_{7/2}$ level. The population at ${}^6P_{7/2}$ level now relaxes to the level ${}^6G_{7/2}$ by non-radiative process. We can finally obtain five visible emission peaks located at 564, 578, 601 and 650 nm owing to the emission of Sm^{3+} ions from ${}^6G_{7/2}$ level to ${}^6H_1$ with $J = 5/2, 7/2, 9/2$. The emission peak at 564 nm seems to contain a shoulder at around 555 nm while peak at 601 nm may have two shoulders at around 606 and 616 nm, this may be due to Stark splitting of the ionic levels.

Commission International de l'Éclairage (CIE) is the most widely used method to describe the composition of any color in terms of three primaries (red–green–blue). Artificial "colors", denoted by X, Y, Z , also called tristimulus values, can be added to produce real spectral colors. The x, y, z , i.e., the ratio of X, Y, Z of the light to the sum of the three tristimulus values, are the so-called chromaticity coordinates. (x, y) is usually used to represent the color. In order to determine exact color of emission and color purity of phosphor the Commission International de l'Éclairage (CIE) diagram has been plotted as shown in Fig. 15. CIE chromaticity coordinate of

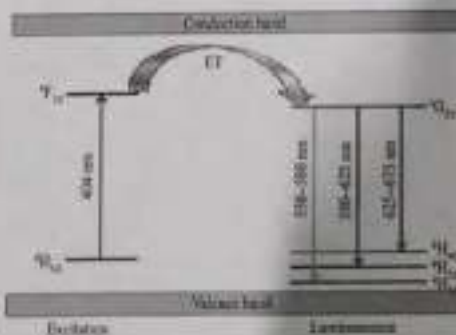


Fig. 10. Schematic energy level diagram of Sm^{3+} ions.

202

S. Sharma et al. / Journal of Alloys and Compounds 571 (2014) 191–202

$\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Sn}^{2+}$ (4 mol%) phosphor was investigated from the corresponding PL emission data when samples were excited under 404 nm wavelength. The chromaticity coordinates of the phosphor are (0.332, 0.431) which are located in yellow-orange region and marked with circle in CIE diagram.

Color purity of the sample was determined by using formula written below:

$$\text{Color purity} = \frac{\sqrt{(x - x_0)^2 + (y - y_0)^2}}{\sqrt{(x_0 - x_1)^2 + (y_0 - y_1)^2}} \times 100\% \quad (6)$$

where (x, y) are the chromaticity coordinates of the emitted light, for $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Sn}^{2+}$ (4 mol%) phosphor $(x, y) = (0.332, 0.431)$; $(x_0, y_0) = (0.333, 0.333)$ are the CIE coordinates of typical white light, and (x_1, y_1) are color coordinates of dominant wavelength point, for $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Sn}^{2+}$ (4 mol%) phosphor $(x_1, y_1) = (0.622, 0.377)$ corresponding to dominant wavelength at 601 nm. After substitution of these values in Eq. (6) color purity of $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Sn}^{2+}$ (4 mol%) phosphor resulted out as 70%.

Calculated color temperature (CCT) is the temperature of a black body whose chromaticity most nearly resembles that of a light. Low CCT implies warmer (more yellow-red) light, while high color temperature appears to be a colder (more blue) light.²² The correlated color temperature (CCT) was also estimated using the following equation:²³

$$\text{CCT} = -449n^2 + 3525n - 6823.3n + 5520.33 \quad (7)$$

where $n = \frac{y}{x+y}$ is the inverse slope line and (x_0, y_0) are (0.332, 0.330) chromaticity equities. Calculated value of CCT is 2231 K that can be considered as "warm" in appearance.

3.5. Thermoluminescence study

Imperfections exist in all the solids in the form of intrinsic defects and impurities. During excitation with appropriate radiation

these imperfections capture electrons/holes whose radiative recombination was measured by thermoluminescence analysis. The curve which indicates emission intensity as a function of temperature is called as TL glow curve. Each TL peak is associated with certain trap levels that lie within the forbidden gap. The traps are characterized by intrinsic kinetic parameters which may be retrieved from TL data.^{24–26} The dosimetric properties of TL materials are particularly based on the kinetic parameters which give the valuable information of defect centers that cause the TL emission.

3.5.1. Effect of variable heating rates

In the TL measurement thermal quenching is the serious issue that is relevant to the heating rates, hence one should optimize the heating rate. Consequently the influence of various heating rates (1, 3, 5, 7, 9 and 11 °C/s) on the TL glow curve of $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Sn}^{2+}$ (3 mol%) phosphor was investigated. Comparison of TL glow curves at different heating rates is shown in Fig. 11. For this experiment, each step is followed by 10 min irradiation of sample via 254 nm of UV-light. No change in the glow curve shape has been observed but peak position gradually shifts with heating rates. Inset in Fig. 11 indicates that the whole glow peak shifted towards the higher temperature side as the heating rate increases, shifting takes place from 185 to 226 °C for 1 to 11 °C/s heating rates. TL intensity increases as heating rate increases up to 9 °C/s thereafter TL intensity diminishes as the heating rate increases from 9 to 11 °C/s. This behaviour is consistent with the theoretical aspect that at lower heating rates retrapping of charge carriers (that produces desired TL emission) dominates and recombination with their counterparts suppressed. So at lower heating rates TL efficiency is low. Phenomenon of thermal quenching originates with rise in heating rate. During the thermal quenching process the charge carriers obtained from trap centers or luminescent centers cannot directly reach to the recombination-centred energy is transferred via non-radiative transition. So at higher heating rates TL intensity declines. Similar result was obtained in the work of Kaur et al.,²⁷ they have reported that reduction in TL intensity due to rise in heating rate can originate from thermal quenching of TL. In the present work 9 °C/s is the optimized heating rate at which maximum recombination occurs and hence maximum TL intensity is obtained with this heating rate. Hereafter further TL properties of the phosphors were studied at a constant heating rate of 9 °C/s.

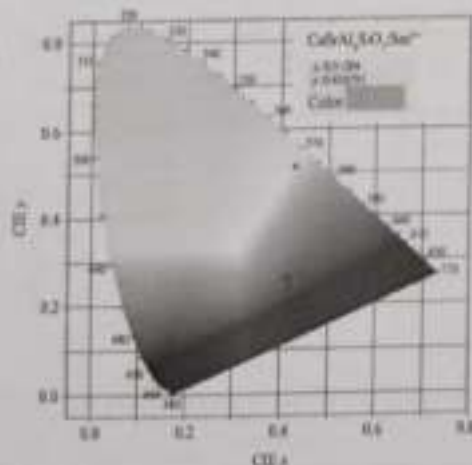


Fig. 11. CIE diagram of $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Sn}^{2+}$ (4 mol%) phosphor excited at 404 nm.

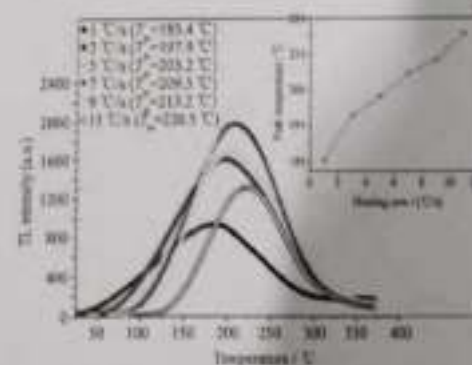


Fig. 12. Comparison of TL properties of $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Sn}^{2+}$ (3 mol%) phosphor at variable heating rates. Inset shows peak temperature versus heating rate.

3.5.2. TL glow curve

Fig. 13 illustrates the concentration dependence on the glow curve of UV-irradiated $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ phosphor, concentration of Sm^{3+} ranging from 1 mol% to 6 mol%. Shape of glow curve and peak position is independent of doping concentration. Doping concentration mainly affects luminescence of any phosphor material. If doping concentration increases up to a specific amount, they may be treated as self-quenchers due to non-radiative transitions and thus gives rise to fall in luminescence intensity. In the present work it was seen that TL intensity increases with increase in Sm^{3+} concentration till it gets equal to 3 mol% afterwards TL intensity seemed to be decreased with increase in Sm^{3+} concentration. This nature may arise due to concentration quenching mechanism. Hence the optimum doping concentration of Sm^{3+} in $\text{CaSrAl}_2\text{SiO}_7$ is found to be 3 mol%.

Fig. 14 shows TL glow curves of $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ (3 mol%) phosphor exposed at different UV-irradiation time ranging from 20 to 90 min. It is seen from Fig. 14 that glow curves recorded for all UV-irradiation time have single prominent peak at around 219 °C. Initially the increment in TL intensity is observed with increase in UV-irradiation time up to 60 min, and then TL intensity decreases for higher UV-irradiation time (80 and 90 min). This may be due to the fact that with increase in UV-irradiation time, more and more trapping centers of luminescent centers responsible for glow peak get filled. On thermal stimulation, the release of charge carriers from the traps or luminescent centers and the subsequent recombination with their counterparts at recombination centers increases the TL intensity of 219 °C glow peak.^{12–14} After a specific exposure time (60 min) traps begin to be destroyed and hence lowers TL intensity. It is also noticed that though the TL intensity of 219 °C glow peak of $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ (3 mol%) phosphor increases with increase in UV-irradiation time, there is no appreciable shift in peak temperature. The stability of peak temperature with increase in UV-irradiation time predicts the first-order kinetics.¹⁵ Since TL glow curve of $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ (3 mol%) phosphor is broad enough and contains single glow peak, that may be due to the superposition of more than one glow peaks. Hence glow curve was deconvoluted and kinetic parameters and activation energies were determined.

3.5.3. Gaussian fitting and kinetic parameters

TL glow curve of $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ (3 mol%) phosphor for 60 min of UV-irradiation was deconvoluted into four symmetrical traps, which are situated at 151.56, 201.02, 258.48 and 363.77 °C. Gaussian fitting of the glow curve is shown in Fig. 15. Peak shape

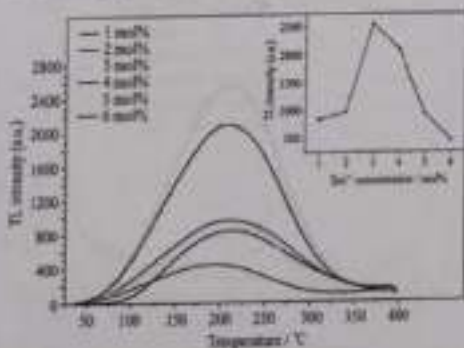


Fig. 13. Glow curves of 20 min UV-irradiated $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ phosphor, best shows TL intensity as a function of Sm^{3+} concentration.

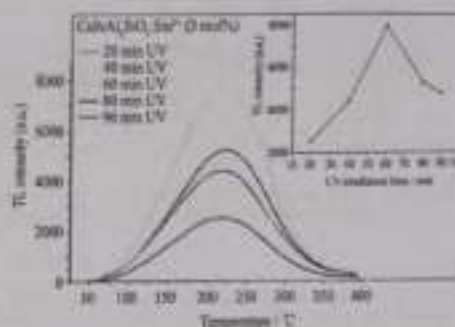


Fig. 14. Glow curves of $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ (3 mol%) phosphor for different UV-irradiation time, best exhibits plot of TL intensity with respect to UV-irradiation time.

method was used for all the traps. This method is based on three temperatures: temperature at maximum TL intensity i.e., T_m , temperatures at half intensity on ascending and descending parts of the glow curve are T_1 and T_2 , respectively. TL parameters for all glow peaks are tabulated in Table 1.

3.5.4. TL emission spectrum

TL spectrum was recorded with the use of interference band pass filter of different wavelengths having range from 500 to 700 nm. Observed TL emission spectrum is shown in Fig. 16. It is analogous to PL emission spectrum of $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ phosphor. Three emission bands were obtained which are positioned at 560, 600 and 650 nm; these are characteristic emission peaks of Sm^{3+} ions. The transitions from $^6G_{7/2}$ level to ^6F_J with $J = 5/2, 7/2, 9/2$ are responsible for originating emission peaks at 560, 600 and 650 nm, respectively. The detail description concerning nature of transitions corresponding to emission process in $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ phosphor has already been discussed in the present communication.

3.6. Decay characteristics

In order to study afterglow property of the sample phosphor, decay curve of $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ (3 mol%) phosphor was

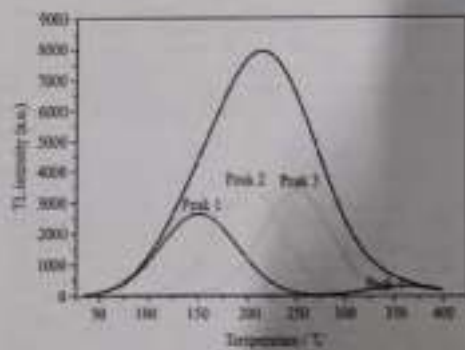


Fig. 15. Gaussian fitting curves of TL glow curve of 60 min UV-irradiated $\text{CaSrAl}_2\text{SiO}_7\text{:Sm}^{3+}$ (3 mol%) phosphor.

Table 1

TL parameters of UV-irradiated $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ (3 mol%) phosphor.

Peak	T_g (°C)	T_1 (°C)	T_2 (°C)	E (eV)	τ (s)	A_1 (%)	A_2	τ_1	τ_2	Activation energy (eV)	Frequency factor s^{-1}
1	191.57	182.13	194.84	43.27	48.46	82.73	0.47	0.67	1	0.44	1.71×10^5
2	201.02	187.73	244.98	43.28	43.27	86.55	0.56	1.09	2	0.46	2.94×10^5
3	234.86	202.24	295.76	43.24	43.17	86.54	0.59	1.09	2	0.82	2.28×10^5
4	302.75	278.49	390.86	37.09	43.18	83.57	0.46	0.46	3	1.15	8.18×10^5

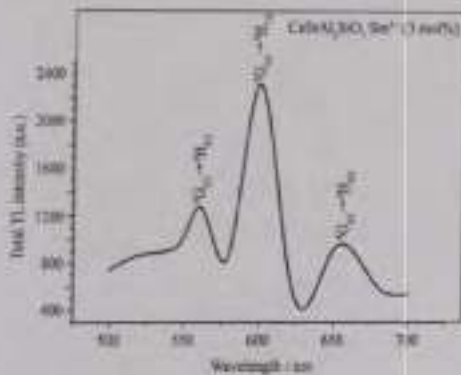


Fig. 16. TL emission spectrum of $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ (3 mol%) phosphor.

measured after removal of UV light (254 nm) source, initial intensity is much stronger, the decay of afterglow intensity is based on exponential function. At first it acts on the first exponential, then when the decay time is longer, the decay acts on the second exponential. The decay ratio slows down when the decay time increases.⁴⁷ Decay curve is plotted as shown in Fig. 17, which is fitted with the use of following double-exponential equation:

$$I(t) = I_0 - A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2) \quad (8)$$

$I(t)$ and I_0 are intensity corresponding to time t and $t = 0$, A_1 and A_2 are fitting constant, τ_1 and τ_2 are average lifetime with respect to

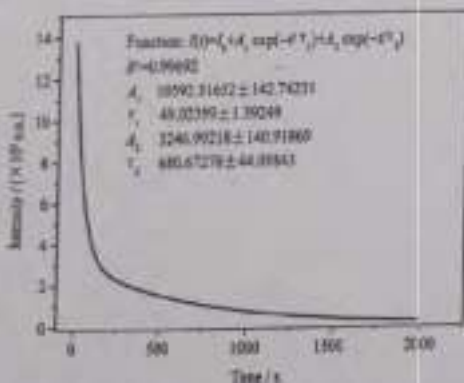


Fig. 17. Afterglow decay curve of $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ (3 mol%) phosphor after irradiation with 254 nm UV light for 10 min.

rapid and slow exponential components, respectively fitting parameters and fast and slow lifetimes are written in Table 1.

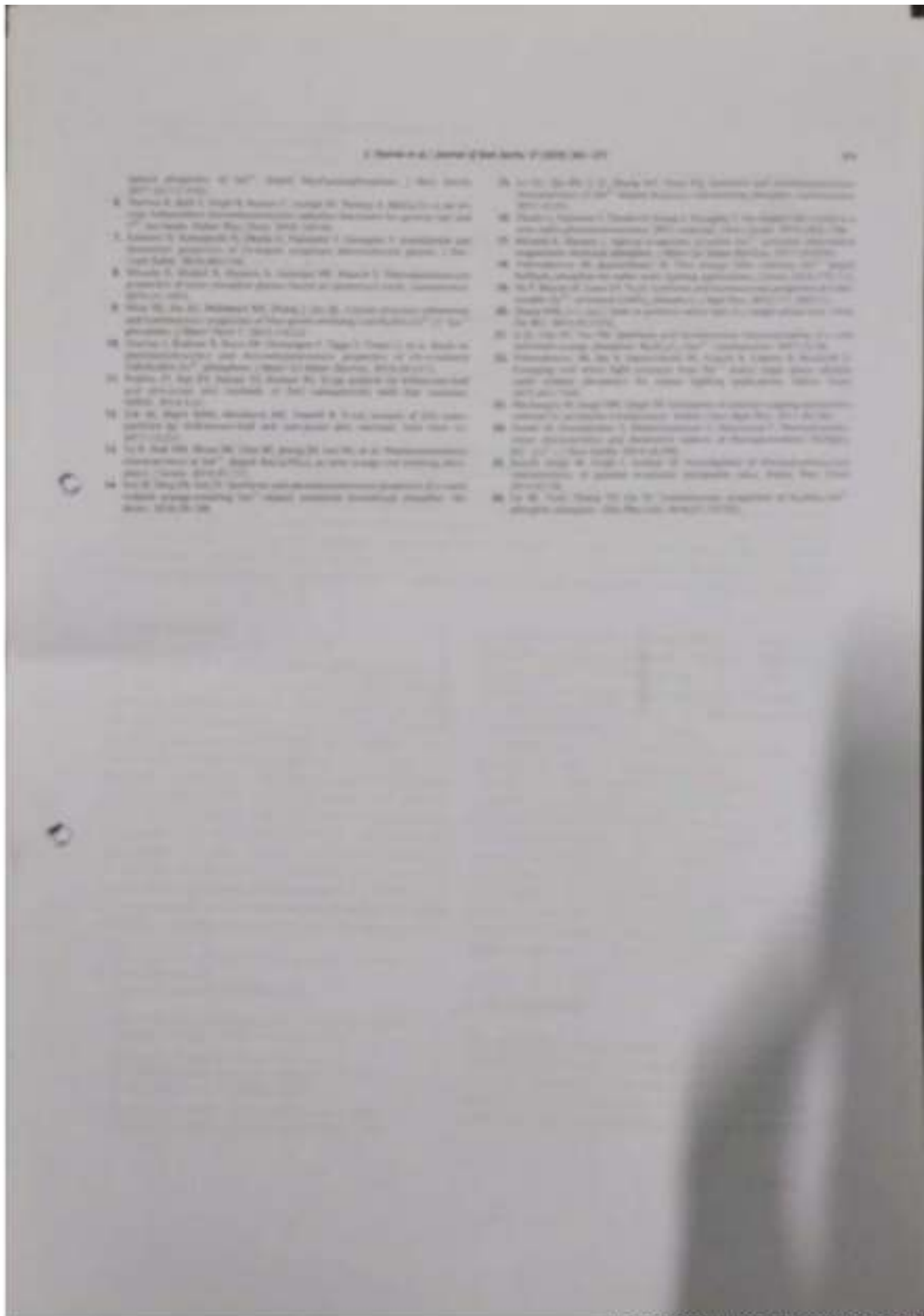
4. Conclusions

Well crystallized tetragonal phase Sm^{3+} activated $\text{CaSnAl}_2\text{SiO}_7$ phosphor were synthesized by solid state reaction route. Average crystalline size of synthesized sample was evaluated from powder XRD data by using Scherrer method and Williamson-Hall method, it is found to be 51 and 55 nm from Scherrer method and Williamson-Hall method, respectively. Concentration quenching in PL of present phosphor is due to dipole-quadrupole interaction. 4 mol% of Sm^{3+} is the optimized concentration in $\text{CaSnAl}_2\text{SiO}_7$. Present phosphor is efficiently excitable in near UV LED region (250–420 nm) and produces maximum emission centered at 601 nm which is due to $^4G_{5/2} \rightarrow ^6H_{5/2}$ transition of Sm^{3+} ions. CR coordinate for $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ (4 mol%) phosphor was calculated as (0.512, 0.431), which is in yellow-orange region. The photoluminescence decay curve was satisfactory fitted with double exponential approximation, fast and slow lifetimes were determined. Lower CCT of $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ phosphor indicates potentiality of the phosphor in warm light applications. The above mentioned results propose that $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ is a notable candidate for display and lighting applications. Although color purity of the sample is found to be 70%, it may be increased by charge compensation approaches. Enhancement of color purity and luminescence efficiency will be reported in our next communication.

Influence of various heating rates on TL glow curve of Sm^{3+} activated $\text{CaSnAl}_2\text{SiO}_7$ phosphor was investigated. The behaviour of TL intensity and glow peak with change in heating rate suggests existence of thermal quenching process. An up-gradation in TL intensity was found with rise in UV irradiation time up to 60 min and then lowers for further irradiation time. A linear TL response up to 60 min of UV exposure makes $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ a feasible thermoluminescent sample for low dose UV dosimetry. Deconvolution of glow curve of 60 min of UV-irradiated $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ phosphor was carried out. Single peak at 210 °C can be characterized by superposition of four glow peaks. Activation energies for all four peaks were estimated as 0.44, 0.56, 0.82 and 1.15 eV. Observed TL emission spectrum was similar to PL spectrum that gives emission in yellow-orange region; this result makes $\text{CaSnAl}_2\text{SiO}_7/\text{Sm}^{3+}$ phosphor a potential candidate for display applications.

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Thermoluminescence glow curve for UV induced $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor with its structural characterization

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Abstract

The present paper reports the synthesis and characterization of $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor. The phosphor was prepared by solid state reaction method. Average crystal size for the phosphor yielded was in nanometer range, which was calculated by using X-ray diffraction analysis and confirmed by field emission gun-scanning electron microscopy and transmission electron microscopy technique. Sample shows cubic structure and the particle size calculated by Scherrer's formula was around 48 nm. For recording TL glow curve every time 2 mg phosphor was irradiated by 254 nm UV source. Sample shows well resolved peak at 523 °C. Various parameters like heating rate and exposure time were optimized. The obtained glow curve for the optimized condition of heating rate and UV exposure time was analyzed by computerized glow curve deconvolution technique technique. Kinetic parameters were calculated by using Chen's peak shape method for all the deconvoluted peaks.

1 Introduction

Silicates with silicic acid have high stability and stable crystal structure. Because of these advantages their phosphors attracted much attention [1]. These materials also show the structural polymorphism. From this group of materials, $\text{Sr}_3\text{MgSi}_2\text{O}_8$ based materials show good thermally stimulated luminescence (TSL) behaviour. It has tendency to show luminescence after long time of removal of excitation source [2]. Though this material has excellent phosphorescence property only few works has been reported to explain the luminescence study of the activated strontium magnesium silicate phosphor. Pan and Hong reported synthesis of $\text{Sr}_3\text{MgSi}_2\text{O}_8$ activated with Eu^{2+} , Dy^{3+} by solid state reaction method and explained its optical property [3]. Talsou et al. has synthesized two different silicate phosphors activated with Eu^{2+} i.e. $\text{Sr}_3\text{MgSi}_2\text{O}_8/\text{Eu}^{2+}$ and $\text{Sr}_3\text{MgSi}_2\text{O}_8/\text{Eu}^{2+}$

phosphors by combustion synthesis method and studied their photoluminescence properties [4]. Chen et al. synthesized $\text{Sr}_3\text{MgSi}_2\text{O}_8/\text{Ce}^{3+}$, Th^{3+} for application in near ultraviolet excitable white light-emitting diodes [5]. Literature reveals no study was carried out to explain the luminescence behaviour of the pure $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor.

In present work we have synthesized the pure $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor by solid state synthesis method and studied the structural and thermoluminescence (TL) behaviour of the material. The $\text{Sr}_3\text{MgSi}_2\text{O}_8$ shows same spherical form and its TL behaviour have been studied for the application of radiation dosimetry. The main aim is to find out low cost material for radiation dosimetric applications. TL behaviour is studied under 254 nm UV excitation. The analysis of obtained TL glow curve is done by using computerized glow curve deconvolution (GCD) method. We compare the different TLD materials and $\text{Sr}_3\text{MgSi}_2\text{O}_8$ pure phosphor, which is novel for TL dosimetric application.

2 Experimental

The pure $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor was prepared by solid state method. The raw materials are: microsilica (carbonate) [SiO_2 (99.97%)], magnesium oxide [MgO (99.00%)], silica dioxide [SiO_2 (99.99%)]. Raw material samples were checked and sealed thoroughly in an airtight container for 2 h, then transferred

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in to a corundum crucible, and subsequently calcined at 1250 °C for 3 h [6].

The crystal structure of the prepared phosphor were characterized by powder X-ray diffraction (XRD). Powder XRD pattern has been obtained from Bruker D8 advanced X-ray powder diffractometer. The X-rays were produced using a sealed tube (Cu K α) radiation source and the wavelength of X-ray was 1.54060 Å. The X-rays were detected using a fast counting detector based on Silicon strip technology (Bruker LynxEye detector). TL glow curves were recorded with the help of TLD reader 1000 by Nucletron (Hyderabad, India Pvt. Ltd.)

2 Results and discussion

2.1 Structural characterization

The phase purity and crystal structure of the pure $\text{Sr}_2\text{MgSi}_2\text{O}_8$ is determined by PXRD analysis. Figure 1 shows the PXRD pattern of the as prepared sample. The XRD pattern well matched with the JCPDS card no 10-04175 of $\text{Sr}_2\text{MgSi}_2\text{O}_8$ [6]. The XRD pattern does not show any significant change in any diffraction angle which confirms that the pure $\text{Sr}_2\text{MgSi}_2\text{O}_8$ was synthesized well. The formed phosphor has pure cubic crystal phase. The crystal size of the phosphor was determined by using Scherrer's formula [7], the crystal size obtained around 48 nm.

Surface morphology of the phosphor was determined by scanning electron microscopy (SEM) analysis. Figure 2 shows the SEM image of the prepared phosphor. From the image it is clear that the phosphor is agglomerated and has pseudo-spherical shape with regular morphological characteristics. Figure 3 represents the transmission electron microscopy (TEM) image of the prepared phosphor. The



Fig. 2 SEM image of $\text{Sr}_2\text{MgSi}_2\text{O}_8$

TEM image is in well agreement with the XRD data as the particle size obtained is around 50 nm range.

2.2 Thermoluminescence (TL) glow curve

TL behaviour of pure $\text{Sr}_2\text{MgSi}_2\text{O}_8$ has been studied under 254 nm UV excitation. The TL glow curve was recorded for 5 min UV exposure time at 4 °C s $^{-1}$. The T_m was obtained at 125 °C (Fig. 4).

The TL glow curve was recorded for different heating rate from 3 to 7 °C s $^{-1}$ for 5 min UV exposure time (Fig. 5). It was observed that the T_m shifts towards the higher temperature side with increase in heating rate along with the increase in intensity. The intensity increases from 3 to 4 °C s $^{-1}$ after that intensity starts to decrease. At lower heating rate the phosphor spent long time at a particular temperature so that the thermal recombination of electrons takes place at lower

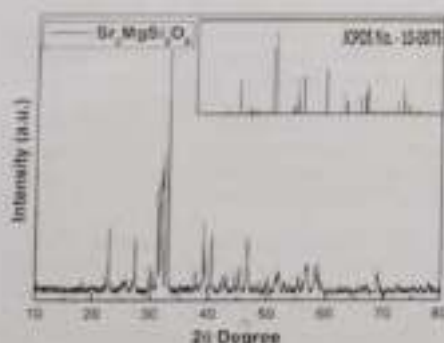


Fig. 1 XRD pattern of $\text{Sr}_2\text{MgSi}_2\text{O}_8$



Fig. 3 TEM image of $\text{Sr}_2\text{MgSi}_2\text{O}_8$

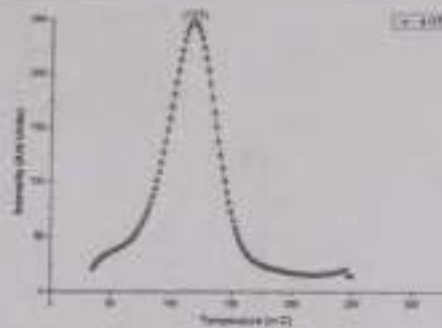


Fig. 4 TL glow curve of $\text{SrMgSi}_2\text{O}_6$ under 254 nm UV exposure for 3 min at 4°C s^{-1} heating rate

temperature. As the heating rate increases the time spent at a particular temperature decreases as a result the thermal recombination of electron also decreases. To release same number of electron it needs higher temperature, therefore the T_m shifts towards the higher temperature side [8].

From Fig. 5a, it was observed that the glow peaks were slightly shifting towards higher temperature side with increasing heating rate. This might be due to different types of luminescent centers/traps created and site symmetry of the incorporated dopant ion in the host lattice. For the phosphor to be quite useful in radiation dosimetry, linear response over a wide range of heating rate was expected. A good linearity was observed in the plot between heating rate and peak temperature from heating rate $5\text{--}7^\circ\text{C s}^{-1}$. The prepared phosphor shows opposite response with increasing heating when compared with the response of other phosphors towards the heating rate [2, 9, 10].

This behaviour can be explained by the fact that with lower heating rates the charge carriers travelling towards the recombination centers for producing the desired luminescence have enough time to get retrapped and do not involve in producing actual luminescence. Whereas, when the heating rate is high, phenomena such as thermal quenching of TL intensity due to larger heating rates arises. This requires an optimum heating rate at which maximum recombination may occur. Using the above results the heating rate was optimized to 4°C s^{-1} [11–13].

Kinetic parameters for all the peaks obtained for different heating rates were calculated by using Chen's peak shape method [14]. The activation energy ranges from 0.79 to 0.85 eV (Table 1). For lower heating rate TL glow peak is at lower temperature and for the same the activation energy also has lower value. The activation energy

value also increases with increase in T_m value. The value of shape factor is around 0.48 which shows the TL peak follows first order kinetics. The highest intensity was obtained for 4°C s^{-1} so that it was used as optimized heating rate [15–17].

The relationship between the frequency factor ' s ' and the activation energy ' E ' is given by the Eq. (1)

$$\frac{bE}{kT_m} = s \left[1 + (b-1) \frac{2kT_m}{E} \right] \exp(-E/kT_m) \quad (1)$$

where k is Boltzmann constant, E is activation energy, b is order of kinetics, T_m is temperature of peak position, and β is the heating rate.

3.3 Effect of UV exposure time

To study the effect of UV exposure time on TL behaviour the TL glow curve was recorded for the different UV exposure time from 5 to 10 min. The TL glow curve intensity increases with increasing UV exposure time up to 20 min UV exposure time after that quenching in TL intensity is observed (Fig. 6). With increasing the UV exposure dose the inter trap and recombination centres distance decreases as a result electron from one recombination centre can occupy another recombination centre as a result TL intensity increases. The sample shows resolved TL glow peak at 123°C . The peak intensity shows linear response up to 20 min UV exposure time after that the peak intensity decreases with increasing UV exposure time. These results show the prepared phosphor may be useful for UV dosimetric application. The activation energy for all the peaks were same i.e. 0.83 eV [18] (Table 2).

3.4 Analysis of TL glow curve

The TL glow curve obtained for optimized heating rate and UV exposure time was analysed by using CGCD method [19, 20]. As shown in Fig. 7, the deconvoluted curve has two peaks, which confirms that there is more than one imp level present inside the crystal. The kinetic parameters were calculated for both the deconvoluted peaks. Activation energy, shape factor and frequency factor were calculated by Chen peak shape method. The activation energy was found 0.39 eV and 0.90 eV respectively. The activation energy for both the peaks is very much different. Therefore it is clear that some traps are shallow and some are deep traps. For deconvoluted curve the shape factor is around 0.50, which shows that the TL glow curve follow first order kinetics (Table 3).

Fig. 3 a TL glow curve of $\text{Sr}_2\text{MgSi}_2\text{Cl}_6$ under 254 nm UV exposure for 3 min for different heating rates, b Heating rate versus peak temperature graph of $\text{Sr}_2\text{MgSi}_2\text{Cl}_6$ under 254 nm UV exposure for 3 min

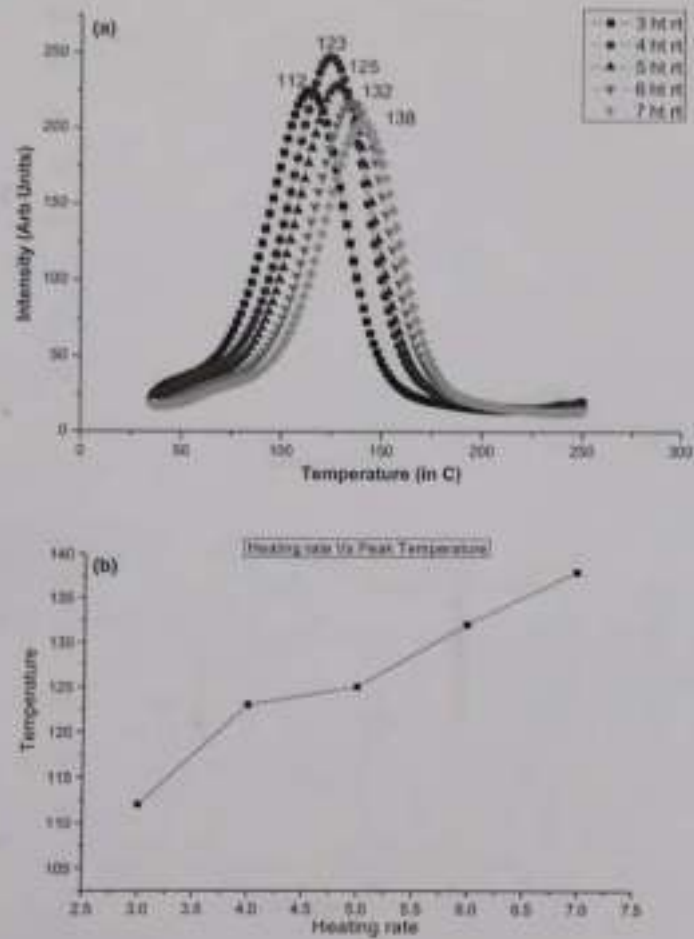


Table 1 Analysis of glow curves by $\text{Sr}_2\text{MgSi}_2\text{Cl}_6$ under 254 nm UV exposure for 3 min for different heating rates and its trapping parameters

Heating rate	T_1 (°C)	T_m (°C)	T_2 (°C)	τ (s)	δ (°C)	w (°C)	$\mu_2 = \delta/w$	Activation energy (eV)	Frequency factor (s^{-1})
3	88.1	112.2	135.2	24.1	23	47.1	0.48	0.79	4.5×10^{11}
4	95.5	122.8	145.6	24.3	22.8	47.1	0.48	0.93	6.94×10^{11}
5	101.3	126.3	149.3	25.2	23	48.2	0.48	0.81	3.5×10^{11}
6	108.3	132.4	156.3	24.3	22.9	47.2	0.48	0.88	1.3×10^{12}
7	113.1	138.7	163.2	25.6	22.5	48.3	0.47	0.85	4.6×10^{11}

Fig. 8 a TL glow curve of $\text{Sr}_2\text{MgSi}_2\text{O}_8$ under 254 nm for different UV exposure time at 4°C s^{-1} heating rate. b TL glow curve of $\text{Sr}_2\text{MgSi}_2\text{O}_8$ with intensity vs UV exposure time at 4°C s^{-1} heating rate

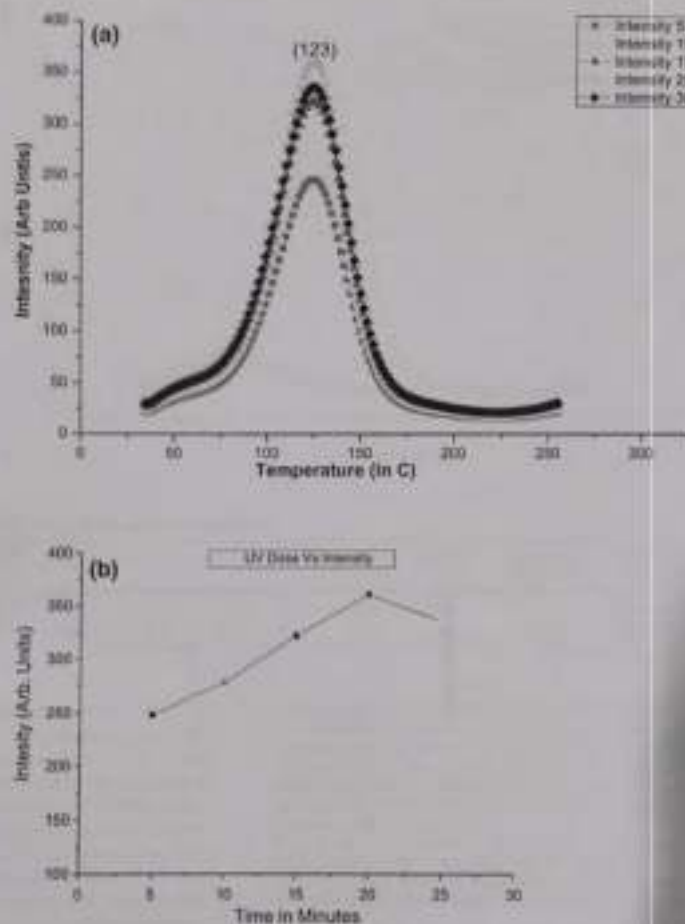


Table 2 Analysis of glow curves by $\text{Sr}_2\text{MgSi}_2\text{O}_8$ under 254 nm for different UV exposure time at 4°C s^{-1} heating rate

UV exposure time	T_1 (°C)	T_m (°C)	T_2 (°C)	τ (°C)	δ (°C)	ω (°C)	$\beta_0 = \beta_{\text{fit}}$	Activation energy (eV)	Frequency factor (s^{-1})
5–25	88.93	123.1	145.76	24.33	22.88	47.09	0.48019	0.82536	3.4×10^{11}

4 Conclusions

Pure $\text{Sr}_2\text{MgSi}_2\text{O}_8$ phosphor was synthesized by solid state synthesis method. The structural characterization was carried out by PXRD method and reveals the formation

of cubic phase phosphor. The crystal size was obtained around 48 nm by XRD method which was further confirmed by TEM analysis according to it the crystal size was obtained around 50 nm. A morphological study by SEM analysis confirms the presence of pseudo spherical shaped phosphors. TL glow curve was recorded for 254 nm UV

Fig. 2 Effect of UV exposure time on TL glow curve of $\text{Sr}_2\text{MgSi}_2\text{O}_8$ phosphor

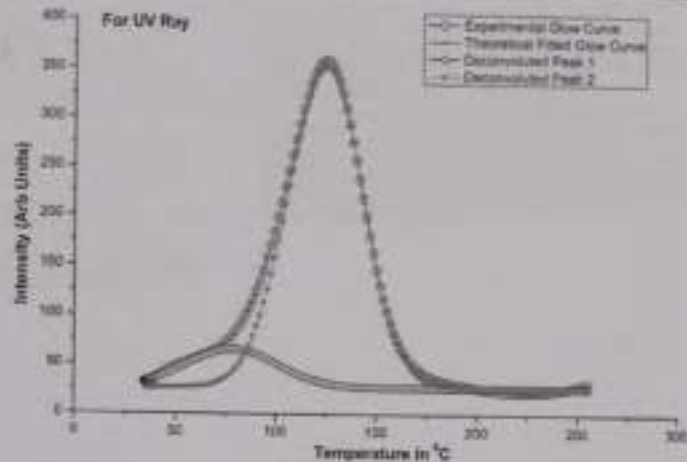


Table 2 Analysis of glow curves by CGCD curve fitting and trapping parameters

Phosphor name		T_1 (°C)	T_m (°C)	T_2 (°C)	τ (°C)	θ (°C)	ω (°C)	$A_0 = S_0$	Activation energy (eV)	Frequency factor (s^{-1})
$\text{Sr}_2\text{MgSi}_2\text{O}_8$	Peak 1	25.8	73.5	121.1	39.7	43.8	85.3	0.33	0.79	6.2×10^6
	Peak 2	100.3	122.8	145.7	22.2	22.8	45.4	0.30	0.90	5.7×10^{12}

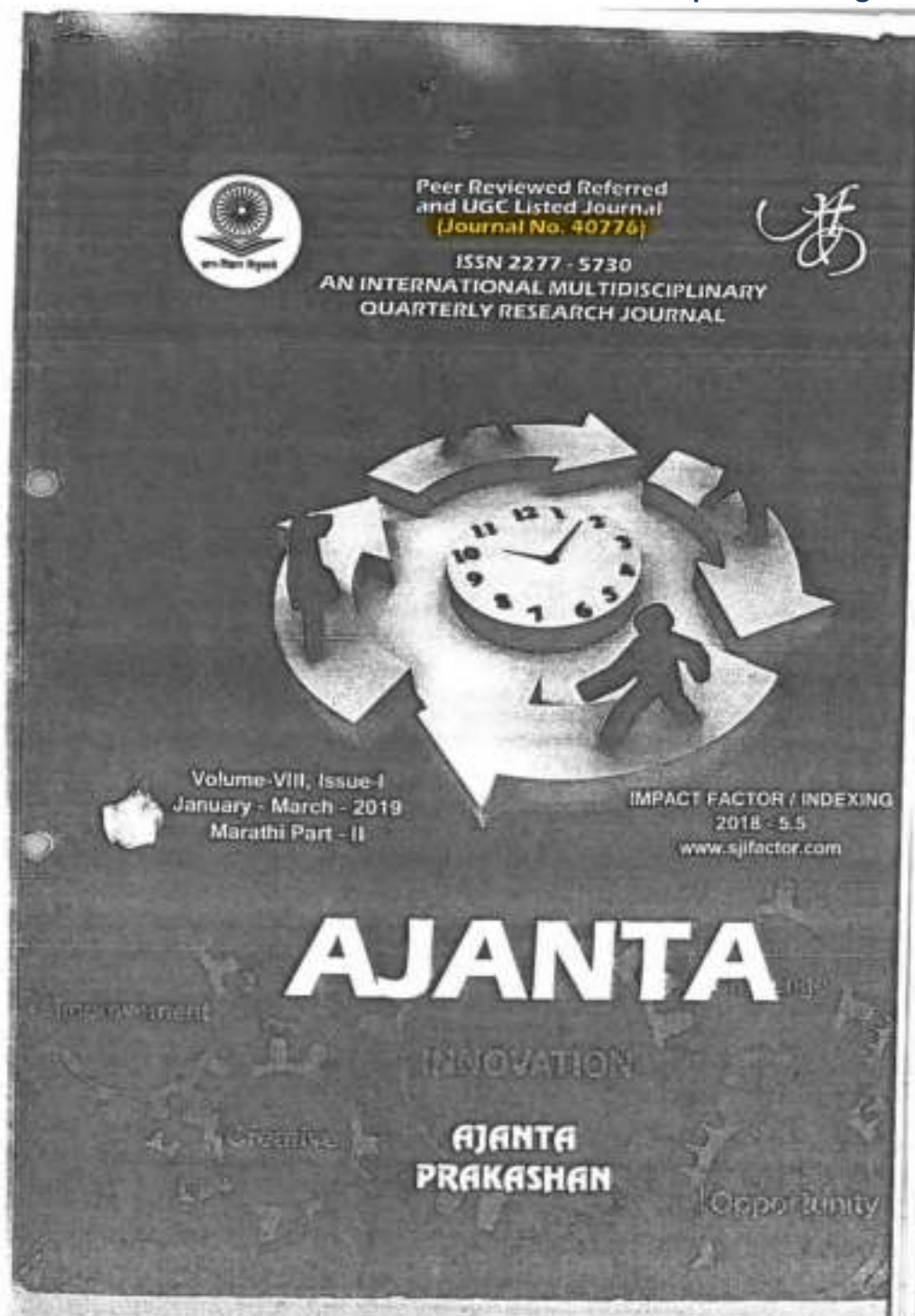
exposure. The heating rate was optimized at 4°C s^{-1} as the maximum intensity was obtained for that heating rate. The optimized UV exposure time was 30 min. The TL was analysed by using CGCD method and it shows presence of two peaks centred at 75°C and 123°C . The kinetic parameters were calculated for different heating rate, for UV optimization TL glow peak and for the CGCD curves. The activation energy was obtained around $0.79\text{--}0.90\text{ eV}$. For CGCD glow peaks also the activation energy was obtained as 0.90 eV for higher temperature peak. The shape factor value confirms the presence of first order peaks for all the TL glow peaks obtained.

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VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

CONTENTS OF MARATHI PART - II

अ.क्र.	लेख आणि लेखकाचे नाव	पृष्ठ क्र.
१	भारतीय लोकशाही व्यवस्थेचा घटनात्मक विकास : एक ऐतिहासिक समालोचन प्रा. डॉ. विशाल अ. दौलतकार	१-६
२	आधुनिक व्यापार पध्दती भारतीय व्यापार करिता एक आव्हान डॉ. प्रा. अश्वि दुळे	७-१०
३	ई-वाणिज्य आणि ऑनलाईन व्यापार प्रा. सुनिल एन. डेरे	११-१५
४	महाराष्ट्र राज्यातील इंजिनीयरींग महाविद्यालयांच्या संघातच वेबपेजेस द्वारे संघटनयामध्ये उपलब्ध इलेक्ट्रॉनिक पाठ्य सामुग्रीचा अभ्यास श्री विकास एस. मगराळे	१६-२३
५	भारतातील वर सुधारणांचा आख्या संदर्भ जी.एस.टी. प्रा. राजान्न रहाटे	२४-२८
६	ग्रामीण साहित्य संस्कृती व जागतिकीकरण प्रा. डॉ. मोना जुनघरे	२९-३०
७	विषयाच्या विकासात स्त्री शिक्षण तंत्राचे मूल्यमापक योगदान प्रा. डॉ. अर्चना पी. गुल्हाने	३१-३४
८	भारतातील शेतीसंबंधीत आव्हाने व उपाय डॉ. विठ्ठल धिनमिने	३५-४१
९	इतिहास आणि पर्यटन एकाच नाण्याच्या दोन बाजू डॉ. किशोर भुसजा अगुवार	४२-४४
१०	ऑनलाईन मार्केटिंग : नव उद्योजकता संघी प्रा. अमोल स. राऊत	४५-४८
११	व्यवसाय विकासातील अलसुक्त शिबिर योजनेचे माँगोलिक विश्लेषण प्रा. यशवंत प्रधान राठोड	४९-५२
१२	मानवी जीवनात पोषणाचे महत्त्व प्रा. कु. सरोज या. लखदिवे	५३-५५
१३	ऑनलाईन व्यवहार आणि त्याची उपयुक्तता प्रा. माधुरी पंडीतराव राखुडे	५६-५९

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
 AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

❧ CONTENTS OF MARATHI PART - II ❧

अ.क्र.	लेख आणि लेखकाचे नाव	पृष्ठ क्र.
२६	शैक्षणिक दृष्ट्या सगळी भाषा साहित्याचे मूल्य प्र. डॉ. सुवराज श्रीराम मानकर	११९-१२४
२७	शैक्षणिक मुल्यांचे सगळी भाषा व सगळी वाङ्मयातील महत्त्व व स्थान : एक आढावा प्र. सिध्दार्थ कुंडलिक इंगोले	१२५-१२८
२८	रोती व क्षेत्र व नानावी देशमुख कृषी संजीवनी योजना डॉ. शंकर मारोती सावंत	१२९-१३३
२९	जगाविश्वीकरणानंतर कृषी क्षेत्राची दशा व दिसा डॉ. दिपक जी. शृंगार	१३४-१३७
३०	इतिहास लेखनातील प्रवाह व पर्यटन प्र. डॉ. राहुल वि. दखणे	१३८-१४०
३१	भाषातंत्र विचारवंतांचा शैक्षणिक मूल्य विचार प्र. अनुप अरुण नांदगावकर	१४१-१४४

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 5.5 (www.sjifactor.com)

२९. जागतिकीकरणानंतर कृषी क्षेत्राची दशा व दिशा

डॉ. दिपक को. झंगरे

आदर्श महाविद्यालय, धामंगाव रेल्वे, जि. अमरावती.

प्रस्तुत लेखात जागतिकीकरणानंतर भारतातील कृषी क्षेत्रावर त्याचा अनुकूल आणि प्रतिकूल झालेला परिणाम व आर्थिक सुधारणानंतर कृषी क्षेत्राची झालेली दशा व दिशा याचा अभ्यास करण्यात आलेला आहे. भारतीय अर्थव्यवस्था ही कृषीप्रधान अर्थव्यवस्था म्हणून ओळखली जाते. प्राचीन काळापासून तर वर्तमान बदलत्या अर्थव्यवस्थेमध्येसुद्धा कृषी हा भारतीय अर्थव्यवस्थेचा कणा मानला जातो. या क्षेत्राला जनन्य साधारण महत्त्व आहे. राष्ट्रीय उत्पन्न समितीनुसार 1950-51 मध्ये भारताच्या सधुत देशांतर्गत उत्पादनात शेतीक्षेत्राचा वाटा 56.5 टक्के होता. त्याचबरोबर दोन तृतीयांश लोकसंख्या कृषी व कृषीआधारीत व पुरक व्यवसायात गुंतलेली आहे. म्हणून कृषी हा भारतीय अर्थव्यवस्थेचा आत्मा आहे.

1991 मध्ये भारताने मुक्त व्यापार धोरण शिक्कारले भारतीय अर्थव्यवस्था जागतिक अर्थव्यवस्थेची एकरूप व्हावी असे नियोजन आखण्यात आले. जागातील निरनिराळ्या देशांची आर्थिक व्यवहार याद्वारे परस्परांभी संबंध विकसित व्हावे अशी अपेक्षा करण्यात आली. त्यावेळचे पंतप्रधान म्. पी. वि. नरसिंहराव आणि अर्थमंत्री डॉ. नानो भारतीय अर्थव्यवस्थेला अधिक सक्षम बनविण्यासाठी नविन आर्थिक धोरणाचा शिक्कार केला. "कोणत्याही देशाच्या अर्थव्यवस्थेचे जागतिकीकरण म्हणजे देशांतर्गत किमतीचे अंतरराष्ट्रीय स्तरावरील किमतीची समानीकरण करणे हीच वास्तवी उत्पादक सक्षमता कोणत्याही देशात मुक्त प्रवेश होऊ देणे व त्या माध्यमातून उत्कृष्ट वाजारेपेठ निर्माण करून सर्वोपे स्वल्प प्राप्ता करून देणे होय". मुक्त आर्थिक धोरणामुळे प्रामुख्याने शेतीक्षेत्रावरील झालेल्या परिणामांचा आढावा पुढीलप्रमाणे आहे.

जागतिकीकरणाने कृषीक्षेत्रावर परिणाम

जागतिकीकरणामुळे नविन तंत्रज्ञान, सुधारीत बी-बियाणे, रासायनिक खते, किटकनाशके, अक्रुनिक कृषी अवजारे यांच्या वावरातुळे भारतीय कृषीच्या उत्पादनात वाढ होण्यास मदत झाली. पडीक जमीन लागवडीखाली आणून कृषी व कृषीआधारीत उद्योगाच्या गुंतवणूकीस कालना मिळाली. सहकार शेती, करार शेती, व्यापारी शेती, मटपती अशा नविन संकल्पना कृषी क्षेत्रात रुजू झाल्या तसेच मातृपालन, कुक्कुटपालन, पशुपालन, बैलीपालन, वस्त्र साहाम्यपालन इत्यादी कृषीला पुरक व्यवसायांना प्रोत्साहन मिळाले. हे जरी खरे असले तरी नंतरच्या काळात शेतीला वायदा कमी व नुकसान जास्त झाले.

भारताने आर्थिक नियोजनाची कात धरली तेव्हापासून कृषी क्षेत्राच्या विकासाकडे विशेष लक्ष देण्यात आले. विकासाकडे लक्ष देण्यात आल्यामुळे शेतीच्या विकासाला गती प्राप्त झाली त्यासाठी भारत सरकारने शेतीसहाय्यावर अनुदान मंजूर केले, सिंचनाच्या सुविधा वाढ केली, भूसुधारण कार्यक्रम राबविण्यात आला. या सवासा परिणाम भारत धनधान्य उत्पादनाच्या काहीप्रमाणात स्वतःपूर्ण झाला परंतु नंतरच्या काळात व्यावसायिक शेतीकडे शेतकऱ्यांचा गळ

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019
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पावू लागला. अन्नाधानाच्या उत्पादनाकडे दुर्लक्ष होवून फळपेटी, फुलपेटी या पिकांना प्राधान्य देण्यात आले. 1970 मध्ये हस्तिनाकातीने शेतीला जीवदान दिले परंतु 1981 नंतर भारतात मानिनिताने बहुउद्येय कंपन्यांनी प्रवेश केला. या कंपन्यांची बियाणे, रासायनिक खते, औषधे अकरावे यांनी भारतीय बाजारपेठ काबीज केली. याबाबतीत स्वयंजंबी असलेल्या शेतकरी बहुराष्ट्रीय कंपन्यांच्या उत्पादनातून आहारी गेला. त्यामुळे कृषीवर झालेल्या अनुकूल आणि प्रतिफल परिणाम पुढीलप्रमाणे सांगता येईल.

जागतिकीकरणाने कृषीक्षेत्रावर अनुकूल परीणाम

1. अन्नाधानाच्या उत्पादनात वाढ

आर्थिक सुधारणापूर्वी पहिल्या व दुसऱ्या पंचवर्षिक योजनेच्या काळात कृषी उत्पादनात वाढ झाली. सिंचनाच्या सुविधा वाढल्या, नगरी पिकांमध्ये कपूत, तीळ, जुट आणि कस या पिकांच्या उत्पादनात वाढ झाली. 1975 पर्यंत भारत अमेरिकेकडून अन्नाधानाची आयात करीत होता. अन्नाधानासारख्या वस्तुवस्त परदेशावर अवलंबून राहणे योग्य नाही. जागतिकीकरणामुळे हे अवलंबित्व घटत गेलेले दिसून येते.

2. राष्ट्रीय उत्पादनात कृषी क्षेत्राचा वाटा

राष्ट्रीय उत्पादनात कृषी क्षेत्राचा मोठा हिस्सा राहिलेला आहे. विकसित राष्ट्रात हा वाटा 2 ते 5 टक्क्यांच्या जवळपास दिसून येतो. जागतिकीकरणामुळे आधुनिक संज व संज्याच्या साहाय्याने उत्पादन घेण्यात आले त्यामुळे राष्ट्रीय उत्पादन कृषी क्षेत्रात वाटा वाढत गेलेला दिसून येतो.

3. शेजारांच्या संघीत वाढ

भारतीय अर्थव्यवस्थेत शेती व शेतीपुरक तसेच शेतीवर आधारित प्रक्रिया उद्योगात शेजारा देण्याची क्षमता जास्त आहे. कारण यात अल्पप्रधान संज्या वापर अधिक होते. शेती व उद्योग यांच्या परस्पर संबंधामुळे अन्नाधान प्रक्रिया उद्योग, व्यापार, दळणवळण सेवा इत्यादी क्षेत्रातूनही शेजारांच्या सोयी निर्माण झाल्या.

4. विदेशी व्यापारात वाढ

नविन आर्थिक धोरणत आयात व निर्यातीवरील प्रतिबंध कमी झाले. अतिकडच्या काळात भारताच्या निर्यातीत कृषी व कृषी संलग्न वस्तुचे योगदान 60 टक्के आहे. त्यामुळे निर्याती व मुल्यात वाढ झालेली दिसून येते. यात प्रामुख्याने बहू, तंबाखू, तेलबिया, मसाल्याचे पदार्थ इत्यादींचा समावेश आहे.

5. बाजारपेठेचा विस्तार

कृषीमालाच्या निर्यातीसाठी बाजारपेठेचा विस्तार झाल्यास शेतीची उत्पादन क्षमता वाढण्यास प्रोत्साहन मिळते. आधुनिकीकरणामुळे कृषीमालाचे उत्पादन वाढले. त्या उत्पादनाला जागतिक बाजारपेठ प्राप्त झाली व शेतकऱ्यांच्या कृषी मालाला योग्य भाव मिळाला.

6. नैसर्गिक सधनांचा उपयोग

कृषिच्या विकासदराने देशातील लघु व कुटीर उद्योगांना कच्चा मालाचा पुरवठा झाला. शेतमालाला मागणी वाढत गेल्यामुळे शेतीत सुगीचे दिवस आले. शेतकऱ्यांची खरेदी शक्ती वाढली. त्यांच्या राहणीमानात सुधारणा झाली नैसर्गिक साधनसामग्रीचा वापर योग्य प्रमाणात झाल्यामुळे कृषिवर आधारित छोट्या मोठ्या उद्योगांचा विकास झाला.

VOLUME - VIII, ISSUE - I - JANUARY - MARCH - 2019

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मुक्त व्यापार धोरणामुळे जगाप्रमाणे कृषीवर अनुसूल प्रभाव झाला त्याचप्रमाणे जागतिकीकरण वरदान कमी व हात अधिक अमी परीस्थिती निर्माण झाली. मुक्त बाजारपेठेमुळे स्वदेशी बाजारात बाह्यराष्ट्रीय कंपन्यांनी निर्यात केल्यामुळे विदेशातील कृषीमात खस्त किमतीला मिळू लागला. कृषी व्यवस्थेचे मांडवतीकरण झाले. भारतीय कृषी मालांना योग्य किंमता मिळेनाही अशी त्यापुढेच निराशाच्या लहरीपणामुळे भारतीय शेती तोंटवात गेली. विकसित राष्ट्रांच्या दबावामुळे शेतीक्षेत्रातील अनुदान कमी करण्यात आले. म्हणजेच जागतिकीकरणाच्या काळात कृषी व संबंधित क्षेत्राचा दर घटल्याचे दिसून आले. त्याचप्रमाणे कृषीक्षेत्रातील सार्वजनिक गुंतवणुकीचा खटा इतक्या कमी होत गेला. शेतीच्या प्रगतीवर जागतिकीकरणाने झालेले प्रतिकूल परिणाम पुढीलप्रमाणे आहेत.

जागतिकीकरणाने कृषीक्षेत्रावर प्रतिकूल परिणाम

1. शेतीच्या उत्पादन खर्चात वाढ

मुक्त आर्थिक दोरग्यांनंतर बाजारपेठेत कृषीमात विकित स्वर्ण निर्माण झाली. नि-बिद्याणे, खते, फिटकणामुळे वाढत्या किंमती जास्त प्रमाणात वाढल्या. याबिवाध शेतीसाहित्य मोठी अकडारे यांची आवश्यकता वाढली अनुदान कमी झाले. निराशाची अवस्था या सर्वांचा परिणाम शेतकऱ्यांचा शेतीलागवडीचा खर्च वाढला.

2. अन्न सुरक्षेचा प्रश्न

जागतिकीकरणामुळे अन्नाधानाच्या उत्पादनात वाढ झाली परंतु अन्नाधानाच्या तुलनेत व्यापारी पिकांचे उत्पादन जास्त प्रमाणात वाढत गेले. यामुळे अन्नसुरक्षेचा प्रश्न निर्माण झाला. वाढत्या लोकसंख्येच्या तुलनेत अन्नाधाना उत्पादन यादीचा दर कमी झाला. त्याचा परिणाम अन्नाधानाच्या किमती वाढत गेल्या. भारत हा कृषिप्रधान देश असून खुद्दा या काळात भारताला अन्नाधानाची आयात करावी लागली.

3. कृषिक्षेत्रातील रोजगारीत घट

भारतातील 70 टक्के लोकसंख्या कृषिवर प्रत्यक्ष अवलंबित्वा अवलंबून आहे. रोजगार देण्याचे प्रमुख क्षेत्र म्हणजे कृषी होय. परंतु जागतिकीकरणानंतर शेतीचा लागवड खर्च कमी करण्यासाठी आधुनिक यंत्र व तंत्राचा वापर करण्यात आला. मांडवतीकरण तंत्राकडे शेतकऱ्यांचा कल वाढल्यामुळे शेतीक्षेत्रात रोजगाराच्या संधी कमी झाल्या.

4. कृषिक्षेत्रातील अनुदानात कपात

जागतिक व्यापार संघटनेच्या अटीनुसार विकसनशील देशांनी 13.3 टक्के अनुदानात कपात करावी तर विकसित देशांची 20 टक्के कपात करावी असा प्रस्ताव मांडण्यात आला. विकसनशील राष्ट्रांनी अनुदानात कपात करण्यात सुरुवात केली. परंतु विकसित राष्ट्रांनी या अटीचे पालन न करता अनुदानात वाढ केली. त्यामुळे भारतीय शेतीतील मागासलेपणा वाढला. 1991 नंतर भारत सरकारने धान्य, खते, विद्याणे, फिटकणामुळे, विज इत्यादीवर दिल्या जाणाऱ्या अनुदानात सलगत्याने घट केली.

5. शेतकऱ्यांच्या आत्महत्येची समस्या

भारतात प्रामुख्याने महाराष्ट्र, अंध्रप्रदेश इत्यादी राज्यात शेतकऱ्यांच्या आत्महत्या केल्या आहेत. याचे कारण ससाजनिक खत, फिटकणामुळे, विद्याणे, मजुरीचे उच्च दर, शेतमागाला कमी भाव, सरकारने अनुदानात केलेली कपात आणि संघटीत क्षेत्रातील कार्यवाजारीपणा हे आहे. शेतकऱ्यांची स्वर्ण जागतिक बाजारपेठेची

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आहे. सावकारी कर्जांचा सापळा, हलात, मोठे व्यापारी यांचे बाजारपेठेत वर्षेभर विकृत येते. या धनिक वर्गासमोर शेतकरी हलाक झाला आहे. त्यामुळे शेतकऱ्यांच्या आत्महत्येची निव्वणता दिवसेंदिवस वाढत आहे.

जागतिकीकरणामुळे आंतरराष्ट्रीय बाजारात शेतीमालाचा योग्य भाव मिळेल, शेतमालाची निर्यात वाढेल, शेतकऱ्यांचा नफा वाढेल, त्यांचे दरबोई उत्पन्न वाढून राहणीमानात सुधारणा होईल अशी अपेक्षा होती. पण असा अनुभव आला नाही. विकसित राष्ट्रांनी त्यांच्या देशात दिली जाणारी शेती क्षेत्राला अनुदाने कमी करायला, शेतमालाच्या आयातीवर लादलेली बंधने शिथिल करायला त्यामुळे भारतीय कृषीला शुगीचे दिवस येतील. त्यासाठी कृषीमध्ये कामगाररूपाच्या सुधारणा करणे आवश्यक आहे. शेतकऱ्यांच्या शेतमालास निर्यात सक्ता मिळून देणे. संरक्षण व अर्थसाहाय्य या बाबीकडे लक्ष देवून शेतकऱ्यांच्या वाढत चाललेल्या जलमहत्या कमी करायला येईल व शेतीकडे पाहण्याचा सकारात्मक दृष्टिकोन निर्माण करण्यासाठी सरकारने स्वतंत्र कृषीआयोग स्थापन करावा. कृषीमालाचा आंतरराष्ट्रीय बाजारपेठ उपलब्ध करून देण्यासाठी पुढाकार घ्यावा लागेल.

संदर्भ सूची

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2019-2020

Cr(III),Mn(III),Fe(III) and VO(IV) complexes of 2,4-dihydroxyacetophenone and sulphapyridine.: Vishnu Anandrao Sadafale



“Cr(III), Mn(III), Fe(III) and VO(IV) complexes of 2,4-dihydroxyacetophenone and sulphapyridine.”

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Abstract: Complexes of Cr(III), Mn(III), Fe(III) and VO(IV) with Schiff base derived from 2,4-dihydroxyacetophenone and sulphapyridine were synthesized and characterized using elemental analysis, molar conductance, magnetic susceptibility measurements, spectral studies (IR, ¹H NMR, UV-Visible) mass and TGA. Elemental analysis of the complexes suggested 1:2 metal:ligand stoichiometry in case of VO(IV) complex whereas 1:1 metal:ligand ratio for Cr(III), Mn(III) and Fe(III) complexes. The molar conductance measurements reveal the non-electrolyte nature of all the complexes. IR spectra of the compounds show monobasic bidentate (ON) nature of the ligand, coordinating mainly through hydroxyl oxygen and azomethine nitrogen. The electronic spectral data supported octahedral geometry for Cr(III), Mn(III) and Fe(III) complexes while square pyramidal for VO(IV) complex. Thermogravimetric analysis shows that the complexes are thermally more stable than that of the ligand. Kinetic parameters were calculated by using Freeman-Carroll and Sharp-Wentworth methods and results are comparable. All the compounds have been tested *in vitro* to evaluate their antimicrobial activity against *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Klebsiella pneumoniae* using well-diffusion method.

Index Term: 2,4-dihydroxyacetophenone, sulphapyridine, well-diffusion.

I. INTRODUCTION

Recently the research field dealing with Schiff base coordination chemistry has expanded enormously. The importance of Schiff base complexes for bioinorganic chemistry, biomedical applications, supramolecular chemistry, catalysis and material science, separation and formation of compounds with unusual properties and structures has been well recognized and reviewed [1]. In addition to important role of Schiff base complexes in catalysis and organic syntheses, they have also clinical, analytical and industrial applications [2].

Sulphur ligands are widespread among coordination compounds and are important components of biological transition metal complexes which possess many applications such as diuretic, antidiabetic, antidiarrhoeal or antiepileptic drugs. [3]

The biological activity of sulphur containing ligand increases on complexation. salicylaldehyde, benzaldehyde and furfural moiety when coupled with sulfamides yields sulfamide-imine which may show superior biological activity.

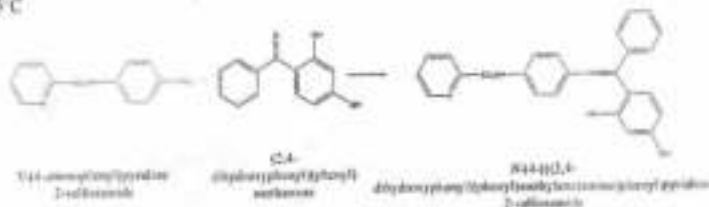
II. Material and Method

All the metal salts and solvents used were analytical reagent grade supplied by SD Fine and Merck. Pure sample of dopamine was obtained from Shri Scientific Pvt. Ltd, Mumbai. Solvents were purified and dried before use by literature method [4]. The ligands used in the present work are not commercially available, hence were synthesized in laboratory. Manganese acetate dihydrate was prepared by the oxidation of Mn(OAc)₂·4H₂O using Christensen's method [5] while all other metal salts are commercially available and used as received. The amounts of metal present in the complexes were estimated by metal oxide method [6]. The molar conductance of the complexes in dimethyl formamide (DMF) solution (10⁻³ M), was measured using Equiptronic digital conductivity meter EQ-660 with a cell constant of 1.00cm² at room temperature. The infrared Fourier-transform infrared (IR) spectra of ligand and complexes were recorded as KBr pellets using Shimadzu spectrometer (IR Affinity-1S). The thermal analysis was performed with a Perkin Elmer (TGS-2 model) thermal analyzer at a heating rate of 10°C min⁻¹ in the temperature range 40-500°C.

Synthesis of N-(4-((2,4-dihydroxy(phenyl)methylene)amino)phenyl)pyridine-2-sulfonamide (L¹H₂)

Equimolar (0.01m) solutions of 2,4-dihydroxyacetophenone (2.14g) and sulphapyridine (2.49g) were separately dissolved in ethanol and refluxed for three hours. The volume of reaction mixture was reduced to one third and cooled at 0°C. The solid residue was filtered off and recrystallized by ethanol. The purity of the compound was checked by thin layer chromatography (TLC).

Yield- 80%, M.P- 145°C



Synthesis of Metal complex

The complexes were synthesized by adding ligand and respective metal salt, i.e., CrCl₃·6H₂O, Mn(OAc)₂·2H₂O, FeCl₃·6H₂O, VO(SO₄)₂·5H₂O in 1:1 ratio to the ethanol. Few drops of 1N KOH were added to the reaction mixture and the reaction mixture was refluxed in water bath for about 10-12 h. The coloured solids obtained were filtered off, washed several times with small amount of ethanol followed by petroleum ether and finally dried over fused calcium chloride.

III. Results & Discussion

In the present study, all the complexes are found to be coloured solids, air stable and non-hygroscopic. The analytical data indicate 1:1 metal to ligand stoichiometry. The complexes are insoluble in water and common organic solvents but soluble in coordinating solvents like DMF and DMSO. Lower values of molar conductance indicate that complexes are nonelectrolytes. The analytical and physical data of ligand and its complexes are given in Table 1.

Table 1: Elemental analysis and kinetic data for ligand and metal complexes

S. N.	Compounds	Formula weight g mol ⁻¹	Colour	Time of Reflux	Elemental Analysis		
					C%	H%	N%
1	L ⁹⁴	443.11	Yellow	3 h	64.71 (64.69)	4.30 (4.28)	9.43 (9.36)
2	Cr- L ⁹⁴ complex	621.43	Brown	10 h	46.39 (46.47)	3.89 (3.98)	6.76 (6.83)
3	Mn- L ⁹⁴ complex	671.56	Black Cherry	11 h	50.08 (50.17)	4.50 (4.81)	6.26 (6.06)
4	Fe- L ⁹⁴ complex	625.28	Black	12 h	46.10 (46.31)	3.87 (3.94)	6.72 (6.87)
5	VO- L ⁹⁴ complex	955.21	Brown	11 h	60.31 (60.45)	3.80 (3.95)	8.79 (8.91)

¹H NMR spectra of ligand

The ¹H nuclear magnetic resonance (NMR) spectra of Schiff base ligand was recorded in dimethyl sulfoxide (DMSO) using TMS as an internal standard. The spectrum shows following peaks.

¹H NMR (DMSO) δ ppm: 12.5 (s, 2H, phenolic OH), 5.90-7.69 (m, 16H, aromatic), 2.61 (s, 3H, -CH₃) [7-9].

IR Spectra

To study the binding modes of ligand toward metal ion IR spectral data of Schiff base ligand was compared with the complexes (Table 2). The broad band that appeared in the spectra of the ligand and their metal complexes in the range 3309-3402 cm⁻¹ is assigned to the stretching vibration of ν(N-H) [10-11]. The IR spectral bands in the spectra of the ligand and its metal complexes indicates that the band at 3232 cm⁻¹ due to intramolecular hydrogen bonded ν(OH) group in the free ligand spectrum disappeared in spectra of all the complexes suggesting the coordination of ligand through phenolic oxygen via deprotonation [12]. Further the bands at 1620 cm⁻¹ (aromatic C=N) shifted its position to lower frequency on complexation [13]. The bands at 1350 cm⁻¹ and 1122 cm⁻¹ in the ligand are assigned to ν₂(SO₂) and ν₁(SO₂) [14-15]. These bands remain almost at the same position in the complexes suggesting non-involvement of sulfonyl oxygen in coordination with the metal. The spectra of VO(IV) complex show new band at around 994 cm⁻¹ due to ν(V=O) vibration. The ν(M-N) and ν(M-O) bands have been assigned in the region 505-559 cm⁻¹ and 443-459 cm⁻¹ respectively [16]. The coordination of water in Cr(III), Mn(III) and Fe(III) complexes is indicated by appearance of bands at 3410- 3466 cm⁻¹, 1536-1546 cm⁻¹, 820-840 cm⁻¹, 756-771 cm⁻¹ assignable to ν(OH), δ(OH), ν(H₂O), ρ(H₂O) mode respectively [17-18].

Table 2: IR spectral data for Schiff base ligand and its metal complexes

S. N.	Compound	ν(O-H) hydrogen bonded	ν(NH)	ν(C=N)	ν(SO ₂)		ν(M-O)	ν(M-N)
					asym	sym		
1	L ⁹⁴	3232	3402	1620	1350	1122	—	—
2	[Cr(L ⁹⁴)]	—	3335	1598	1338	1135	540	446
3	[Mn(L ⁹⁴)]	—	3303	1596	1340	1122	542	458
4	[Fe(L ⁹⁴)]	—	3321	1607	1369	1145	559	458
5	[VO(L ⁹⁴)]	—	3358	1600	1332	1128	536	454

Mass Spectra

Mass spectrometry has been successfully used to determine the molecular ion peak for Schiff base ligand. The various fragmentation peaks obtained for ligand, Cr(III), Fe(III), Mn(III) and VO(IV) complexes are in good agreement with proposed structure. Mass spectrum of ligand was shown in Figure.

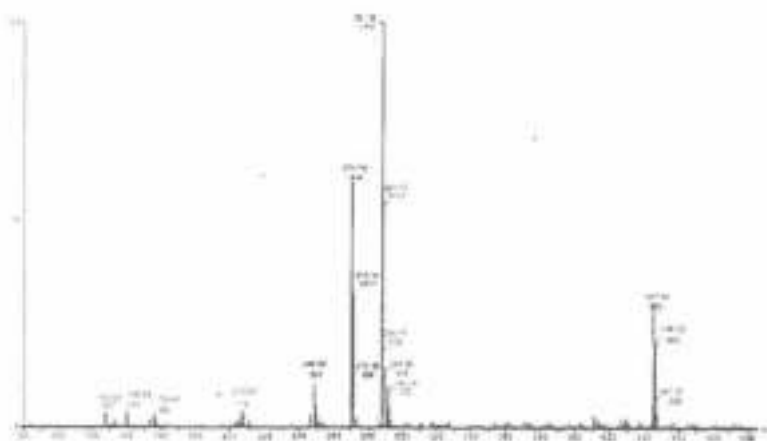


Fig.2 Mass spectrum of Ligand L'H1.

Electronic Spectra

The electronic absorption spectra of metal complexes were recorded in DMSO in the range 200-800 nm. The electronic spectrum of Cr(III) complex shows bands at 302, 462 and 607 nm corresponding to transition ${}^4A_{1g} \rightarrow {}^4T_{1g}(F)$, ${}^4A_{1g} \rightarrow {}^4T_{2g}(F)$, ${}^4A_{1g} \rightarrow {}^4E_g(F)$ respectively, indicating octahedral geometry around Cr(III) ion [19]. For Mn(III) complex band appears at 288, 550 and 800 nm for transition ${}^6B_{1g} \rightarrow {}^6E_g$, ${}^6B_{1g} \rightarrow {}^6A_{1g}$ and ${}^6B_{1g} \rightarrow {}^6E_g$. All these transitions indicate octahedral geometry for Mn(III) complex [20]. The electronic spectrum of Fe(III) complex shows bands at 780, 539 and 343 nm corresponding to transition ${}^6A_{1g} \rightarrow {}^6T_{1g}(D)$, ${}^6A_{1g} \rightarrow {}^6T_{2g}$ and LMCT transition respectively, indicating octahedral geometry around Fe(III) ion [21]. The electronic spectrum of VO(IV) complex shows bands at 809, 463, 247 and 208 nm corresponding to transition ${}^3B_2 \rightarrow {}^1A_1$, ${}^3B_2 \rightarrow {}^1E$, CT respectively, suggesting square pyramidal geometry for complexes [22]. The absorption region, band assignment and proposed geometry of the complexes are given in Table 3.

Table 3: Electronic absorption spectral data of the complexes.

Compounds	μ_{eff} B.M.	Δ_{oct} cm^{-1}	Absorption (nm)	Band assignment	Geometry
[Cr (L'H)]	3.64	10.12	288 482 671	${}^4A_{1g} \rightarrow {}^4T_{1g}(F)$ ${}^4A_{1g} \rightarrow {}^4T_{2g}(F)$ ${}^4A_{1g} \rightarrow {}^4E_g(F)$	octahedral
[Mn (L'H)]	5.10	13.25	275 579 797	${}^6B_{1g} \rightarrow {}^6E_g$ ${}^6B_{1g} \rightarrow {}^6A_{1g}$ ${}^6B_{1g} \rightarrow {}^6E_g$	octahedral
[Fe (L'H)]	3.50	8.24	762 537 343	${}^6A_{1g} \rightarrow {}^6T_{1g}(D)$ ${}^6A_{1g} \rightarrow {}^6T_{2g}$ LMCT	octahedral
[VO (L'H)]	1.62	8.92	809 463 247 208	${}^3B_2 \rightarrow {}^1A_1$ ${}^3B_2 \rightarrow {}^1E$ LMCT LMCT	square pyramidal

Thermal Analysis

Thermal analysis is performed to determine the thermal stability of complexes and its degradation pattern in which the change in the weight of the substance is recorded as function of temperature or time. Thermogravimetric analysis (TGA) was performed in nitrogen atmosphere with heating rate $10^{\circ}\text{C min}^{-1}$ and thermograms were recorded in temperature range $40-500^{\circ}\text{C}$. Using this technique several kinetic parameters such as activation energy (Ea), order of reaction (n), entropy change (ΔS), free energy change (ΔF), apparent entropy change (ΔS^*) were calculated. The thermal data have been analyzed using Freeman- Carroll and Sharp-Wentworth methods [23-24].

An analysis of TG curves of L^{H} and its complexes reveals a four stage decomposition pattern for Cr(III) , Fe(III) and Mn(III) complexes and one stage decomposition pattern for ligand and VO(IV) complex. All the complexes except VO(IV) are stable upto $60-70^{\circ}\text{C}$. There is a weight loss upto 120°C in the VO(IV) complex indicating the absence of any water molecules (lattice or coordinated). The TG curve of Cr(III) , Fe(III) and Mn(III) complexes are stable upto 60°C . Decomposition of one lattice water was observed for Cr(III) , Fe(III) and Mn(III) complexes at 120°C with % weight loss observed: Cr(III) : 2.97/2.90, Mn(III) : 2.73/2.68, Fe(III) : 2.95/2.88. The further weight loss was observed for Cr(III) , Fe(III) and Mn(III) complexes from $120-220^{\circ}\text{C}$ indicates the presence of two coordinate water molecules in Cr(III) , Fe(III) and Mn(III) complexes [% weight loss observed: Cr(III) : 5.85/5.80, Mn(III) : 5.43/5.36, Fe(III) : 5.81/5.76]. In all the complexes continuous and rapid weight loss has been observed above 220°C corresponding to thermal degradation of free part of the coordinated ligand along with the other groups present there in. A gradual weight loss above 340°C corresponds to the degradation of actual coordination part of the ligand. Finally the horizontal level beyond 440°C suggests the formation of final decomposition products corresponding to respective metal Cr_2O_3 , Mn_2O_3 , Fe_2O_3 and V_2O_5 . Kinetic parameters are assigned in Table 4. The relative thermal stability on the basis of half decomposition temperature is found to be $\text{Fe(III)} > \text{VO(IV)} > \text{Cr(III)} > \text{Mn(III)} > \text{L}^{\text{H}}$.

Table 4: Thermal decomposition data of ligand and its complexes

Compound	Half decomposition temperature ($^{\circ}\text{C}$)	Activation energy (kJ/mol)		Order of reaction (n)	Entropy change ΔS (J/mol/K)	Free energy change ΔF (kJ/mol)	Apparent entropy change ΔS^* (kJ)
		FC	SW				
L^{H}	220	24.38	24.55	0.95	-278.96	111.69	-38.76
$[\text{Cr}(\text{L}^{\text{H}})]$	280	12.25	12.44	0.99	-274.588	98.20	-37.62
$[\text{Mn}(\text{L}^{\text{H}})]$	270	13.56	13.72	0.97	-320.643	115.92	-37.91
$[\text{Fe}(\text{L}^{\text{H}})]$	350	22.45	22.63	0.98	-313.261	120.50	-38.71
$[\text{VO}(\text{L}^{\text{H}})]$	310	29.82	29.88	0.99	-278.315	116.93	-38.90

Antimicrobial activity

Antibacterial screening of ligand (L^{H}) and its complexes against *E. coli*, *S. aureus*, *P. aeruginosa* and *K. pneumoniae* strains were carried out. Ligand L^{H} found to be active against all bacterial species where it showed high activity towards *K. pneumoniae* and moderate activity towards *S. aureus*, *E. coli* and *P. aeruginosa*. Ligand and all metal complexes were bactericidal towards *E. coli*. All metal complexes showed good to moderate activity against *S. aureus* except Fe(III) and VO(IV) . Bacteriocidal activity of ligand decreased on complexation with metal ions for bacteria *K. pneumoniae*. Results of studies of antibacterial effect of ligand (L^{H}) and its complexes are summarized in Table 5.



Figure: Photographic plate showing zones of inhibition in *S. aureus*

Figure :Photographic plate showing zones of inhibition in *P. aeruginosa*.

Table 5: Results of antibacterial effect of ligand and its metal complexes

Sr. No.	Ligand and its complexes	<i>E. coli</i> (mm)	<i>S. aureus</i> (mm)	<i>P. aeruginosa</i> (mm)	<i>K. pneumoniae</i> (mm)
1	L'H ₂	S ₁₀	S ₂₅	S ₁₀	S ₁₇
2	Cr-L'H ₂	S ₁₁	S ₁₃	S ₁₂	S ₁₈
3	Mn-L'H ₂	S ₁₁	S ₁₃	S ₁₆	S ₁₇
4	Fe-L'H ₂	S ₁₁	R	R	R
5	VO-L'H ₂	S ₁₂	S ₁₁	R	R

Conclusion

The present article includes the synthesis and characterization of new Schiff base ligand and its Cr(III), Mn(III), Fe(III) and VO(IV) complexes. Characterization of compounds includes IR, mass, ¹H NMR, and TGA. Electronic spectrum suggests octahedral geometry for Cr(III), Mn(III) and Fe(III) complexes and square pyramidal geometry for VO(IV) complex. The thermal study revealed that complexes are thermally stable. The relative thermal stabilities of the complexes was in the order Fe(III) > VO(IV) > Cr(III) > Mn(III) > L'H₂. The Schiff base ligand and its complexes have been tested *in vitro* to evaluate their antimicrobial activity against *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Klebsiella pneumoniae* using well-diffusion method. It has been found that the Schiff base ligand and its complexes show significant antimicrobial activity.

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SYNTHESIS AND ANTIBACTERIAL SCREENING OF SCHIFF BASES DERIVED FROM 3-(5-BROMOTHIOPHEN-2-YL)-1-(4-CHLOROPHENYL)-1H-PYRAZOLE-4-CARBALDEHYDE:

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SYNTHESIS AND ANTIBACTERIAL SCREENING OF SCHIFF BASES DERIVED FROM 3-(5-BROMOTHIOPHEN-2-YL)-1-(4-CHLOROPHENYL)-1H-PYRAZOLE-4-CARBALDEHYDE

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Keywords:
Hydrazone,
Pyrazole Schiff bases,
Antibacterial activity

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ABSTRACT: A series of new Schiff bases were synthesized by condensation of 3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazole-4-carbaldehyde with different aromatic aldehydes. The 3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazole-4-carbaldehyde was prepared from 1-(1-(5-bromothiophen-2-yl)ethylidene)-3-(4-chlorophenyl)hydrazine by the Vilsmeier Haack reaction. The 1-(1-(5-bromothiophen-2-yl)ethylidene)-3-(4-chlorophenyl) hydrazine was prepared by the condensation reaction of 3-acetyl-5-bromothiophene with 4-chlorophenylhydrazine hydrochloride. The structures of newly synthesized compounds were elucidated by NMR, IR, and Mass spectral data. Prepared Schiff bases were evaluated for antibacterial activity against four organisms: *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Klebsiella pneumoniae* using streptomycin as a standard drug. Agar well-diffusion method was followed to determine the antimicrobial activity. All prepared Schiff bases showed poor to good activity against test organisms. Based on the zone of inhibition results, it is observed that the newly prepared Schiff bases showed better activity against *Klebsiella pneumoniae* than *Escherichia coli*, *Staphylococcus aureus*, and *Pseudomonas aeruginosa*. Schiff base 4c showed good activity against *Pseudomonas aeruginosa*. Schiff base 4i showed good activity against *Staphylococcus aureus* and all the Schiff bases 4a-4i except 4c showed good activities against *Klebsiella pneumoniae*.

INTRODUCTION: Heterocyclic compounds have great importance in medicinal chemistry. Pyrazole is an important class of heterocyclic compounds widely used in various areas of chemistry and related sciences.

Pyrazole derivatives have attracted much attention of chemists on account of their wide applications in medicinal chemistry.

These compounds show antimicrobial^{1,2} anti-inflammatory^{3,4} antitubercular⁵, antimalarial⁶, insecticidal⁷, antifungal^{8,9} and anticancer^{10,11} activities. Schiff bases an important scaffold have attracted the interest of chemists due to wide applications in synthetic chemistry and biological point of views. They are also serving as backbone for the synthesis of various heterocyclic compounds.

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They are important compounds due to their wide range of pharmacological activities such as antimicrobial¹⁴⁻²⁰, anticancer²¹⁻²², antifungal^{23, 24}, anti-inflammatory^{25, 26} and DNA- cleavage²⁷ activities. In the present article, we present the synthesis, characterization and antimicrobial evaluation of Schiff bases prepared from 3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazole-4-carbaldehyde.

MATERIALS AND METHODS: All the chemicals were procured from Sigma Aldrich and SDFINE chemicals and used without further purification. Melting points were determined in open capillary tube and are uncorrected. The progress of the reactions as well as purity of the compounds was monitored by thin layer chromatography with F-252 silica gel precoated aluminum sheets using petroleum ether-ethyl acetate (9:1) as a developing solvent and spots were visualized with near UV and Iodine. The IR spectra were recorded on Shimadzu spectrophotometer using KBr pellets and expressed in cm^{-1} . The NMR spectra were recorded on BRUKER ADVANCE (400 FT-NMR) spectrophotometer in DMSO using tetramethylsilane as an internal standard, and chemical shift values are expressed in δ ppm. The MASS spectra were recorded on Waters UPLC-TQC MASS spectrometer.

Procedure for Preparation of (hydrazone) 1-(1-(5-bromothiophen-2-yl) ethylidene)-2-(4-chlorophenyl) hydrazine 2: 2-acetyl-5-bromothiophene (0.01 mol), p-chlorophenylhydrazine hydrochloride (0.01 mol) and 2-3 drops of glacial acetic dissolved in 20 ml ethanol. The reaction mixture was stirred on a magnetic stirrer at room temperature for three hours. After the completion of reaction yellow precipitate was obtained, which was filtered, washed and purified by recrystallization with ethanol. Yellow solid, melting point -148 °C, yield- 70%.

Procedure for Preparation of (Pyrazole) 3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazole-4-carbaldehyde 3: 0.03 mol of POCl_3 was added dropwise to an ice-cold solution of 1-(1-(5-bromothiophen-2-yl)ethylidene)-2-(4-chlorophenyl) hydrazine (3) (0.01 mol) in 10 ml DMF. The reaction mixture was stirred for half an hour at room temperature first and then at 70 °C for about

five hours on a magnetic stirrer. The resulting mixture was poured onto crushed ice and neutralized with a saturated sodium bicarbonate solution. The pale green precipitate obtained was purified by recrystallization with ethanol.

Yellow solid, melting point-158 °C, yield- 80%, IR (KBr cm^{-1}): 1674 cm^{-1} (C=O), 2785 cm^{-1} (CHO), 3182 cm^{-1} (H-Ar), 1531 cm^{-1} (C=N). $^1\text{H-NMR}$ (DMSO- d_6 , δ , ppm): 9.9 (s, 1H, CHO), 9.3 (s, 1H, Pyrazole), 7.9 (d, 1H, Ar-H), 7.2 (d, 1H, Ar-H), 7.8 (d, 2H, Ar-H), 7.5(d, 2H, Ar-H). Mass; (M^+) 365, ($M^+ + 2$) 367.

Procedure for Preparation of Schiff Bases 4a-4l: Equimolar quantities of 3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazole-4-carbaldehyde 3 (0.01 mol) and different aromatic amines (0.01 mol) and 2-3 drops of glacial acetic were dissolved in 20 ml ethanol. The reaction mixture was refluxed for 3 to 4 h in water bath and cooled at room temperature. Yellow precipitate was obtained which was filtered, washed and purified by recrystallization with ethanol. Physical and Spectral data of representative Schiff bases are as given below.

1-(3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazol-4-yl)-N-phenyl methanimine 4a: Yellow solid, yield- 70%, melting point- 118 °C. IR (KBr, cm^{-1}): 3104 cm^{-1} (H-Ar), 1620 cm^{-1} (HC=N), 1589 cm^{-1} (C=N), 1500 cm^{-1} (C=C). $^1\text{H-NMR}$ (DMSO- d_6 , δ , ppm): 9.2 (s, 1H, pyrazole), 8.6 (s, 1H, HC=N), 6.9-8.0 (m, 11H, Ar-H). Mass; (M^+) 442.

1-(3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazol-4-yl)-N-(4-chlorophenyl) methanimine 4b: Yellow solid, yield-85%, melting point-136 °C. IR (KBr, cm^{-1}): 3140 cm^{-1} (H-Ar), 1612 cm^{-1} (HC=N), 1535 cm^{-1} (C=N), 1500 cm^{-1} (C=C). $^1\text{H-NMR}$ (DMSO- d_6 , δ , ppm): 9.1 (s, 1H, pyrazole), 8.6 (s, 1H, HC=N), 6.6-8.0 (m, 10H, Ar-H). Mass; (M^+) 478.

1-(3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazol-4-yl)-N-(4-bromophenyl) methanimine 4c: Yellow solid, yield-50%, melting point-142 °C. IR (KBr, cm^{-1}): 3142 cm^{-1} (H-Ar), 1631 cm^{-1} (HC=N), 1539 cm^{-1} (C=N), 1500 cm^{-1} (C=C). $^1\text{H-NMR}$ (DMSO- d_6 , δ , ppm): 9.4 (s, 1H,

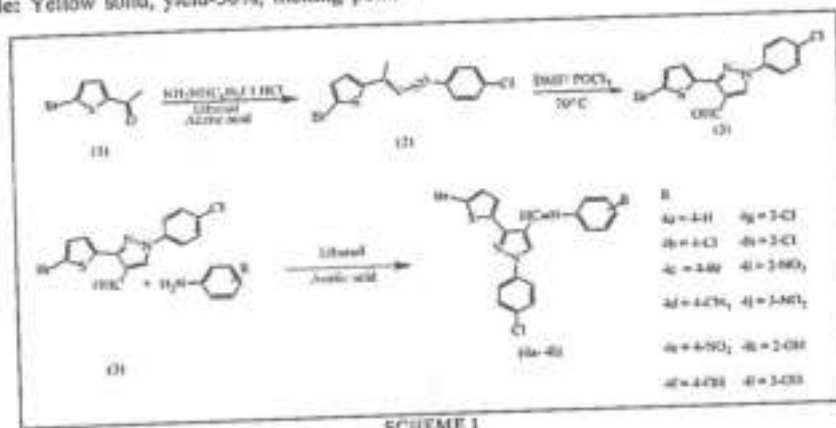
pyrazole), 8.6 (s, 1H, HC=N), 6.5-8.0 (m, 10H, Ar-H). Mass; (M^+) 478.

1-(3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazol-4-yl)-N-(p-tolyl) methanimine 3d: Yellow solid, yield-55%, melting point -120 °C. IR (KBr, cm^{-1}): 3136 cm^{-1} (H-Ar), 1624 cm^{-1} (HC=N), 1597 cm^{-1} (C=N), 1500 cm^{-1} (C=C). $^1\text{H-NMR}$ (DMSO- d_6 , δ , ppm): 9.1 (s, 1H, pyrazole), 8.6 (s, 1H, HC=N), 2.5 (s, 3H CH_3), 6.4-8.0 (m, 10H, Ar-H). Mass; (M^+) 456.

1-(3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazol-4-yl)-N-(4-nitrophenyl) methanimine 4e: Yellow solid, yield-50%, melting point-

128 °C. IR (KBr, cm^{-1}): 3128 cm^{-1} (H-Ar), 1631 cm^{-1} (HC=N), 1597 cm^{-1} (C=N), 1527 cm^{-1} (C=C). $^1\text{H-NMR}$ (DMSO- d_6 , δ , ppm): 9.4 (s, 1H, pyrazole), 8.6 (s, 1H, HC=N), 6.6-8.0 (m, 10H, Ar-H). Mass; (M^+) 489.

1-(3-(5-bromothiophen-2-yl)-1-(4-chlorophenyl)-1H-pyrazol-4-yl)-N-(4-hydroxyphenyl) methanimine 4f: Yellow solid, yield-60%, melting point-158 °C. IR (KBr, cm^{-1}): 3097 cm^{-1} (H-Ar), 1620 cm^{-1} (HC=N), 1546 cm^{-1} (C=N), 1508 cm^{-1} (C=C). $^1\text{H-NMR}$ (DMSO- d_6 , δ , ppm): 9.1 (s, 1H, pyrazole), 8.6 (s, 1H, HC=N), 6.4-8.0 (m, 10H, Ar-H). Mass; (M^+) 458.



SCHEME 1

TABLE 1: ANTIMICROBIAL ACTIVITY OF SCHIFF BASES 4a-4l

Compound	Antibacterial activity (Zone of inhibition in mm)			
	<i>Escherichia coli</i>	<i>Staphylococcus aureus</i>	<i>Pseudomonas aeruginosa</i>	<i>Klebsiella pneumoniae</i>
4a	10	-	12	12
4b	-	-	13	10
4c	-	10	14	-
4d	10	10	12	12
4e	12	-	10	10
4f	11	11	11	11
4g	-	10	10	11
4h	-	-	11	14
4i	10	10	13	12
4j	12	-	11	11
4k	-	11	12	10
4l	-	14	12	19
Streptomycin	28	27	27	

-No activity detectable under experimental condition

Antibacterial Assay: All the synthesized Schiff bases were screened for antimicrobial activity by using four organisms namely *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Klebsiella pneumoniae* using streptomycin as a standard drug. Agar well-diffusion method was followed to determine the antimicrobial activity. Bacterial cultures were grown in exponential phase

in nutrient broth at 37 °C for 8 h and adjusted to a final concentration 0.5 McFarland turbidity standard. The nutrient agar plate (thickness 4 to 5 mm) surface is inoculated by spreading a volume of the microbial inoculum over the entire agar surface. A hole with a diameter 6 to 8 mm has punched aseptically with a sterile stainless steel borer. 20 µl of the chemical agent of desired concentration was introduced into the well with the help of micropipette and allowed to diffuse at room temperature for 1 h. The plates were incubated at 37 °C for 18 h for bacterial pathogens. The diameter of the inhibition zone (mm) was measured with zone reading scale. The antibacterial activity of the synthesized Schiff bases against mentioned organisms is given in Table 1.

RESULTS AND DISCUSSION:

Chemistry: The Schiff bases have been prepared from 3- (5-bromothiophen-2-yl)-1- (4-chlorophenyl)-1H-pyrazole-4-carbaldehyde (3) according to synthetic route as mentioned in the scheme. Structures of all the compounds were established by IR, ¹H-NMR spectroscopy and Mass spectrometry data. The compound 1- (1-(5-bromothiophen-2-yl) ethylidene)-2- (4-chlorophenyl) hydrazine (2) obtained from the reaction between 2-acetyl-5-bromothiophene and p-chlorophenyl hydrazine hydrochloride. Compound (2) then subjected to Vilsmeier-Haack reaction to form 3- (5-bromothiophen-2-yl)-1- (4-chlorophenyl)-1H-pyrazole-4-carbaldehyde (3). The formation of compound (3) was confirmed by a sharp signal at 1674 cm⁻¹ for C=O stretching and two signals at 2885 cm⁻¹ and 1785 cm⁻¹ for (H—CO) in the IR spectrum, singlet signal at δ 9.9 ppm for CHO proton and also singlet signal at 9.3 for pyrazolyl proton in the ¹H-NMR spectrum.

In the mass spectrum of compound (3) the molecular ion peak was observed at m/z = 365, which corresponds to molecular weight. Schiff bases 4a-4l were formed, when compound (3) was reacted with different aromatic amines in the presence of glacial acetic acid as a catalyst.

The structure of newly synthesized Schiff bases were confirmed by the presence of strong signal at 1620-1635 cm⁻¹ in the IR spectrum, the ¹H-NMR spectra showed the sharp singlets at δ 9.1-9.4 ppm for pyrazolyl proton and δ 8.6 due to proton of

HC=N group of Schiff bases and multiples for aromatic protons are observed in the range of 6.4-8.0 ppm. The m/z values for the characterized Schiff bases obtained are in good agreement with the molecular weights.

Antimicrobial Activity: The results of antibacterial screening are shown in Table 1. All prepared Schiff bases showed poor to good activity against test organisms. Based on the zone of inhibition results, it is observed that the newly prepared Schiff bases showed better activity against *Klebsiella pneumoniae* than *Escherichia coli*, *Staphylococcus aureus*, and *Pseudomonas aeruginosa*. Schiff base 4c showed good activity against *Pseudomonas aeruginosa*, Schiff base 4l showed good activity against *Staphylococcus aureus* and all the Schiff bases 4a-4l except 4c showed good activities against *Klebsiella pneumoniae*. Schiff bases 4a-4l showed poor activity against *Escherichia coli*.

CONCLUSION: A new series of Schiff bases were prepared and the structures of newly synthesized Schiff bases were confirmed by IR spectroscopy, ¹H-NMR spectroscopy, and Mass spectrometry. These newly prepared Schiff bases showed poor to good activity against the test organisms.

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Research Journal of Biotechnology	0.222 for year 2010 Quarterly indexed in WOS of Science and SSCI	0.188 for year 2010 ISI Index 18	0.178 for year 2008	0.7 for year 2010	6.80 for year 2010	Yes	Yes
Research Journal of Chemistry and Environment	ISI Impact factor was 0.026 for year 2012	0.124 for year 2010 ISI Index 16	0.228 for year 2010	0.1 for year 2010	3.81 for year 2010	Yes	Yes
Discover chemicals	ISI impact factor was	0.14 for year 2010	0.07 for year 2010	0.4 for year 2010	-	Yes	Yes

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Synthesis and antimicrobial evaluation of pyrimidines derived from chalcones

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Abstract

In present study, 2-acetyl-5-bromothiophene (1) on condensation with various aromatic aldehydes in alcoholic KOH solution yielded corresponding chalcones (3). The chalcones were further reacted with guanidine hydrochloride in presence of base in ethanol which led to the formation of pyrimidine derivatives (4). The newly synthesized heterocycles were characterized on the basis of NMR, IR and Mass spectroscopic data. All newly synthesized compounds were evaluated for their antimicrobial activities.

Keywords: Chalcones, Pyrimidines, Antimicrobial Activity.

Introduction

The nitrogen heterocycles in general and pyrimidine in particular are found in several biologically active natural products and depict considerable therapeutic potential. Pyrimidine is one of the most prominent structures found in nucleic acid chemistry. Many classes of chemotherapeutic agents containing pyrimidine nucleus are in clinical use such as antibacterial (sulfadiazine, sulfamazine and sulfamethazine), anticancer (5-fluorouracil and thiothiopyran), antiviral (iodoxuridine, trifluridine and zidovudine), antifungal (flucytosine) and antimalarial (pyrimethamine) agents.

Pyrimidines attract great attention on account of their wide range of biological and pharmaceutical properties, such as anticancer¹⁻³, antibacterial⁴⁻¹¹, antituberculosis¹²⁻¹⁴, anti-inflammatory¹⁵⁻¹⁷, antiviral¹⁸, anti-HIV¹⁹, antihypertensive²⁰⁻²¹, anticonvulsant²² and analgesic²³.

The increase in drug-resistant bacterial strain isolates during recent years presents a therapeutic challenge to physicians selecting antimicrobial agents. Thus, development of new agents with potent antibacterial activities and fewer adverse effects is urgently desired. In the present investigation, we report the synthesis of some new aminopyrimidines and their antibacterial activity.

Material and Methods

All the chemicals were purchased from Sigma Aldrich and S.D. Fine Chemical and used further without purification. Melting points were determined in open capillary tube and are uncorrected. The progress of the reactions as well as purity of the compounds was monitored by thin layer chromatography with F-252 silica gel precoated aluminum

sheets using petroleum ether – ethyl acetate (9:1) as a developing solvent and spots were visualized with near UV and iodine. The IR spectra were recorded Shimadzu spectrophotometer using KBr pellets and expressed in cm^{-1} .

The NMR spectra were recorded on Bruker Advance (400 FT-NMR) spectrophotometer in DMSO using tetramethylsilane as an internal standard and chemical shift values are expressed in δ ppm. Mass spectra were recorded on Waters UPLC-TQC MASS Spectrometer.

Preparation of 1-(5-bromothiophen-2-yl)-3-aryl-prop-2-en-1-one (3a-f): The substituted chalcones were prepared by reacting equimolar quantity of aromatic aldehydes (0.001 mol) and 2-acetyl-5-bromothiophene in ethanol. 40% KOH was added to the reaction mixture drop wise with constant stirring at room temperature.

After the completion of reaction, the precipitate thus obtained was separated by filtration, washed with cold ethanol, dried and purified by recrystallization from ethanol.

Spectral data of some of the newly synthesized chalcones (3a-f)

1-(5-bromothiophen-2-yl)-3-(4-chlorophenyl)prop-2-en-1-one (3a): ¹H-NMR (DMSO-D₆, 400MHz, δ in ppm): 7.51 (d, 1H, Ar-H), 8.22 (d, 1H, Ar-H), 7.55 (d, 2H, Ar-H), 7.95 (d, 2H, Ar-H), 7.74 (d, 1H, H-C=C), 7.90 (d, 1H, =CH-C=O). IR (KBr, cm^{-1}): 3078 cm^{-1} (C-H Aromatic), 1674 cm^{-1} (C=O), 1589 cm^{-1} (C=C Aromatic), 1411 cm^{-1} (C=C). Mass (ESI-MS): 371 (M+44).

1-(5-bromothiophen-2-yl)-3-(4-methoxyphenyl)prop-2-en-1-one (3b): ¹H-NMR (DMSO-D₆, 400MHz, δ in ppm): 3.82 (s, 3H, CH₃), 7.03 (d, 2H, Ar-H), 7.85 (d, 2H, Ar-H), 7.70 (s, 2H, Ar-H), 7.45 (d, 1H, H-C=C), 8.16 (d, 1H, =CH-C=O). IR (KBr, cm^{-1}): 3086 cm^{-1} (C-H Aromatic), 1647 cm^{-1} (C=O), 1585 cm^{-1} (C=C Aromatic), 1415 cm^{-1} (C=C). Mass (ESI-MS): 323 (M⁺), 325 (M+2).

1-(5-bromothiophen-2-yl)-3-phenylprop-2-en-1-one (3c): ¹H-NMR (DMSO-D₆, 400MHz, δ in ppm): 7.51-7.30 (m, 7H, Ar-H), 7.54 (d, 1H, H-C=C), 7.99 (d, 1H, =CH-C=O). IR (KBr, cm^{-1}): 3090 cm^{-1} (C-H Aromatic), 1651 cm^{-1} (C=O), 1597 cm^{-1} (C=C Aromatic), 1411 cm^{-1} (C=C). Mass (ESI-MS): 334 (M+41).

1-(5-bromothiophen-2-yl)-3-(thiophen-2-yl)prop-2-en-1-one (3d): ¹H-NMR (DMSO-D₆, 400MHz, δ in ppm): 7.21 (m, 1H, Ar-H), 7.82 (d, 1H, Ar-H), 7.50 (d, 1H, Ar-H), 7.92 (d, 1H, Ar-H), 7.71 (d, 1H, Ar-H), 7.46 (d, 1H, H-

C=O), 8.13 (d, 1H, =CH—C=O). IR (KBr, cm^{-1}): 3074 cm^{-1} (C—H Aromatic), 1643 cm^{-1} (C=O), 1577 cm^{-1} (C=C Aromatic), 1415 cm^{-1} (C=C). Mass (ESI-MS): 299 (M^+), 301 ($M+2$).

1-(5-bromothiophen-2-yl)-3-(p-tolyl)prop-2-en-1-one (3e): $^1\text{H-NMR}$ (DMSO- D_6 , 400MHz, δ in ppm): 7.28 (d, 2H, Ar—H), 7.71 (d, 1H, Ar—H), 7.78 (d, 2H, Ar—H), 7.81 (d, 1H, Ar—H), 7.47 (d, 1H, H—C=C), 8.18 (d, 1H, =CH—C=O), 2.35 (s, 3H, CH_3). IR (KBr, cm^{-1}): 3012 cm^{-1} (C—H Aromatic), 1647 cm^{-1} (C=O), 1593 cm^{-1} (C=C Aromatic), 1415 cm^{-1} (C=C). Mass (ESI-MS): 307 (M^+), 309 ($M+2$).

Preparation of 4-(5-bromothiophen-2-yl)-6-arylpurimidine-2-amine (4a-f): Substituted purimidines were prepared by refluxing a mixture of substituted chalcones (0.001mol), guanidine hydrochloride (0.001mol) and alkali (KOH) in 20 ml ethanol. After the completion of reaction, the reaction mixture was poured in ice cold water. The precipitate thus obtained was filtered, washed with ethanol, dried and purified by recrystallization from ethanol.

Spectral data of some of the newly synthesized purimidines (4a-f)

4-(5-bromothiophen-2-yl)-6-(4-chlorophenyl) purimidine-2-amine (4a): $^1\text{H-NMR}$ (DMSO- D_6 , 400MHz, δ in ppm): 6.80 (s, 2H, —NH $_2$), 7.74 (s, 1H, purimidinyl proton), 7.11 (d, 2H, Ar—H), 7.63 (d, 2H, Ar—H), 7.99 (d, 1H, Ar—H), 8.23 (d, 1H, Ar—H). IR (KBr, cm^{-1}): 3414 cm^{-1} , 3356 cm^{-1} (—NH $_2$), 3182 cm^{-1} (C—H Aromatic), 1643 cm^{-1} (C=C Aromatic), 1570 cm^{-1} (C=N). Mass (ESI-MS): 366 (M^+), 368 ($M+2$).

4-(5-bromothiophen-2-yl)-6-(4-methoxyphenyl) purimidine-2-amine (4b): $^1\text{H-NMR}$ (DMSO- D_6 , 400MHz, δ in ppm): 6.70 (s, 2H, —NH $_2$), 7.64 (s, 1H, purimidinyl proton), 7.06 (d, 2H, Ar—H), 8.17 (d, 2H, Ar—H), 7.34 (d, 1H, Ar—H), 7.9 (s, 1H, Ar—H). IR (KBr, cm^{-1}): 3402 cm^{-1} , 3371 cm^{-1} (—NH $_2$), 3255 cm^{-1} (C—H Aromatic), 1651 cm^{-1} (C=C Aromatic), 1512 cm^{-1} (C=N). Mass (ESI-MS): 362 (M^+), 364 ($M+2$).

4-(5-bromothiophen-2-yl)-6-phenylpurimidine-2-amine (4c): $^1\text{H-NMR}$ (DMSO- D_6 , 400MHz, δ in ppm): 6.79 (s, 2H, —NH $_2$), 7.70 (s, 1H, purimidinyl proton), 7.0–8.19 (m, Ar—H). IR (KBr, cm^{-1}): 3321 cm^{-1} (—NH $_2$), 3198 cm^{-1} (C—H Aromatic), 1600 cm^{-1} (C=C Aromatic), 1539 cm^{-1} (C=N). Mass (ESI-MS): 332 (M^+), 334 ($M+2$).

4-(5-bromothiophen-2-yl)-6-(thiophen-2-yl) purimidine-2-amine (4d): $^1\text{H-NMR}$ (DMSO- D_6 , 400MHz, δ in ppm): 6.78 (s, 2H, —NH $_2$), 7.65 (s, 1H, purimidinyl proton), 8.04 (d, 2H, Ar—H), 7.25 (m, 2H, Ar—H), 7.75 (d, 1H, Ar—H), 7.91 (d, 1H, Ar—H). IR (KBr, cm^{-1}): 3479 cm^{-1} , 3364 cm^{-1} (—NH $_2$), 3101 cm^{-1} (C—H Aromatic), 1612 cm^{-1} (C=C Aromatic), 1570 cm^{-1} (C=N). Mass (ESI-MS): 342 ($M+4$), 344 ($M+6$).

4-(5-bromothiophen-2-yl)-6-(p-tolyl) purimidine-2-amine (4e): $^1\text{H-NMR}$ (DMSO- D_6 , 400MHz, δ in ppm): 6.74 (s, 2H, —NH $_2$), 7.67 (s, 1H, purimidinyl proton), 7.08 (d, 2H, Ar—H), 8.10 (d, 2H, Ar—H), 7.38 (d, 1H, Ar—H), 7.96 (d, 1H, Ar—H), 2.37 (s, 3H, CH_3). IR (KBr, cm^{-1}): 3479 cm^{-1} , 3313 cm^{-1} (—NH $_2$), 3198 cm^{-1} (C—H Aromatic), 1643 cm^{-1} (C=C Aromatic), 1570 cm^{-1} (C=N). Mass (ESI-MS): 346 (M^+), 348 ($M+2$).

Results and Discussion

The synthetic route for the preparation of the title compounds is accomplished in two stages. First stage involves the reaction of 2-acetyl-5-bromothiophene with different aromatic aldehydes in ethanol in the presence of alkali at room temperature afforded 1-(5-bromothiophen-2-yl)-3-aryl-prop-2-en-1-one (3a-f). The IR spectra of 3a-e showed the characteristic bands for C=O at 1647–1651 cm^{-1} and C=C at 1411–1415 cm^{-1} respectively.

The NMR spectra showed the presence of doublet doublet pattern equivalent to two protons in the range δ -7.45–8.18 ppm characteristic for HC=CH—C=O. The cyclocondensation reaction of 1-(5-bromothiophen-2-yl)-3-aryl-prop-2-en-1-one (3a-f), with guanidine hydrochloride in presence of alkali in ethanol occurred to yield 4-(5-bromothiophen-2-yl)-6-arylpurimidine-2-amine (4a-f) in the second stage.

IR spectra of 4a-e showed the characteristic bands for —NH $_2$ in the range 3313–3479 cm^{-1} . The NMR spectra showed the presence of singlet equivalent to one proton in the range δ , 7.64–7.74 ppm characteristic for purimidinyl proton while the primary —NH $_2$ protons appeared as a singlet at δ -6.70–6.80 ppm.

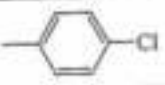
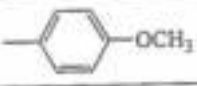
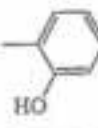
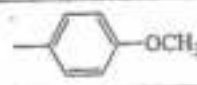
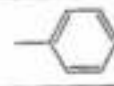
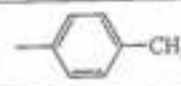
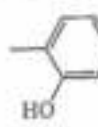
Antimicrobial Activity: Compounds (4a-f) were tested for antibacterial activity against four organisms: *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Klebsiella pneumoniae* using streptomycin as a standard drug. All the compounds exhibited good antibacterial activity but the compounds 4a, 4b, 4c and 4f exhibited higher antibacterial activity close to the standard. Observations are shown in table 2.

Conclusion

In the present study, 1-(5-bromothiophen-2-yl)-3-aryl-prop-2-en-1-one (3a-f) and 4-(5-bromothiophen-2-yl)-6-arylpurimidine-2-amine (4a-f), have been synthesized and characterized on the basis of spectral analysis.

The compounds (4a-f) were evaluated for *in vitro* antibacterial activity against four organisms: *Escherichia coli*, *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Klebsiella pneumoniae* using streptomycin as a standard drug. The compounds 4a, 4b, 4c and 4f exhibited higher antibacterial activity close to the standard.

Table 1
Physical data of newly synthesized compounds

S. N.	Entry	Ar	Molecular formula	Colour	% Yield	Melting point
1	3a		$C_{10}H_9BrClOS$	Yellow	93	139°C
2	3b		$C_{14}H_{11}BrO_2S$	Pale yellow	92	141°C
3	3c		$C_{13}H_9BrOS$	Green-yellow	84	135°C
4	3d		$C_{13}H_9BrOS_2$	Pale green	86	110°C
5	3e		$C_{14}H_{11}BrOS$	Pale yellow	85	150°C
6	3f		$C_{13}H_9BrO_2S$	Green	82	146°C
7	4a		$C_{14}H_9BrClN_3S$	Yellow	77	178°C
8	4b		$C_{18}H_{15}BrN_3OS$	Yellow	83	176°C
9	4c		$C_{14}H_9BrN_3S$	Pale yellow	73	118°C
10	4d		$C_{18}H_9BrN_3S_2$	Green-yellow	72	158°C
11	4e		$C_{19}H_{11}BrN_3S$	Yellow	71	130°C
12	4f		$C_{14}H_9BrN_3OS$	Brown	65	142°C

Scheme 1

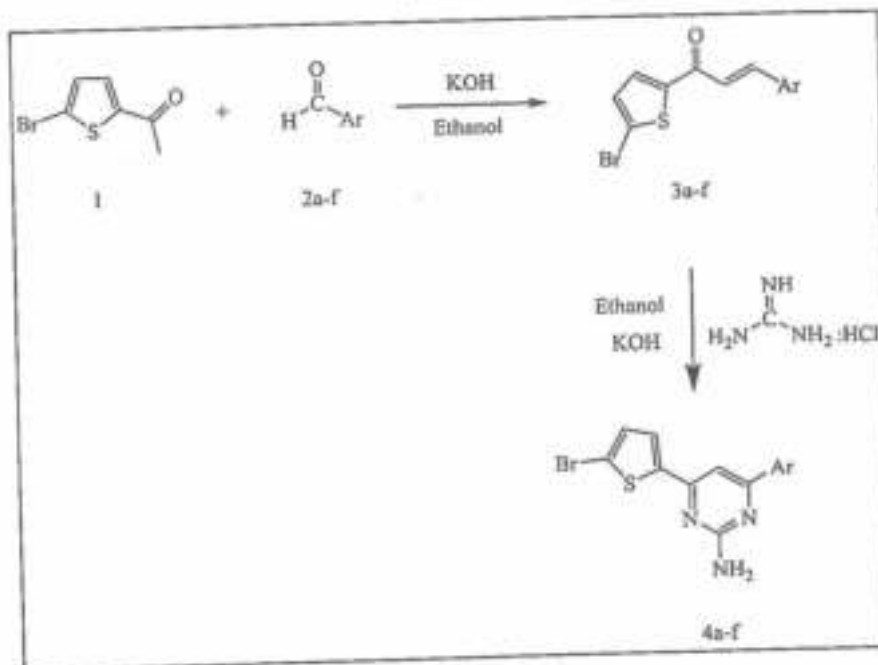


Table 2
 Antibacterial activity of synthesized compounds (4a-f)

Compound	Antibacterial activity (Zone of inhibition in mm)			
	<i>Escherichia coli</i>	<i>Staphylococcus aureus</i>	<i>Pseudomonas aeruginosa</i>	<i>Micobacteria pneumoniae</i>
4a	14	20	13	16
4b	15	19	13	14
4c	15	19	13	13
4d	17	18	15	10
4e	15	19	12	10
4f	17	20	19	15
Streptomycin	28	27	27	19

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Influence of Dy³⁺ concentration on spectroscopic behavior of Sr₃MgSi₂O₈:Dy³⁺ phosphors: Influence of Dy³⁺ concentration on spectroscopic behavior of Sr₃MgSi₂O₈:Dy³⁺ phosphors: Dr. Shweta S Sharma

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Influence of Dy³⁺ concentration on spectroscopic behaviour of Sr₃MgSi₂O₈:Dy³⁺ phosphors

Pradeep Dewangan^a, R. B. D. P. Bisen^a, Narmada Brahme^a, **Shweta Sharma^a**, Ranvik Kumar Tamiakar^b, Ishwar Prasad Sahu^a, Kanchan Upadhyay^a

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Abstract

White light emitting novel Sr₃MgSi₂O₈:Dy³⁺ were synthesized by solid state reaction (SSR) process. The structural and morphological properties were analyzed by XRD and TEM. XRD pattern confirms presence of monoclinic structure with space group P121/a1. Emission spectra of Sr₃MgSi₂O₈:Dy³⁺ phosphors were studied as a function of Dy³⁺ ion concentration. Emission spectra recorded at 350 nm excitation consist of characteristic blue and yellow emission bands centered at 480 nm, 493 nm and 572 nm, which correspond to ⁴F_{3/2} → ⁶H_{15/2} and ⁴F_{3/2} → ⁶H_{13/2} characteristic transitions of Dy³⁺ ions respectively. Emitted color of the phosphor was determined by CIE chromaticity graph. Sr₃MgSi₂O₈:Dy³⁺ phosphors are depended upon to find potential applications, for instance, WLEDs and optical feature structure.

Introduction

Rare earth doped inorganic phosphors, for example, silicate based phosphor materials have increased more consideration for their utilization in numerous reasonable applications in solid state lighting innovation and biomedical applications due to their properties. It is

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140

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Synthesis, Characterization and Gas Sensing Study of SnO₂ Thick Film Sensor towards H₂S, NH₃, LPG and CO₂

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Abstract

SnO₂ is synthesized by microwave assisted technique. Crystal structure and particle size is studied by XRD. Particle size of synthesized sample is investigated by TEM, FE-SEM and Scherrer formula. Crystalline nature is observed by SAED. FE-SEM of sample is carried out for knowing surface morphology and particle size. Energy dispersive analysis by X-ray (EDAX) is done for studying elemental composition and their mass percentage in the sample. The thick film of SnO₂ is fabricated by screen printing technique for exploring V-I characteristics and conductivity. The gas sensing response of SnO₂ thick film sensor towards H₂S, LPG, NH₃ and CO₂ is studied.

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Keywords

SnO₂ synthesis; microwave technique; characterization; gas response

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Studies on Cytopathic Effects of BmNPV infection in Three Lepidopteran Insect Cell Lines in MGM-448 Medium: Syed Obaid Qureshi

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Studies on cytopathic effects of BmNPV infection in three lepidopteran insect cell lines in MGM-448 medium

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ABSTRACT

Three insect cell lines, namely, DENV-1-Bm-1, DENV-1-Bm-12, and NIAS-MbB-92, were tested for susceptibility to *Bombus morio* nucleopolyhedrovirus (BmNPV). The cytopathic effects of infecting an infected cell were observed. BmNPV was then serially passaged in these cell lines to assess their long-term ability to support BmNPV replication. The cell lines were cultured in MGM-448 medium supplemented with 10% fetal bovine serum. Virus inoculum was prepared from B. morio larvae infected with BmNPV. The early signs of infection started appearing within 16–36 hours post inoculation (hpi). DENV-1-Bm-1 and DENV-1-Bm-12 cells showed heavy clumping, NIAS-MbB-92 cells lost their morphology and tended to form loose aggregation. Cytopathic effects such as hypertrophy of nuclei and inclusion in cell size were prominent in all three cell lines. Beginning of occlusion body (OB) formation in DENV-1-Bm-1 was first observed at about 36–42 hpi, whereas, in DENV-1-Bm-12, they were observed only after 42 hpi. NIAS-MbB-92 cells exhibited OB formation only after 48 hpi. The large mature OBs were visible in DENV-1-Bm-1 cells by 86 hpi, whereas, in DENV-1-Bm-12 cells by 72 hpi. Mature OBs were observed in NIAS-MbB-92 cells only after 120 hpi. A number of OBs per cell varied from 12 to 92 in DENV-1-Bm-1, 3 to 23 in DENV-1-Bm-12, and 3 to 14 in NIAS-MbB-92 cells. In this study, DENV-1-Bm-1 and DENV-1-Bm-12 cell lines were found to be better than NIAS-MbB-92 in terms of percentage infections. The yield of OBs per infected cell is also the highest in DENV-1-Bm-1, whereas DENV-1-Bm-12 and NIAS-MbB-92 have comparable yields of OBs per cell.

1. INTRODUCTION

Insect cell lines have served a wide range of purposes in areas, such as genetics, cell biology, agriculture, entomology, and virology. In recent years, lepidopteran cell lines have been used for the biomanufacturing of proteins of human interest, biopesticides, and vaccines [1–4]. Baculoviruses are DNA viruses that infect arthropods. Many of these baculoviruses have been shown to possess the potential to be used as biopesticides. Two properties of nucleopolyhedroviruses have made them attractive for use as biopesticides. First, they are safe for mass spraying because they do not infect vertebrates and other non-target animals [5]. The second desirable property of these baculoviruses is their high pathogenicity [6]. Advances in recombinant DNA technology have now armed researchers with the ability to engineer novel

nucleopolyhedroviruses with high insecticidal potential [7–9]. Baculoviruses are also being employed as gene vectors forming baculovirus expression vector systems (BEVS) [10]. Insect cell lines that have a high susceptibility to a baculovirus can be used to develop efficient insect cell-baculovirus expression vector system (IC-BEVS) [11]. An efficient IC-BEVS production platform can be used for foreign gene expression, production of therapeutic compounds, and vaccines. *Bombus morio* nucleopolyhedrovirus (BmNPV) has been successfully utilized in BEVS for the expression of recombinant proteins in susceptible cell lines [12,13]. BmNPV has another important economic implication as it causes a devastating disease of silkworms called grasserie. Studying the cytopathic effects and progression of virus replication in permissive cell lines is the first step toward understanding the replication cycle of BmNPV. The studies carried out on the abilities of different baculoviruses to infect heterologous cell lines have given varying results. *Heliothis virescens* nuclear polyhedrosis virus (HNPV) and BmNPV have been reported to replicate only in respective cell lines and appeared to be highly species specific,

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whereas AcNPV could infect seven cell lines from different insects [14]. However, BmNPV has been shown to replicate in cell line from *Mamestra brassicae* [15]. In the present study, three insect cell lines [16-18], namely, DZNU-Bm-1, DZNU-Bm-12, and NIAS-MaBr-92, were tested for susceptibility to BmNPV. DZNU-Bm-1 and DZNU-Bm-12 have been previously used for growth rates and karyotype studies [19]. Cytopathic effects on cultured cells were observed after successful infection with the virus. The BmNPV was then serially passaged in these cell lines to assess their long-term ability to support BmNPV replication.

2. MATERIALS AND METHODS

2.1. Cell Lines

Three cell lines such as DZNU-Bm-1, DZNU-Bm-12, and NIAS-MaBr-92 were cultured in MGM-448 medium supplemented with 10% fetal bovine serum (FBS). No antibiotics were added to the medium. The cell lines were grown in glass tissue culture flasks, incubated at $25 \pm 1^\circ\text{C}$, and passaged regularly.

2.2. Preparation of Virus Inoculum

The virus inoculum was prepared from the dissolved larvae of silkworm *B. mori*. Fresh mulberry leaves were cut into pieces of 2 cm² size. Each piece was then smeared with a 10- μl suspension of occlusion bodies (OBs) of BmNPV obtained from the Centre for Sericulture and Biological Pest Management, Amravati, R.T.M. Nagpur University, Nagpur. Each smeared piece of leaf was fed to a healthy larva of *B. mori* for inoculation. The larvae were further reared till the fifth instar stage by feeding them with fresh mulberry leaves. At the fifth instar stage, an incision was made on proleg of the inoculated larva to collect hemolymph. After centrifugation (1,000 g for 10 minutes), the supernatant was diluted with an equal volume of MGM-448 medium supplemented with 10% FBS medium and sterilized by passing through 0.45- μm pore size membrane filter. This served as BmNPV inoculum for infecting the cell lines.

2.3. Inoculation

BmNPV inoculum was taken in a sterile pipette, and 3-4 drops of it were added to each culture flask containing growing cells. The infected cultures were incubated at $25 \pm 1^\circ\text{C}$ and examined for the cytopathic effects and formation of OBs. The contents of the inoculated cultures were collected after 10-12 days post inoculation and centrifuged at 3,000 rpm for 15 minutes. The supernatant containing BmNPV-laden virus was collected in a sterile centrifuge tube. The supernatant was stored in the refrigerator at 4°C to be used as a virus inoculum for subsequent passaging of the virus. Serial passaging of virus was carried out by inoculating healthy cells with undiluted supernatant from the previous passage of the virus. Harvesting of OBs was done by resuspending the precipitate containing the infected cells in sterile distilled water, washed with 0.5% (w/v) of sodium lauryl sulfate, and rinsed thrice in distilled water.

2.4. Phase-Contrast Microscopy

The cultures were examined regularly for the presence of infected cells with a Magnus INVI-inverted phase-contrast microscope. The criterion of BmNPV infection was the presence of polyhedral inclusion bodies in a cell. In cultures that showed heavy clumping of cells, a small number of cells were removed from the inoculated cultures and transferred to microscopic glass slides. The cells were then spread out by placing a cover glass over the slide. From each flask, about 200-300 cells were used for making differential counts of healthy and infected cells. The percentage infection and number of OBs per cell were determined for each cell line.

3. RESULTS AND DISCUSSION

The observation of inoculated cultures under phase-contrast microscope indicated that the early signs of infection started appearing within 16-36 hours post inoculation (hr pi). By 24 hr pi, all infected cultures of DZNU-Bm-1 and DZNU-Bm-12 cells showed heavy clumping. Cells from NIAS-MaBr-92 did not show heavy clumping but tended to form loose aggregations. The infected cells of NIAS-MaBr-92 lost their motility. All three cell lines exhibited loose adherence to the bottom of the culture flask. The cytopathic effects such as hypertrophy of nuclei and increase in cell size were prominent in all three cell lines.

The beginning of OB formation in DZNU-Bm-1 was first evidenced from the appearance of refractive bodies in the nuclei of infected cells at about 36-42 hr pi, whereas, in DZNU-Bm-12, they were observed only after 42 hr pi. The nuclei of NIAS-MaBr-92 cells exhibited OB formation only after 84 hr pi. The large mature OBs were visible in the nuclei of aggregated cells of DZNU-Bm-1 by 60 hr pi, whereas in DZNU-Bm-12 cells by 72 hr pi. Mature OBs were observed in NIAS-MaBr-92 cells only after 120 hr pi. OB formation by 40-48 hr pi in *B. mori* cells has been reported in the previous studies [20]. Small OBs have been observed in MB-19 cell line by 52 hr pi, whereas mature OBs have been reported by 76 hr pi [15]. Some of the cell aggregates were taken out from the infected cultures and examined under a microscope for the determination of the percentage of infected cells. The percentage infection in DZNU-Bm-1 cells remained between 89% and 93% (Table 1) for five serial passages of BmNPV. DZNU-Bm-12 cell line showed a consistent percentage infection that ranged from 82% to 88% (Table 2). These results agree with other studies that have reported no decrease in the yield of OBs even after 10 passages [21,22]. During passage I of virus, about 62% of NIAS-MaBr-92 cells were infected. The percentage infection gradually increased to 88% by passage III. It declined thereafter to a level of about 77% by passage V (Table 3). This agrees with the other reports of a decrease in percentage infection and yield of OBs following serial passaging of NPV in permissive cell lines [23-25]. A number of OBs per cell varied from 12 to 82 in DZNU-Bm-1, whereas, in DZNU-Bm-12, it ranged from 3 to 29. The number of OBs per cell depended on the cell size. NIAS-MaBr-92 exhibited about 3 to 19 OBs per infected cell. Infected cells started to lyse by 72 hr pi in DZNU-Bm-1 cells. This is consistent with the previous observations on DZNU-Bm-1 cells [26]. DZNU-Bm-12 cells did not

Table 1. Five serial passages of BmNPV in DZNU-Bm-1 cells.

Serial passage	Days p.i.	Cells per ml ($\times 10^6$)	Percentage infection ^a	Infected cells per ml ($\times 10^6$) ^b	OBs per infected cell ^c
I	4	5.12	88.88	4.55	20 $\pm$ 2
II	4	4.89	92.43	4.52	24 $\pm$ 2
III	4	4.62	96.21	4.44	28 $\pm$ 3
IV	4	5.32	91.52	5.11	30 $\pm$ 3
V	4	5.68	93.21	5.28	31 $\pm$ 3

Table 2. Five serial passages of BmNPV in DZNU-Bm-12 cells.

Serial passage	Days p.i.	Cells per ml ($\times 10^6$)	Percentage infection ^a	Infected cells per ml ($\times 10^6$) ^b	OBs per infected cell ^c
I	5	4.43	82.84	3.68	19 $\pm$ 1
II	6	4.96	86.31	4.28	18 $\pm$ 2
III	6	5.08	87.36	4.41	17 $\pm$ 2
IV	6	5.15	88.88	4.58	12 $\pm$ 2
V	6	5.10	85.37	4.31	11 $\pm$ 2

Table 3. Five serial passages of BmNPV in NIAS-Mafr-92 cells.

Serial passage	Days p.i.	Cells per ml ($\times 10^6$)	Percentage infection ^a	Infected cells per ml ($\times 10^6$) ^b	OBs per infected cell ^c
I	9	5.52	42.21	2.33	06 $\pm$ 2
II	7	5.28	78.87	4.13	08 $\pm$ 2
III	7	5.24	88.84	4.63	13 $\pm$ 2
IV	8	5.86	87.16	5.11	14 $\pm$ 2
V	8	5.34	77.85	4.15	11 $\pm$ 2

^aThe presence of OBs in a cell was the criterion of its infection with BmNPV.^bCalculated by multiplying the cell number by percentage infection.^cMean $\pm$ standard error (SE).

show much cell lysis even after 96 hr p.i. The lysis of NIAS-Mafr-92 cells started at 172 hr p.i. In the present study, DZNU-Bm-1 showed an average of 21 OBs/cell, whereas DZNU-Bm-12 gave an average yield of about 10 OBs/cell over five passages. All three cell lines have a higher susceptibility to BmNPV than BmN cell line that has been reported to give 64%-73% of infection [27].

4. CONCLUSION

The results point that DZNU-Bm-1 and DZNU-Bm-12 cell lines are better than NIAS-Mafr-92 in terms of percentage infection in MCM-448 culture medium. The yield of OBs per infected cell is the highest in DZNU-Bm-1, whereas DZNU-Bm-12 and NIAS-Mafr-92 have comparable yields of OBs per cell. These cell lines should be adapted to low-cost serum-free media and checked for percentage infection and OB yield. Furthermore, the cell lines should also be screened for susceptibility to other baculoviruses such as HaNPV to ascertain their efficiency for the large-scale production of such biopesticides.

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CONFLICT OF INTEREST

The authors declare that they do not have any conflicts of interest.

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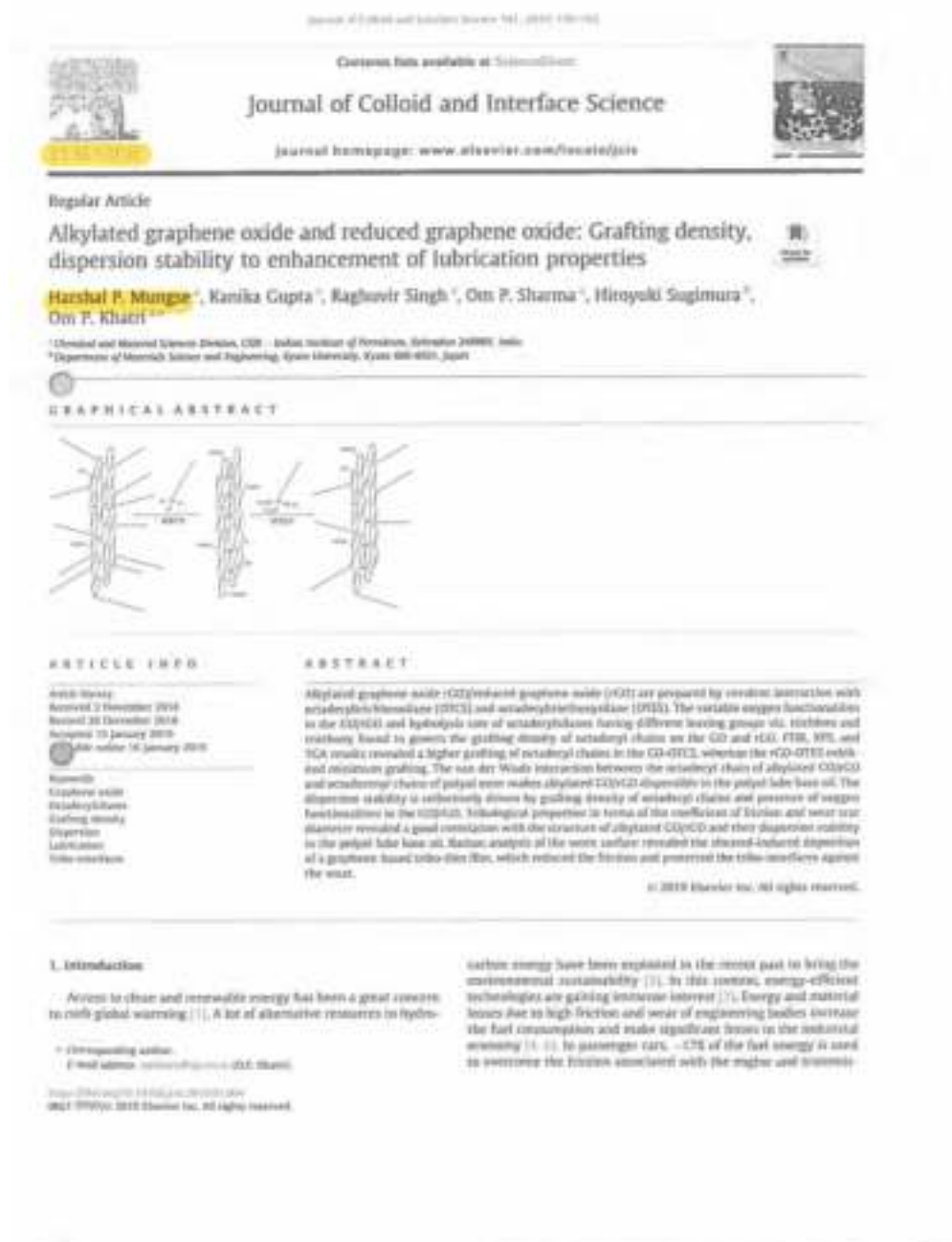
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Alkylated graphene oxide and reduced graphene oxide: Grafting density, dispersion stability to enhancement of lubrication properties: Dr. Harshal P. Mungse



NEONATAL SEPSIS DUE TO COAGULASE-NEGATIVE (CONS) STAPHYLOCOCCI: FREQUENCY, ANTIBIOTIC SUSCEPTIBILITY AND MOLECULAR CHARACTERIZATION BY 16S RNA ANALYSIS: G. N. Budhlani



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NEONATAL SEPSIS DUE TO COAGULASE-NEGATIVE (CONS) STAPHYLOCOCCI: FREQUENCY, ANTIBIOTIC SUSCEPTIBILITY AND MOLECULAR CHARACTERIZATION BY 16S RNA ANALYSIS

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ABSTRACT

Sepsis is a significant cause of morbidity and mortality in neonates. Hence, the present study was undertaken to identify the frequent organisms and to detect antibiotic sensitivity pattern against the isolated organisms in respect to better treatment. In present investigation, Out of 1800 neonates who admitted to preterm and intensive care unit (ICU) from different hospitals in Akola city, 104 (5.7%) neonates were blood culture positive. *Epidermidis* (CONS) predominate isolates among the gram positive organisms showed high rate of antibiotic sensitivity to Imipenem, Meropenem. In present investigation, antibiotic resistance of all isolates of *S. epidermidis* (CONS) was observed against Ampicillin and Penicillin. This study of *S. epidermidis* (CONS) causing neonatal sepsis and their sensitivity pattern is useful as this guidelines can be prepared for empirical antibiotic therapy.

KEYWORDS: Neonatal Sepsis, *Staphylococcus* (CONS), Antibiotic sensitivity, 16S rRNA analysis.

INTRODUCTION

Neonatal sepsis is a systemic inflammatory response caused by bacterial production of toxins after gaining entry into the bloodstream and multiplying^[1]. It is classified into the early-onset neonatal sepsis (≤ 72 hrs) due to bacteria acquired before or during delivery, and late onset neonatal sepsis (> 72 hrs-28 days) involving pathogens acquired after delivery.^[1,2,3] Generally, it is a life-threatening case. In pre-term babies, this can cross the blood-brain barrier since the baby is not fully developed, which might result in increased morbidity and mortality globally.^[3,10] This demands immediate empirical antibiotic treatment after collection of blood culture samples.^[11]

Coagulase-negative staphylococci (CONS) are the major pathogens involved in LONS (late-onset neonatal sepsis), particularly in infants born at a lower gestational age. According to more recent data from the National Institutes^[12] CONS are common inhabitants of the skin and mucous membranes; although a small proportion of neonates acquire CONS by vertical transmission, acquisition primarily occurs horizontally.^[10,13] Consequently, infants admitted to a hospital obtain most of their microorganisms from the hospital environment, their parents, and staff.^[11]

Neonatal septicemia is one of the leading causes of neonatal mortality and morbidity worldwide. Hence, the present study was undertaken to isolate the bacteria,

Coagulase-negative staphylococci causing neonatal sepsis and determine their antibiotic susceptibility pattern of Coagulase-negative staphylococci (CONS) isolates from blood cultures of neonates at a neonatal care unit in Akola city. Antibiotic resistance in skin-residing strains has been found to be low at birth but to increase rapidly during the first week of hospitalization.^[17]

MATERIALS AND METHODS

The present study is based on a prospective analysis of 104 neonates who admitted to preterm unit and intensive care unit (ICU) from Jan 2014 to Jan 2017 in the different hospitals of Akola city. All Neonates admitted during the period of study with one or more symptoms/sign suggestive of sepsis with predisposing factors, risk factors were included into study. Babies who had received antibiotics prior to presentation as well as those whose mothers had received antibiotics within one week prior to delivery were excluded from the study.

BLOOD SAMPLES COLLECTION AND PROCESSING

Using aseptic conditions, 2 ml. blood was drawn and inoculated into brain heart infusion broth and incubated at 37°C. Subcultures were made on nutrient agar, blood agar, and MacConkey's agar. The inoculated plates were incubated under aerobic conditions for 24 hr. For confirmation again subcultures were made on selective

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283

and specific media. Isolates were identified by std. Microbiological techniques.

Simultaneously, by using Kirby-Bauer disc diffusion method (1966) according to Clinical and Laboratory Standards Institute guidelines (CLSI).^[12] Antibiotic susceptibility patterns of the coagulase negative *Staphylococcus* (CONS) isolates were determined against Chloramphenicol, Ampicillin, Cefotaxime, Penicillin, Cefazidime, Ceftriaxime, Ciprofloxacin, Erythromycin, Carbapenem, Norfloxacin, Imipenem, Gentamicin, Meropenem, Nalidixic acid, Tetracycline, Amoxyclov, Vancomycin, Amikacin, Furazolidone, Azithromycin. For molecular level confirmation, multidrug resistant strains were sent for characterization by 16S rRNA analysis in Yaazh Xenomics DNA sequencing service, Madurai (Chennai Branch), Tamil Nadu, (India).

RESULTS AND DISCUSSION

The present study was conducted in nine different hospitals of Akola city of Maharashtra in which neonates admitted in NICU with signs and symptoms of sepsis were enrolled for study. Out of 1000 blood samples collected from different hospitals, 736 (73.6%) were found to be culture positive. Among gram positive isolates, 207 (76.2%) isolates were found to cause early onset sepsis (EOS) whereas only 65 isolates were associated with late onset sepsis (LOS). In present investigation, total four different types of gram positive organisms were isolated and identified. Details of these

isolates are provided in Table 1 and Graph 1, Graph 2. Frequency of *Staphylococcus epidermidis* (CONS) was 104 (14.1%) (Table 2).

High rates of (CONS) infections were reported in the Middle East, Southeast Asia, and Latin America.^[14] *Staphylococcus epidermidis* (CONS) has been identified as the causative organism for EOS as proved in other studies.^[5,17,18] In present investigation, among gram positive isolates the most predominant isolated organism was *Staphylococcus epidermidis* (CONS) 104 (14.1%) which is comparable to the findings of workers.^[5,14] The increasing prevalence of (CONS) infections is attributable to their increasing antibiotic resistance as reported by earlier researchers.^[13,41]

In view of the high morbidity and mortality associated with neonatal sepsis, the culture report cannot be awaited to administer antibiotics. Hence, area-based knowledge of the bacteriological spectrum and their antibiotic sensitivity pattern is essential in formulate an empirical therapy.^[10] *S. epidermidis* (CONS) prevalent isolates amongst the gram positive organisms showed high rate of antibiotic sensitivity to Imipenem, Meropenem as reported by investigators.^[10] In the present study, high rate of antibiotic resistance of *S. epidermidis* (CONS) was observed to commonly used antibiotics, the majority of which were MR as reported by researchers.^[13,41]

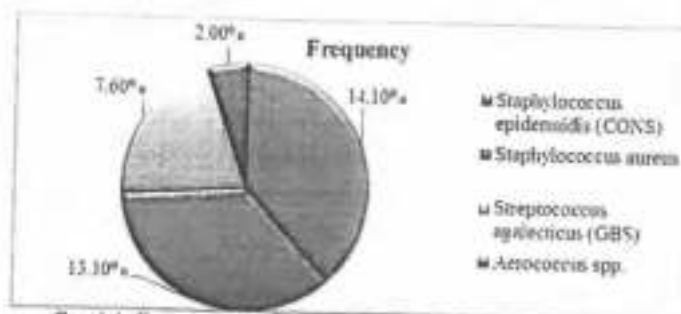
Table 1: Bacteriological Profile and Percent Distribution of Gram Positive Isolates Causing EOS and LOS.

Gm +ve Organisms	Early onset Sepsis (EOS)		Late onset Sepsis (LOS)	
	N	%	N	%
<i>Staphylococcus epidermidis</i> (CONS)	49	23.67%	55	84.61%
<i>Staphylococcus aureus</i>	77	37.19%	20	30.76%
<i>Streptococcus agalactiae</i> (GBS)	47	22.70%	9	13.84%
<i>Aeracoccus</i> spp.	9	4.34%	8	9.23%
Total	207	76.2%	65	23.8%

Table 2: Frequency Distribution of *Staphylococcus epidermidis* (CONS).

Gm +ve Organisms	Frequency (n)	Percent
<i>Staphylococcus epidermidis</i> (CONS)	104	14.1%

In present investigation, high resistance of all isolates of *S. epidermidis* (CONS) was observed against Ampicillin as observed in the previous study^[13] and Penicillin comparable to results reported in the study.^[13,41]



Graph 1: Frequency Distribution of Gram Positive Bacterial Isolates.

In addition, it was observed that all the isolates of *S. epidermidis* (CONS) were uniformly sensitive to Amikacin and Vancomycin where as high rate of resistance was found against Nalidixic acid, Norfloxacin, Amoxicillin, Cefazidime, Tetracycline and Azithromycin, Ciprofloxacin, Erythromycin, Cefotaxime, Fusidic acid and Ceftiofur (Table 3, Graph 03 and Photoplate 2). The increasing trend in the prevalence of MR-(CONS) has also been reported in other studies.^(14, 15) This is because of the indiscriminate use of antibiotics (Table 3, Graph 03). Gram positive isolates as well as the Gram negative isolates were highly sensitive to Imipenem (97.55%) and Meropenem (92.00%).

The primary identification of obtained isolates of *Staphylococcus epidermidis* (CONS) obtained was carried out based on cultural and biochemical characteristics (Photoplate 1). Alignment Search Tool (BLAST) data base of National Center for Biotechnology (NCBI)⁽¹⁶⁾ Information was used to compare the sequence of 16S rRNA of the multidrug resistant strains

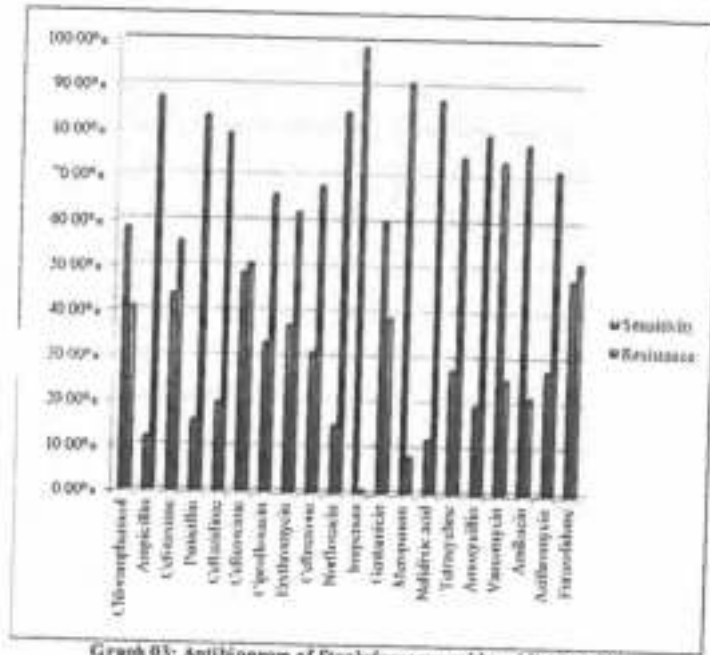
with known 16S rRNA sequences of bacteria, with the help of Yaazh Xenomics, DNA sequencing service, Madurai (Chennai Branch), Tamil Nadu. The revealed sequence accession number is NC 004461.1, NCBI, provided us taxonomy report with 74% similarity in BLAST analysis (Fig. 01, 02 and Table 04).

CONCLUSION

Staphylococcus epidermidis (CONS) predominant organism of neonatal septicemia was the leading causative agent in late onset sepsis (LOS). It was reported as hospital acquired infection and found to be almost resistant to commonly used antibiotics, Ampicillin and Penicillin. High rate of sensitivity was observed against Imipenem, Meropenem and Amikacin. This Resistance pattern of microorganisms responsible for neonatal infections is helpful to design a specific empirical antibiotic regimen. Molecular characterization by 16S rRNA analysis confirmed that neonatal septicemia is commonly caused by *Staphylococcus epidermidis* (CONS).

Table 3: Antibigram of *Staphylococcus epidermidis* (CONS).

Antibiotics	<i>S. epidermidis</i> (CONS) n=104			
	Sensitivity	Percentage	Resistance	Percentage
Chloramphenicol	61	58.7%	43	41.3%
Ampicillin	13	12.5%	91	87.5%
Cefazidime	46	44.2%	58	55.8%
Penicillin	17	16.3%	87	83.6%
Cefazidime	21	20.2%	83	79.8%
Cefuroxime	51	49.0%	53	51.0%
Ciprofloxacin	35	33.7%	69	66.3%
Erythromycin	39	37.5%	65	62.5%
Ceftriaxone	33	31.7%	71	68.3%
Norfloxacin	16	15.4%	88	84.6%
Imipenem	103	99.0%	1	1.0%
Meropenem	63	60.6%	41	39.4%
Nalidixic acid	13	12.5%	91	87.5%
Tetracycline	39	37.5%	65	62.5%
Amoxicillin	21	20.2%	83	79.8%
Vancomycin	77	74.0%	27	26.0%
Amikacin	81	77.9%	23	22.1%
Azithromycin	29	27.9%	75	72.1%
Fusidic acid	50	48.1%	54	51.9%



Graph 03: Antibiogram of *Staphylococcus epidermidis* (CONS).

Table 04: Sequences producing significant alignments of *Staphylococcus epidermidis* (CONS) isolate.

Accession No.	Description	Total Score	Query Coverage	E value	Max ident.
NC_004461.1	<i>Staphylococcus epidermidis</i> ATCC 12228 chromosome, complete Genome	1993	82%	2e-109	74%

>15

CTATGGGTGGGGCGTGCTATAATGCAGTCGAGCGGACAGATGGGGCTGCTCCCTTTTGTGTTCGGGCA
 CAGGGGAGAGACACGTGGTAACCCCTCTGTATGACTGTGGTAICTCTTGGAAACCGAGCTAATACCGG
 ATATAACCTTGAACCCGTGGGTCTGGGTGAAAAAGGGTTTTCCTGGCACTTCTAATGGGCCCCCGC
 CCATTACTTATGGGAGGGAAACCGGTTTCCAGGGGACCATTCTGAACCCAACCTGGAAGGGGGTGGG
 GGGGGTGGGACTGAAAACCGGGGCAATCCCTCCGGGGGGAACCATAGGGAATTTCCCAATGGGG
 GAAAAGCTGACGGAAACACCCCGGGGTGATGAAGGTTTTCGAATCTAAAAATTGTGTTAGGGA
 AAAACAATGGTGGAACTGCTGACCTTTCGGGACCCCTACCCAAAGCCCGGTAAATACCTTG
 CCACAGCCCGGTAAATACCTTAGTGGGACGGGTTGTCGGAATTTTGGCGTAAAGGCCCGCAGGGCGGT
 TTTAGCTGAATGTGAAGGCCCGCTCCACCGGAGGGCCATGGAAATGGGAACCTGAATGAAAAAG
 AAGTGGAAATCCAGGTGGGTGAATGCTAGAGTTTGGAGGACCCAGTGGGAGGCGCTCTCCGGTCTGT
 AACTGACGGTGTATAGCGAAGCTGGGAATGACAGATAGATACCTTGGTAGTCCACGGCGTAACGATGAT
 GCTAGTGAAGGGTCCGGCTAATGCTGACGTAACCATAGCACTCCCTGGGATAGGATCGCAGCTGAC
 TCAGATGACGGATCGCAGCGTGACATGGTATCGAGCAGGAGACTACAGCTGAATCTGACGCTAGATAG
 CTCTGGACAGCATGGTCTGTTCAGTGTGATGATCCAACGGACGTACATGCCATTGTACTAGTATCGT
 ACGAGAGTGTAGCATCA

Figure 01: 16 S rRNA Sequence of *Staphylococcus epidermidis* (CONS) isolate.

Budhram

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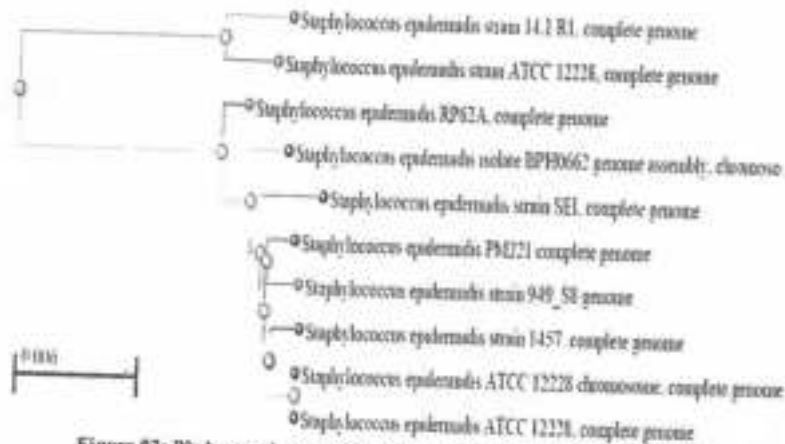


Figure 62: Phylogenetic tree of *Staphylococcus epidermidis* (CONS) isolate.

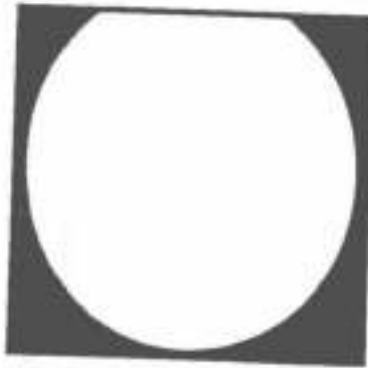


Photo plate 1: Microscopic image of (CONS).

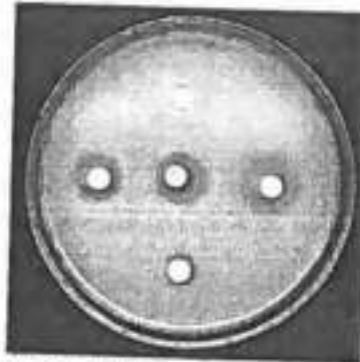


Photo plate 2: *Staphylococcus epidermidis* showing susceptibility to C, CIP, E and MRP.

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Synthesis and evaluation of a novel series of 6-bromo-1-cyclopentyl-1H-indazole-4-carboxylic acid-substituted amide derivatives as anticancer, antiangiogenic and antioxidant agents: Vilas A. Kambale

Medicinal Chemistry Research
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ORIGINAL RESEARCH

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Synthesis and evaluation of a novel series of 6-bromo-1-cyclopentyl-1H-indazole-4-carboxylic acid-substituted amide derivatives as anticancer, antiangiogenic, and antioxidant agents

Ajay S. Sawant¹ · Sonali S. Kambale² · Parthasar M. Pital³ · Rutan J. Meshram⁴ · Sanjay S. Sawant⁵ · Vilas A. Kambale⁶ · Vinod T. Kambale³ · Rajesh H. Gacche⁶

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Abstract
A series of novel indazole derivatives has been synthesized and evaluated for anticancer, antiangiogenic, and antioxidant activities. The capability of the synthesized compounds 11a–e to hinder the viability of three human cancer cells lines, HEP3BPN 11 (liver), MDA 433 (breast), and HL 60 (leukemia), were assessed by using the 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide (MTT) reduction assay. Among the compounds 11a–e screened, 11c and 11d showed the higher inhibitory activity on the viability of HEP3BPN 11 (liver), when compared with the standard metformin. These compounds were further tested to evaluate their potential to inhibit the proangiogenic cytokines associated with tumor development. Compound 11c was found to be a potent antiangiogenic agent against TNF α , VEGF, and bFGF, whereas 11d showed potent antiangiogenic activity against TNF α , VEGF, bFGF, and heparin inhibition. All the compounds 11a–e were screened for their antioxidant activities using 2,2-diphenyl-1-picrylhydrazyl (DPPH), hydroxyl (OH), and ascorbic acid (SOD) scavenging activity. Compounds 11c, 11d, 11e, and 11f have shown significant OH radical scavenging activities, also compounds 11c, 11d, and 11e were found to have a DPPH radical scavenging activity and compounds 11c and 11d exhibited better SOD scavenging activity when compared with the reference compound ascorbic acid. In silico molecular docking analysis revealed important structural insights behind observed anti-TNF α effect by potent indazole compounds.

Keywords Antiangiogenic · Anticancer · Antioxidant · Indazole derivatives

Introduction

Cancer is a life-threatening disease and it is one of the major causes of death in many countries. Cancer has become a public health burden around the world (Wang et al. 2012). It is class of diseases in which a group of cells display uncontrolled growth, invasion, and sometimes metastasis (Wetters and Kays 2002; Chabour and Roberts 2005). Current cancer chemotherapy comprises numerous natural and synthetic drugs while several novel formulations are undergoing clinical trials (Wang et al. 2017). Still, current drugs are fails to kill the cancer cells selectively, and also evoke various side effects. Strategies to block cell division by affecting the mitotic spindle is one of the major (Joshi

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IOT Based Flammable Gas Monitoring System Using ESP32 for Underground Environment: Suryakant Anandarao Rodge

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IOT Based Flammable Gas Monitoring System Using ESP32 for Underground Environment

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Abstract : Our environment is prone to various hazards and pollution. Lots of waste is usually dumped each day or a week without any proper waste management. This also affects the underground environment. The sewage and manholes nearby our areas are also dumped with wastes and garbage. Various kinds of harmful flammable or combustible gases may get created inside the sewage or manhole. The Municipal Authority in charge of the affected areas does not have the prior knowledge about the underground environment. The flammable gases concentration may lead to explosion and other life threatening issues. The proposed system uses ESP32s NodeMCU controller with MQ-5 gas sensor module. The sensor will continuously monitor the concentration of flammable gas in the underground environment. The live data will be viewed by the authority through the IoT platform. This will help to tackle the dangerous situation well before its occurrence or well before the explosion.



Keywords: ESP32s(NodeMCU),MQ-5,IoT,Gas Monitoring.

I. INTRODUCTION

The pollution is increasing to a greater extent from last decade. The sewage and manholes nearby our areas are also dumped with wastes and garbage. Various kinds of harmful flammable or combustible gases may get created inside the sewage or manhole. Sewage gases can be very dangerous for living creatures.

Sewer gas is typically restricted from entering buildings through plumbing traps that create a water seal at potential points of entry. In addition, plumbing vents allow sewer gases to be exhausted outdoors. Infrequently used plumbing fixtures may allow sewer gas to enter a home due to evaporation of water in the trap, especially in warm weather. The result is the most common means of sewer gas entering buildings and can be solved easily by using the fixtures regularly or adding water to their drains. One of the most common traps to dry out are floor drains such as those typically placed near home furnaces, water heaters and rooms with underfloor heating. Infrequently used utility sinks, tubs, showers, and restrooms also are common culprits. Trap primers are available that automatically add water to remote or little used traps such as these. Blocked plumbing vents, typically at the roof, also can cause water seals to fail via siphoning of the water.



Exposure to sewer gas also can happen if the gas seeps in via a leaking plumbing drain or vent pipe, or even through cracks in a building's foundation. Sewer gas is typically denser than atmospheric gases and may accumulate in basements. Individuals who work in sanitation industries or on farms might be exposed on the job if they clean or maintain municipal sewers, manure storage tanks, or septic tanks.

In buildings with HVAC air handlers that admit outside air for ventilation, plumbing vents placed too closely to air intakes or windows can be a source of sewer gas odors. In some cases airflow around buildings and wind effects may contribute to sewer gas odor problems even with appropriately separated vents and air intakes. Increasing vent heights, adding vent pipe filters, or providing powered dilution and exhaust can help reduce occurrences.

II. LITERATURE REVIEW

With the aim to develop a smart IoT based monitoring system, various authors proposed their works. Some of the literature surveys are as follows.

Yash Nandke et al [1] proposed a system for monitoring and managing underground drainage system with different approaches. It also gives a description of water pipe system and detection method to detect leakage defects in sewer pipelines. Also, some part of condition rating model for underground infrastructure Sustainable Water Main and Intelligent system for underground pipeline assessment, rehabilitation and management were explained. ARM7 was used and the system was not that cost effective.

Komal Pokalekar et. al. [2] proposed a IOT based garbage monitoring System. The dustbins are interfaced with Arduino base system having ultrasonic sensor along with central system showing the current status of garbage on display and web browser HTML page with Wi-Fi module. The system uses Arduino UNO which requires additional circuitry for IOT applications. Naren Vasantakumar et.al [3] proposed an intelligent gas leakage detection system with IOT using ESP8266 module. The main objective of the project was to build a Gas leakage detector using LPG gas sensor and also connect it with IoT using ESP module for safety and security. Arduino is used as the main controller. The final output of the project is used to detect leakage if gas leaks from cylinders and also notify the user by connecting via IoT software.

5. Anandhakrishnan et.al [4] proposed leak detection techniques to allow the possibility of locating the leak. The main aim was to propose the design and construction of an SMS based Gas Leakage Alert System. Gas sensor were used to detect gas leakages in a kitchen. With the help of an infrared sensor the issue of gas wastage is also monitored. An alarm goes off whenever the sensor doesn't detect any vessel over the burner beyond a particular time period.

III. BLOCK DIAGRAM OF PROPOSED SYSTEM

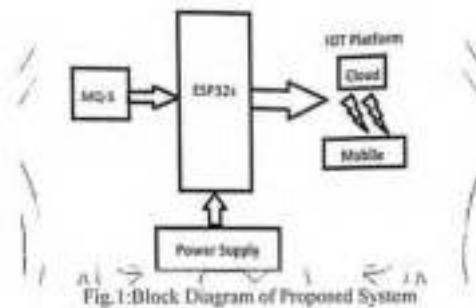


Fig.1:Block Diagram of Proposed System

The Block Diagram Consist of:

- 1)ESP32s (Node MCU)
- 2)MQ-5(Gas Leakage Detector Sensor)
- 3)Power Supply
- 4)IOT Platform/Cloud

The ESP32s is the main block of the system.It receives a constant power from the power supply.MQ-5 is the gas leakage detector sensor connected to the ESP32s.The continuous monitoring results of the sensor will be viewed on a mobile through an IOT platform or a cloud.

IV. SYSTEM PROTOTYPE AND WORKING



Fig.2: Open System



Fig.3: Actual Prototype

Working:

Suppose our system is implanted in underground environment like sewage, manhole,etc of a particular area.The system consist of ESP32 as the main hardware along with sensor MQ-5.The system will get connected to the nearby wifi provider whose password is already fed in the program.The system continuously monitors the concentration of gases in the underground environment.MQ-5 generally senses flammable gases and combustible gases. The higher values or concentration observed will

indicate the danger. These values/concentration can be continuously monitored and displayed on a mobile app called as "Bijak" through IOT platform. The Concerned authority who views the data can take necessary action for safety and precaution.

V. SYSTEM TESTING AND RESULTS



Fig.4: System Testing for LPG Leakage

The Images above shows the system prototype and testing. The mobile charger adapter is used for power supply. LPG lighter and Burning Candle in closed glass was used for detecting gases like LPG and CO under normal environment. The practice or actual implementation can vary if needed according to conditions.



Fig.5: Result in Normal Condition



Fig.6: Result in Presence of CO



Fig.7: Result in presence of LPG Gas

VI. FUTURE SCOPE

Lots of improvement can be made in the future on the developed system. With the increase in urbanization and need of healthy environment, the scope of improvement in IOT based systems in our country is immense. The robustness and durability of our system can be improved by using advanced polymers and resins. The lifespan of battery could be extended. More gas sensor

can be connected to the system so that more gases can be continuously monitored. The maximum values of particular gases can be fed in the system. The alarming facility can also be added with the slurry level detection too.

Different HTML pages or apps can also be developed for viewing the data of the sensor. The scope of a universal module could be increased from urban sector to industrial sector.

VII. CONCLUSION

The system can continuously monitor CO and other flammable gases in the underground environment. The real time data results can be continuously viewed on Blynk application. The system is cost effective.

The concerned authority can view the details of the particular gas and take necessary action on it. This will provide safety indication well before to the people living nearby the affected areas and also to the manhole cleaner.

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2020-2021

Surface Chemistry of Graphene and Graphene Oxide: A Versatile Route for Their Dispersion and Tribological Applications : Ajay Chouhan, Harshal P. Mungse, Om P Khatri



0. Summary and outlook	21
References	24

1. Introduction

Graphene, the atomic-thick two-dimensional honeycomb lattice structure of carbon material, has attracted immense interest due to remarkable mechanical, electrical, thermal, and optical properties [1,2]. The large surface area ($2630 \text{ m}^2 \text{ g}^{-1}$), excellent mechanical strength (Young's modulus: 1 TPa; fracture strength: 130 GPa), and superior thermal conductivity ($5000 \text{ W m}^{-1} \text{ K}^{-1}$) make graphene an ideal material for lubricant applications [3–5]. The graphene as a solid thin film for micro-devices, additive to various liquid lubricants, and filler to composite materials have shown remarkable improvement in friction and wear characteristics [6–10]. Graphene, as a thinnest solid lubricant film, effectively reduced the adhesion and friction forces when coated on various substrates [11]. The weak van der Waals interaction between the atomic-thick lamellae of graphene provides low-resistance to shear and lowers the friction. Therefore, a single sheet of graphene shows higher friction than multilayer-graphene. Lee et al. have explicitly revealed that friction monotonically increased as the number of layers decreased [6]. The significantly high mechanical strength of graphene protects the contact interfaces under the tribo-stress and subsidizes the wear. Recently, Sumant et al. demonstrated that a single layer of graphene on steel substrate could last for 6400 sliding cycles under the contact stress of $\sim 0.5 \text{ GPa}$ and provides excellent wear-resistance [12]. Graphene is impermeable to liquids and gases viz. water and oxygen, and it reduces the corrosive and oxidative events of rubbing surfaces [13]. These findings revealed that graphene could be an excellent candidate for liquid lubricants to reduce the friction and wear of interacting surfaces [14,15]. However, the long-term dispersion stability of graphene in lubricating oils has been a major challenge for the lubricant industries. The graphene shows incompatibility with most of solvents/lubricants and tends to agglomerate driven by high cohesive interaction.

Use of environment-friendly and energy-efficient technologies is gaining immense interest to curb environmental pollution and channelize the energy resources in proficient ways for sustainable growth. The transport sector consumes $\sim 20\%$ of the global energy and accounts for $\sim 18\%$ of the total anthropogenic greenhouse gas emissions [16]. The road transportation is identified as one of the prime sources for rising CO_2 in the environment [17]. The $\sim 33\%$ of fuel energy in a passenger car is consumed to overcome the friction in the engine, transmission, tires, and breaks [18]. The implementation of advanced tribological solutions and lubricant technologies can conserve as much as 17.5% of the energy being utilized in road transportation [19]. The dynamic changes in engine design and materials of engine parts/tools along with stringent environmental policies and emission regulations are posing several challenges to the lubricant industries. The use of good lubricants conserves the energy by lowering the friction, increase the life of engine components/machinery parts by protecting the surfaces against the wear, and decrease the CO_2 emission by the smooth operation of the engine.

Lubricants are composed of base oil and a diversified range of additives. The composition of lubricants governs by their targeted applications. Primarily, they lubricate the engineering surfaces by decreasing the friction and wear, dissipating the heat from contact interfaces, inhibiting the corrosion, subsidizing the oxidative events, and dispersing the foreign particles and oxidized products. Therefore, the lubricant additives include friction-modifier, antiwear, and extreme-pressure additive, corrosion-inhibitor, viscosity-modifier, oxidation-inhibitor, and so on are used to impart performance characteristics to the lubricates [20].

The surface-active antiwear and friction modifier additives play important roles, particularly in the boundary and mixed lubrication regimes, and decrease wear and friction by forming the lubricious tribo thin film. The zinc dialkyldithiophosphates (ZnDTPs)-based compounds are widely used to subsidize the wear and improve the lubrication properties. Over the recent past, the use of sulfur, phosphorus, halogen, and metal constituted conventional additives, including the ZnDTPs, are gaining significant concerns because of their adverse effects on the environment and poisoning effects to exhaust emission catalysts [21,22]. Thus, a lot of efforts have been directed to replace the conventional additive like ZnDTPs. Over the last few years, nanostructural layered materials (viz. graphene, h-BN, MoS_2 , WS_2) have shown significant progress in the fundamental studies signifying their potential for lubricant applications [6,11,23]. In this context, graphene has emerged as one of the most promising candidates because of intriguing low-frictional and antiwear properties to replace the conventional ZnDTPs-based additive.

The intriguing and favourable properties of graphene, h-BN, MoS_2 , and WS_2 nanosheets (Table 1), viz. remarkably high mechanical strength, excellent thermal conductivity, low shear strength, and large surface area protect the tribo-interfaces against the wear, dissipate the heat from contact interfaces, reduce the friction by facilitating the sliding under tribo-stress, and furnish a high coverage on contact interfaces. Fig. 1 shows the atomic structure of graphene, h-BN, MoS_2 , and WS_2 nanosheets. The h-BN nanosheets are made of atomic-thick lamellae, where the boron and nitrogen are alternatively placed and linked with each other by covalent linkages in a hexagonal lattice structure. These atomic-thick lamellae are held together by weak van der Waals interaction. The MoS_2 and WS_2 exhibit molecular layered structure, where each Mo/W atom is sandwiched between two sulfur atoms in a trigonal prismatic arrangement. These molecular lamellae are stacked with each other by weak van der Waals interaction. Under the tribo-stress, the weak interaction between atomic/molecular lamellae in graphene, h-BN, MoS_2 , and WS_2 nanosheets eases the shearing along the sliding direction because of interplanar slip and reduces the friction. The strong inter-atomic covalent bonding within each lamella in these 2D materials provides remarkably high mechanical strength and improves the antiwear properties [28–31]. The graphene, h-BN, MoS_2 , and WS_2 form a tribo-induced thin film on contact surfaces, which facilitates the sliding and avoids the direct contact between the tribo-pair. Therefore, the 2D layered materials are gaining significant interest in lubricant applications.

Graphene oxide (GO), an oxidized form of graphene has been a well-established precursor to prepare the reduced graphene oxide (rGO), graphene-based composites, and chemically-functionalized graphene. The oxygen functionalities in the GO facilitate the interaction with various chemical species via covalent linkages. The other interactive forces, such as the van der Waals linkages, π - π interactions, hydrogen bonding, and ionic interactions, assist for the non-covalent functionalization of graphene, GO, and rGO [32–34].

Table 1
Mechanical and thermal properties of 2D layered materials [26–27].

2D Materials	Young modulus, GPa	Breaking/fracture strength, GPa	Thermal conductivity, W/mK
Graphene	890–1060	130	3000–5000
h-BN	762–942	62–78	251
MoS_2	246–282	16–20	—
WS_2	254–280	—	—

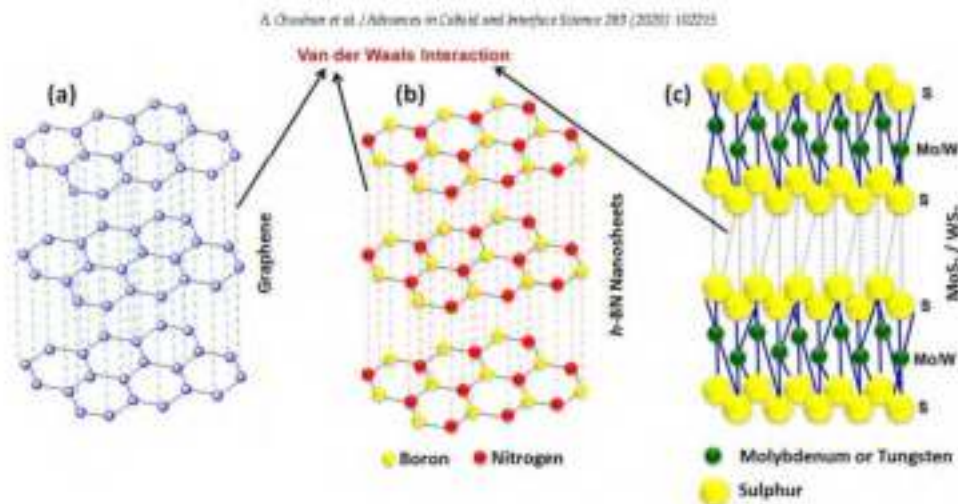


Fig. 1. Representative atomic structure of (a) graphite, (b) h-BN, (c) MoS₂ and WS₂ nanosheets along with illustration of van der Waals interaction between their atomic/molecular layers.

The GO exhibits plenty of oxygen functionalities such as hydroxyl, epoxide, carbonyl, ether, phenolic, and carboxylic groups. Among them, most of the epoxide, hydroxyl, and ether functionalities are located in the basal plane, whereas the carboxylic groups are placed along the edges of GO sheets [35–37]. The chemical reactivity of GO enables its potential for various applications, including lubrication. Hence, chemical and structural features of GO and rGO, their chemical functionalization by different chemical approaches, and their dispersion in aqueous, organic solvents, and a wide range of lubricating oils are critically reviewed. The graphene-based materials are subsequently applied as additives for liquid lubricant applications and discussed their roles in the reduction of friction and wear by emphasizing the lubrication mechanism based on microscopic and spectroscopic analyses of lubricated surfaces.

2. Synthesis and chemical structure of graphite oxide/graphene oxide

2.1. Synthesis of graphite oxide/graphene oxide

Harsh oxidation of graphite powder leads to the formation of graphite oxide. The extended interlamellar spacing in the graphite oxide because of basal plane oxygen functionalities and trapped water molecules facilitates their lateral splitting by the aid of sonication and forms the GO. The graphite oxide was discovered much earlier (in 1859) than the graphene (in 2004). A very first report by the British chemist S. C. Brodie has described the preparation of graphite oxide by oxidation of graphite in the presence of fuming nitric acid and potassium chlorate as oxidizing agents [38]. The C:O ratio of synthesized graphite oxide was reported to be 6L:04:1.85:37.11. Staudenmaier has addressed an improved oxidation method by addition of potassium chlorate in a small amount, and further oxidation was carried out by acidifying the mixture with concentrated sulphuric acid [39]. These methods were considered as hazardous and time-consuming processes. In 1958, Hummers and Offeman developed a rapid and relatively safer approach for preparing the graphite oxide by treating graphite with an anhydrous mixture of sulphuric acid, sodium nitrate, and potassium permanganate [40]. The Hummers' method avoids the use of highly corrosive fuming nitric acid. The use of potassium permanganate and sodium nitrate mixture resulted in an oxygenated-rich form of graphite

oxide. Recently, Tour et al. have improved the Hummers' method to increase the efficiency of the oxidation process. In the improved approach, sodium nitrate was eliminated, the amount of potassium permanganate was raised, and oxidation reaction was carried out in a mixture of sulphuric acid/phosphoric acid (9:1 ratio) to afford a higher amount of oxidized graphite compared to Hummers' method [41]. Improvements over the various synthesis methods of graphite oxide have been pursued continuously to yield effective alternatives. The graphite oxide is a non-stoichiometric material, and its chemical structure and composition are primarily governed by the synthesis approach, use of oxidizing reagents, reaction conditions, and graphite precursor.

Fundamentally, there is no considerable chemical difference in the graphite oxide and the GO. The graphite oxide is the platform material for the preparation of GO by exfoliation. Structurally, the GO consists of a limited number of lamellae (maximum 10), whereas the graphite oxide possesses an unlimited number of layers with expanded interlamellar spacing, driven by ample oxygen functionalities in the basal plane and the trapped water molecules via hydrogen bonding network. The graphite oxide prepared by variable oxidation routes (Fig. 2) is exfoliated in the water or polar solvents by the aid of sonication and afforded the GO [42,43]. The sonication (bath sonication or probe sonication) is well established and widely used approach for exfoliation of graphite oxide. However, it leads to some structural damages in the GO lamellae. The degree of oxygen functionalities in the graphite oxide, sonication medium and time, ultrasonic frequency, and intensity govern the exfoliation and quality of resultant GO.

2.2. Chemical structure of graphite oxide/graphene oxide

Unlike the graphene, GO is a structurally distorted sheet of sp² carbons in the honeycomb lattice structure with the infusion of numerous sp³ carbons due to oxygen functionalities. The atomic-thick lamellae of GO are linked with each other by the interlamellar spacing of 6 to 9 Å. The presence of oxygen functionalities in the basal plane and trapped water molecules by hydrogen interaction expand the interlamellar spacing. Thus the interlamellar spacing in GO (6–9 Å) is noted to be significantly higher than for the graphene (3.34 Å). The chemical structure of GO has been a subject of considerable debate and remains a challenge because of a large degree of structural disorder, non-stoichiometric composition, variation in oxygen functionalities, and lack of consistency

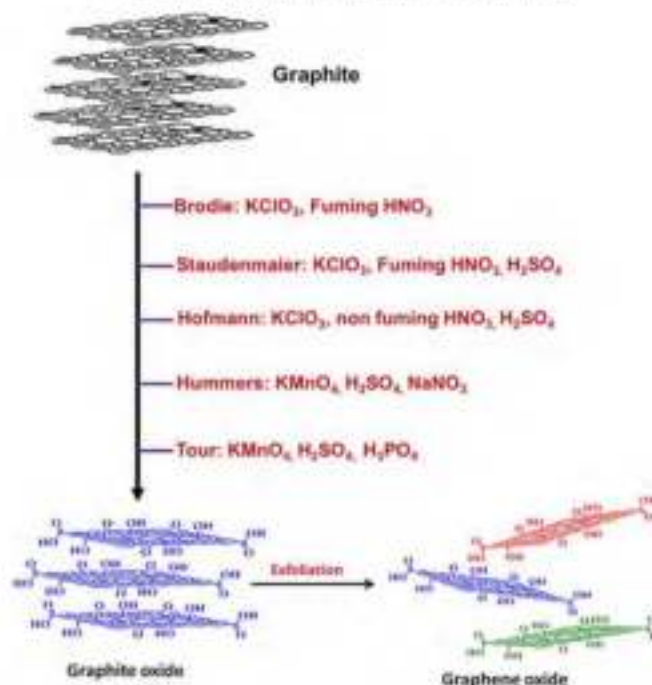


Fig. 2. Various chemical approaches for the synthesis of graphite oxide from graphite and their exfoliation into GO. Chemical synthesis route governs the C to O ratio in graphite oxide.

in the production cycles. Several structural models of graphite oxide and GO have been proposed over the last 60 years. The Hofmann, Ruess, Scholz-Boehm, Nakajima-Matsuo, Lerf-Klinowski, Dekany, and Ajayan models are well-debated structural models (Fig. 3).

A very first model of graphite oxide having repetitive units of 1,2-epoxides was proposed by Hofmann and Holst [44]. C. Ruess has demonstrated a modified structural model containing hydroxyl groups located at the sp^3 hybridized carbon in the basal plane [45]. Scholz and Boehm have proposed the hydroxyl and carbonyl groups at different positions along with $\text{C}=\text{C}$ units within the corrugated layers, whereas the ether-based oxygen groups were ruled out [46]. A new structure model of graphite oxide was proposed by Nakajima and Matsuo consisting of interlined carbon layers by sp^3 bonds perpendicular

to the carbon network. They emphasized the presence of hydroxyl and carbonyl groups below and above the carbon layers [47]. The Lerf and Klinowski model was proposed based on ^{13}C and ^1H NMR. They have demonstrated that graphite oxide is composed of the aromatic region with non-oxidized benzene rings and aliphatic six-membered rings. The presence of the hydroxyl group distorted the configuration and developed the wrinkles in the layers. Furthermore, the oxygen functionalities like 1,2-epoxide and hydroxyl groups are mainly present at the basal plane, while edges carry carbonyl groups [48]. It was further supported by nanostructural studies of the GO based on high-resolution transmission electron microscopic analyses [49]. Chemical and microstructural analysis results support the Lerf and Klinowski model. Therefore, it has gained wide acceptance [35,50]. Dekany et al. have proposed

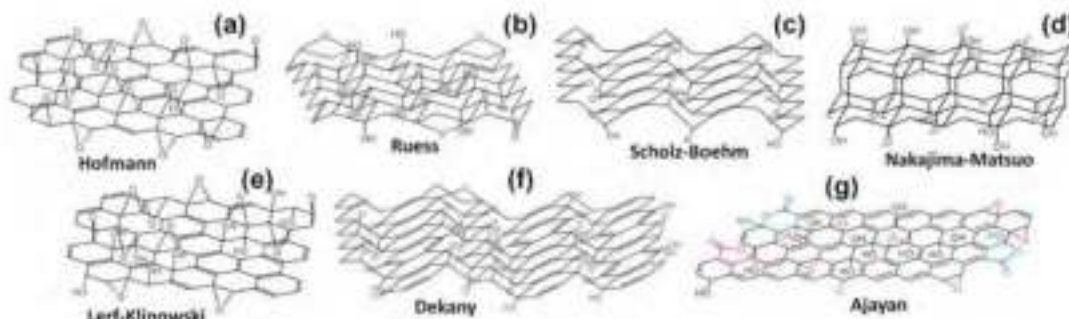


Fig. 3. Structural models of graphite oxide, (a) Hofmann model, (b) Ruess model, (c) Scholz-Boehm model, (d) Nakajima-Matsuo model, (e) Lerf-Klinowski model, (f) Dekany model, and (g) Ajayan model.

the structural model based on elemental analysis, XRD, ^{13}C MAS-NMR, FTIR, XPS, and ESR analyses [35]. The proposed structural model suggests that graphite oxide is comprised of two distinct domains of trans-linked cyclohexane chains and flat hexagon ribbons with C=C linkages (Fig. 3f). The phenolic groups were introduced to explain the acidity of the graphite oxide. Recently, Ajayan and co-workers have suggested the presence of five- and six-membered-ring lactols based on ^{13}C NMR of graphite oxide [31]. The ratio of different functional groups in the graphite oxide is proposed to be 115:1:63:10:9 for hydroxyl and epoxide; lactol O-C-O; graphitic sp^2 carbon; lactol, ester, and acid carbonyl; and ketone carbonyl, respectively.

The structural models of graphite oxide based on experimental results were further compared with density functional calculations. It predicted that partial oxidation is thermodynamically favourable over the complete oxidation [50]. The oxidation conditions primarily govern the distribution and abundance of oxygen functional groups in the graphite oxide. The graphite oxide and GO exhibits excellent dispersibility in water and it is attributed to high polarity and formation of hydrogen bonding by oxygen functionalities with water. The high polarity further extends the compatibility of GO with polar solvents [52]. A wide range of oxygen functionalities in GO facilitates its interaction with organic molecules, polymers, nanomaterials, etc. As a result, GO has been an excellent precursor to prepare the rGO, chemically-functionalized graphene, graphene-based composites, and their derivatives.

3. Synthesis and chemical structure of graphene/rGO

3.1. Synthesis of graphene/rGO

A breakthrough invention of atomically thin carbon films by Geim and Novoselov has exponentially propelled the potential of graphene for fundamental studies to application perspective [53]. Over the last 15 years, a lot of efforts have been directed to synthesize the graphene (Fig. 4). The mechanical exfoliation of highly oriented pyrolytic graphite (HOPG) using the scotch tape yielded a high-quality graphene. However, it has very low throughput and limits its scope for large-scale production. The epitaxial growth is an attractive alternative to prepare good-quality graphene for electronic applications [56,57]. The high temperature, vacuum, cost, and low throughput are few concerns which

limit the production of graphene at mass-scale, particularly for industrial applications. The chemical vapor deposition (CVD) of graphene on a variety of polycrystalline metal substrates has been addressed using the hydrocarbons viz. methane, ethylene, benzene, and acetylene as carbon sources [58]. The transfer and removal of graphene from the metal substrates, low yield, and reproducible growth at low temperature are few of the drawbacks which need to be defeated for the mass-scale industrial applications. The mechanical exfoliation of graphite is one of the promising approaches to produce large-scale graphene at low cost. The mechanical exfoliation by sonication, ball-milling, and recently emerged fluid dynamics approaches are gaining increasing attention for graphene production [53]. The solvents and exfoliating agents govern the quality and quantity of graphene produced by liquid-phase exfoliation. The addition of pre-designed exfoliating reagents in the given solvent improves the exfoliation yield and avoids the aggregation of graphene [59]. The solvents with the surface tension of $\sim 40 \text{ mJ.m}^{-2}$ are considered to be a suitable medium for liquid-phase exfoliation of graphite and then dispersion of resultant graphene. The *N*-methyl-2-pyrrolidone (NMP) has been recognized as one of the most efficient solvents for exfoliation of graphite [60]. The liquid-phase exfoliation is a highly versatile approach to produce good-quality graphene. Nevertheless, liquid-phase exfoliation has some disadvantages such as the requirement of a large quantity solvents, low yield, toxicity and irritant nature of most of the solvents, etc.

Chemical reduction of GO has been a most versatile, scalable, and economical approach to produce the rGO. Although the rGO carries some residual oxygen functionalities yet it exhibits a reasonable extent of graphene features. Therefore, rGO produced by chemical reduction is gaining considerable interest for industrial applications. The density functional calculations revealed that the complete reduction of graphite oxide into pure graphene is a difficult process [58]. Over the last decade, several reducing and dehydrating agents like hydrazine hydrate, sodium borohydride, LiAlH_4 , hydroquinone, thiourea, alcohols, etc. have been addressed to produce the rGO [62–64]. The residual oxygen functionalities and structural features of the rGO are collectively governed by the reducing agents and the reduction parameters. The hydrazine hydrate has been a most established reducing agent and exhibits excellent reduction efficiency to eliminate a significant degree of oxygen functionalities from the GO. Hence, it has been widely accepted for laboratory work to industrial production of the rGO at a mass-scale.

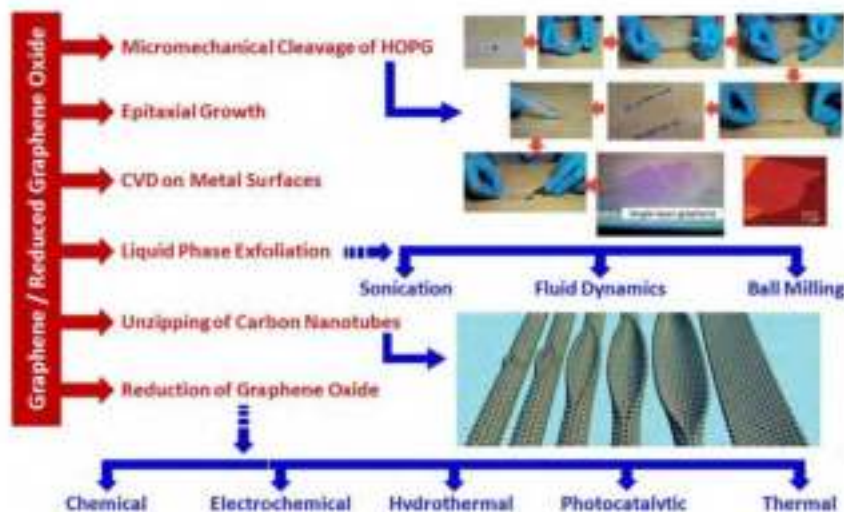


Fig. 4. Overview of variable approaches to prepare graphene and rGO. Reproduced with permission [53,54].

The hazardous, costiveness, and toxicity of most of chemical reducing agents are gaining significant concerns. As a result, green approaches are showing immense interest. Over the recent past, various environmentally-friendly reducing reagents viz. ascorbic acid, amino acids and plant extracts have been demonstrated to prepare the rGO [65–68]. Alternatively, the photocatalytic, electrochemical, and thermal reduction approaches have been developed to produce the rGO by avoiding the heteroatom contamination [69–71]. Ganguly et al. have monitored the deoxygenation events at different stages of thermal reduction process and suggested the formation of phenolic groups via a reaction between the epoxide groups and adjacent hydroxyl groups at $\sim 400^\circ\text{C}$. The high-

temperature reduction generates several structural defects and compromises the quality of the rGO. The sub- and supercritical hydrothermal reduction of GO facilitated the elimination of hydroxyl, carboxylic, epoxide, and carbonyl functionalities and recovered the π -conjugated network at a significant extent (Fig. 5). The hydrothermal reduction of GO is considered as a green and clean approach for regulating the deoxygenation events as a function of temperature [37,63]. The oxidative chemical processing of carbon nanotubes successfully produced the graphene. Tour et al. have developed a simple solution-based oxidative process to yield graphene nanoribbons by longitudinal unzipping of carbon nanotubes. The subsequent chemical reduction restored the chemical conductivity [54].

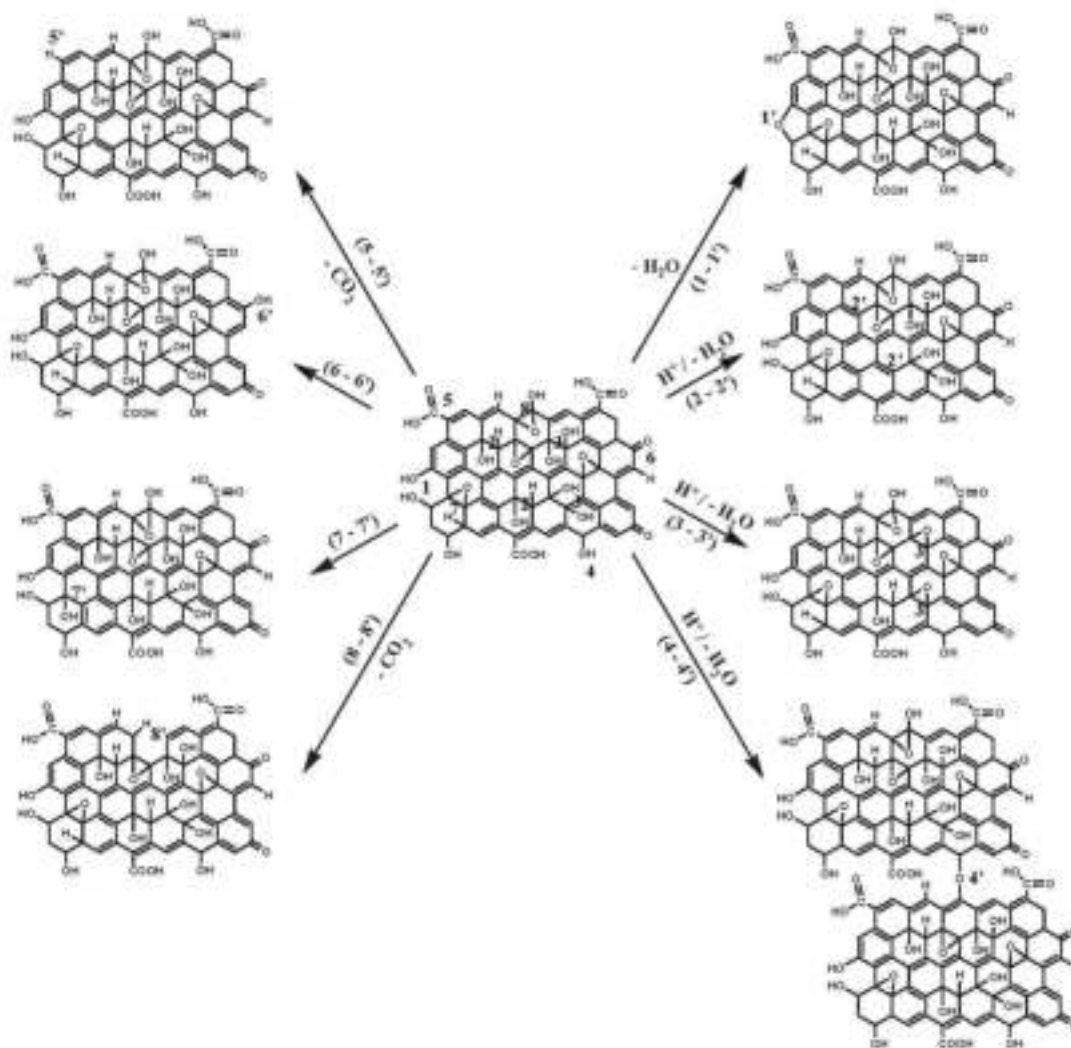


Fig. 5. Deoxygenation of GO by the elimination of hydroxyl, carboxylic, epoxide, and carbonyl groups during hydrothermal treatment using sub- and supercritical water. Chemical changes are indicated by numbering the respective carbons in the GO. The carbon associated with position numbers 1, 2, 3, 4, 5, 6, 7, and 8 represent dehydration to cyclic ether, dehydration to olefinic bond, dehydration to epoxide, intramolecular dehydration to ether, decarboxylation, reduction of carbonyl group into hydroxyl group, epoxide to hydroxyl conversion, and decarboxylation by restoring carbon from GO skeleton, respectively. Reproduced with permission [63].

3.2. Chemical structure of graphene/rGO

Recently, the Carbon Journal's editorial board has recommended the nomenclature of two-dimensional carbon materials and explicitly defined the graphene (monolayer vs. multilayer) and the rGO [72]. Fundamentally, graphene is a single-atom-thick sheet of hexagonally arranged, sp^2 -bonded carbon atoms. The lateral dimension of graphene can vary from a few nanometers to the micron range. It is mostly prepared by chemical vapor deposition on the metal substrate [58]. The stacking of atomic-thick lamellae of graphene via van der Waals interaction yielded multilayer graphene, and the number of lamellae in multilayer graphene varies from 2 to 10 [72]. In most studies, the term graphene includes the multilayer graphene as well. The rGO is made by chemical, thermal (hydrothermal/solvothermal), photochemical, microwave, or microbial processing of GO by reducing the oxygen content. The rGO exhibits structural defects, residual oxygen functionalities, and a little fraction of sp^3 carbon; as a result, it carries structural distortion in the 2D structure. The ratio of C/O in rGO varies by the oxidation process. Yet, the rGO carries the most of the fundamental properties of graphene, to a significant extent. Furthermore, ease of scalability and low cost make rGO favourable for several applications [73].

Graphene exhibits strong intraplanar bonding between the sp^2 hybrid carbons placed in the honeycomb lattice structure, whereas the weak interplanar interaction connects graphene lamellae in the (002) plane. The two-dimensional structure, along with an extended π -electron network of graphene renders remarkable electronic properties. The interactions between electrons and honeycomb lattice structure yield the quasiparticles, which are also known as massless Dirac fermions and offer exceptional electronic phenomena such as anomalous room-temperature quantum hall effect (QHE) and extraordinary high carrier mobility [1]. Graphene shows weak affinity and interaction for chemical transformations. However, the atomic hydrogen can react with graphene and convert the highly conductive metallic state of graphene into an insulator, also known as graphane [74]. The intercalation of atoms and molecules in the basal plane of graphene acted as nanoreactors and enhanced the surface reactions. The small spacing in the graphene leads to new chemistry viz. "catalysis" and "electrochemistry" under the cover [75]. The graphene has been established as a versatile platform for grafting of various catalytic sites for heterogenization of homogeneous catalysts [76,77]. The doping of heteroatoms, chemical grafting, loading of nanomaterials, introduction of structural defects, and chemical functionalization have expanded the potential of graphene and rGO for

a wide range of chemical, electrocatalytic and photocatalytic reactions [78–83]. The N-doped graphene exhibits high photocatalytic activity for reduction of CO_2 and electrocatalytic activity for the oxygen reduction reactions. The nitrogen-doped sites viz. pyridinic N, graphitic N and pyrrolic N in the graphene generate the catalytic centres for the electrocatalysis and photocatalysis [84,85]. The graphene deposited on a solid surface revealed significant differences in their properties, which includes morphological features, surface chemistry, and surface/interface properties. The deposition and interaction of graphene with semiconductors, ceramics, polymers, biomaterials, and metals also influence the properties of graphene. Therefore, the surface/interface properties are gaining significant attention for several applications such as catalysis, optoelectronics, lubrication, corrosion, coatings, composites, and so on [86]. The covalent functionalization in the basal plane of graphene can be obtained by breaking the sp^2 linkages and the formation of sp^3 carbons. The oxidation of graphite into graphite oxide and then exfoliation into GO is one of the simplest examples to generate the oxygen functionalities in the basal plane of graphene. The unpaired electrons which are produced at the adjoining sites to the point of covalent bonding increase the chemical reactivity, leading to a chain reaction from the point of the initial attack [87]. The surface properties of graphene controlled by their preparation routes, have been applied to fabricate the superhydrophobic thin film to bring new opportunities for high-performance coating and multifunctional devices [88]. In the recent past, several efforts have been made towards covalent and non-covalent functionalization of graphene to meet the requirement for a wide range of applications and expand their potential for industrial utilization.

4. Dispersion of GO, graphene, rGO and chemically-functionalized graphene

The GO has been an ideal candidate for production of solution-processable rGO at a large scale for various applications. The presence of ample oxygen functionalities makes GO a highly polar and hydrophilic material, which facilitate long-term dispersibility of GO in water. The dispersion of GO in organic solvents has been gaining significant importance for expanding their potential for a diversified range of applications. The stability of GO dispersion prepared by exfoliation of graphite oxide in different organic solvents via bath sonication was explored to understand the interactive forces [89]. As shown in Fig. 8, the GO could be dispersed in several organic solvents, except in methanol, dichloromethane (DCM), *n*-hexane, *o*-xylene. The poor compatibility of GO with acetone,

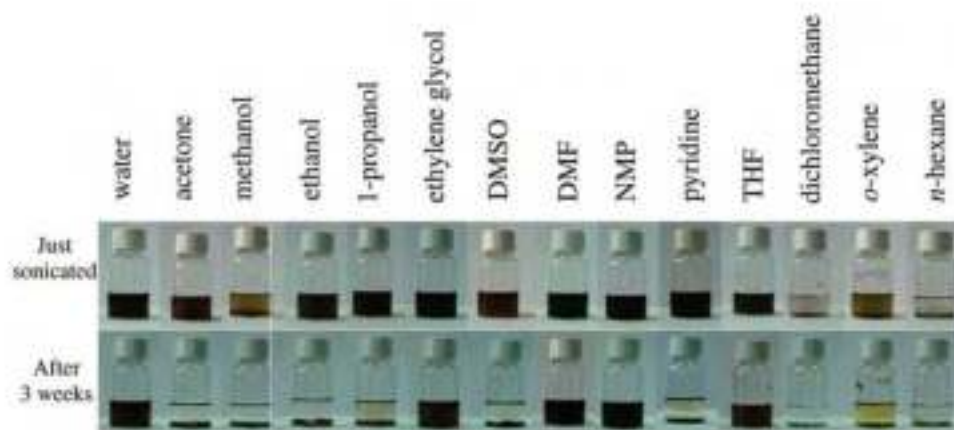


Fig. 8. Digital photographs of GO dispersion prepared via bath sonication in water and organic solvents. Top-line vials show dispersions immediately after sonication, whereas bottom-line vials display dispersions after three weeks in different solvents. Reproduced with permission [89].

ethanol, 1-propanol, dimethyl sulfoxide (DMSO), and pyridine allow GO lamellae to gradually aggregate and settle down in the solvent. The GO dispersion in *N,N*-dimethylformamide (DMF), NMP, tetrahydrofuran (THF), and ethylene glycol exhibits long-term dispersibility. The high electrical dipole moment of water (1.82 D), DMF (3.24 D), NMP (4.09 D), THF (1.75 D), and ethylene glycol (2.31 D) favours the dispersibility of polar GO in these solvents. However, the poor dispersibility of GO in the DMSO (high electrical dipole moment; 4.09 D) signified that besides the polarity of the solvents other parameters are also important for determining the dispersibility [89]. Ryan et al. demonstrated that the dispersion of GO and rGO is governed by surface tension, solvent polarity, and the Hansen solubility parameters [90]. The dispersion cohesion (δ_H), hydrogen bonding cohesion (δ_H), and polarity cohesion (δ_P) collectively represent the Hansen solubility parameters [91]. The dispersion of GO and rGO in highly polar solvents like DMF, NMP, etc. via sonication is considered as an energy-intensive approach, and it furnishes low yield. The homogeneous dispersion of rGO in organic solvents prepared by a chemical approach using GO dispersion in the synergistic ratio of water and applicable organic solvents like NMP, DMF, DMSO, THF, ethanol, acetone, acetonitrile, etc. promises good potential for a wide range of chemically processable applications. The reduction of GO in these solvents using hydrazine hydrate as a reducing agent furnished the homogeneous dispersion of rGO. The NMP, DMF, DMSO, THF, and ethanol having the sum of $\delta_H + \delta_P$ in the range of 13–29 exhibited a good dispersion of rGO. The solvent with $\delta_H + \delta_P$ lesser than 10 and higher than 30 could not disperse the rGO [91]. The surface energies of GO (62 mJ/m²) and rGO (46 mJ/m²) also govern their dispersibility. The polar and hydrogen bonding components are significantly higher in the GO compared to rGO. Therefore, GO exhibited good dispersibility in most polar solvents. The equal values of the Hildebrand solubility parameter (δ_T) of solvents and solute (GO/rGO) favours the dispersion. The higher dispersibility of rGO ($\delta_T = 22$) in dichloromethane ($\delta_T = 20.2$; 1.6 µg/mL), chlorobenzene ($\delta_T = 19.6$; 3.4 µg/mL), *o*-dichlorobenzene ($\delta_T = 20.5$; 8.94 µg/mL), 1-chloronaphthalene ($\delta_T = 20.6$; 8.1 µg/mL) was explained based on compatibility of their Hildebrand solubility parameter [90].

The production of dispersible rGO by chemical reduction routes carries not only residual oxygen functionalities but also exhibits several structural defects, which compromise their electronic, mechanical, and structural properties. Alternatively, liquid-phase exfoliation of graphite in various organic solvents via sonication or intercalation approaches has gained attention for the production of defect-free large-size graphene sheets [53,59]. The liquid-phase exfoliation is one of the simple methods to produce large quantities of graphene, which can be useful for both electronic and biomedical applications. The energy required to exfoliate graphene should be balanced by interaction energy between the graphene and the solvent. Coleman et al. have revealed that solvents with surface tension in a range of 40–50 mJ/m² favour the exfoliation of graphite and dispersion of graphene [60]. The *o*-dichlorobenzene (DCB) exhibits excellent capability to produce the graphene by exfoliation of graphitic materials. The higher surface tension of DCB (36.6 mJ/m²) favoured the exfoliation of graphite and furnished the high yield of graphene. Furthermore, the π - π interaction between the aromatic rings of DCB and graphene facilitates dispersion stability [92]. Several studies have been carried out demonstrating the exfoliation and dispersion of graphene in NMP. However, the low dispersion concentration limits their potential for several applications. A multi-step process of exfoliation could yield a significantly high concentration of graphene in the NMP. The centrifugation of exfoliated graphite in NMP, then filtration to obtain the exfoliated powder, followed by re-dispersion in the NMP via sonication significantly improved the dispersion concentration having an average of 3–4 layers in each sheet of graphene. Even after some sedimentation, the 26–28 mg/mL⁻¹ concentration of dispersed graphene was found with indefinite stability [93].

The chemical stabilizers via surfactants, functionalized polymers, aromatic-rich molecules, and ionic liquids exfoliate the graphite via sonication by overcoming the van der Waals

interactions between the graphene lamellae. Simultaneously, the stabilizers are adsorbed on the exfoliated graphene lamellae through non-covalent interactive forces via π - π , cation- π , and hydrophobic interactions, which avoid the re-stacking to make their stable dispersion [94–98]. The exfoliation of graphite in organic solvents of low boiling points with the presence of stabilizers has been gaining increasing interest. The hyperbranched polyethylene (HBPE) assisted exfoliation of graphite in low boiling points solvents viz. chloroform and THF yielded the stable dispersion, whereas the HBPE provides steric stabilization against restacking of graphene lamellae [96]. The chemical reduction of GO in the controlled reaction media and the presence of stabilizer facilitates the dispersion of rGO in the targeted solvents. The green tea, rich in polyphenols, not only reduced the GO but also made them dispersible in most organic solvents and water (Fig. 7). The π - π interaction between the aromatic rings of polyphenols and rGO furnished the stable dispersibility in NMP, DMSO, DMF, ethanol, and acetone [67]. The surfactants play a critical role to disperse the hydrophobic graphene in water. The polyethylene nonylphenylether (Igepal CO890) a non-ionic surfactant (200–300 ppm) furnished the stable dispersion of graphene in water [99].

The chemistry of surfactants governs their interaction with targeted nanomaterials. The molecular strategy to design the surfactants by introducing the ionic groups in an electron-deficient π -conjugated unit along with flexible alkyl spacers significantly enhanced the stability of graphene in the water. The electron-deficient aromatic units of surfactants show good affinity with graphene via π - π interaction and simultaneously ionic groups facilitate the dispersibility of surfactant-stabilized graphene lamellae in the water. The resultant dispersion of graphene was found to be stable for more than four months [101]. The presence of zwitterionic polymeric dispersant such as succinic anhydride-functionalized polyethylenimine (PEI-SA) facilitated the exfoliation of graphene and simultaneously stabilized the dispersion in water. The cation- π interaction between the protonated amines in dispersant and π -electron-rich structure in graphene along with the steric hindrance of PEI-SA stabilized the graphene dispersion in the water for more than six months [102]. The presence of negatively charged carboxylic group (COO⁻) in the GO and rGO facilitate their dispersion in aqueous and organic solvents based on ionic interaction with surfactants. The hydrophilic end of surfactant-like dioctyltrimethylammonium cation showed ionic interaction with negatively charged carboxylic groups of GO and rGO, whereas hydrophobic tail showed affinity towards organic solvents. A shaking of such mixture transferred the surfactant-stabilized GO and rGO in the chloroform, as demonstrated in Fig. 8 [100]. A simple approach to prepare the graphene by Li⁺-DMF assisted exfoliation

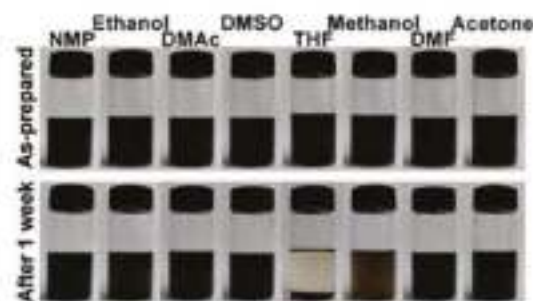


Fig. 7. Dispersion of rGO prepared via reduction of GO using the green tea. Concentration of graphene in organic solvents: 0.2 mg/mL⁻¹. Reproduced with permission [67]. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

showed stable dispersion of graphene in water without sonication of surfactants. The positive charge introduced due to intercalation of Li^+ to graphene attracted the negatively charged hydroxyl groups and made them hydrophilic for stable dispersion in water [103].

Ionic liquids (ILs) are established as an excellent exfoliating and dispersing medium for graphene. The interactions between the π -electron clouds of graphene and positive charge of cationic moieties in ionic liquids facilitate the dispersion. Importantly, most of the ILs have surface tensions in the range of surface energy of graphite, which is the prime requirement for any dispersing media/solvent to exfoliate the graphite [112,113]. The graphite was efficiently exfoliated using the 1-hexyl-3-methylimidazolium hexafluorophosphate (HMM-PF_6) ionic liquid, and resultant dispersion exhibited stability more than three weeks with maximum graphene concentration of 5.33 mg mL^{-1} [112]. The molecular dynamics study and experimental kinetic analysis revealed that collective van der Waals forces between graphene and ILs express the long-range dispersion interaction and short-range cation- π interactions [114]. The low-viscosity ionic liquid-like 1-ethyl-3-methylimidazolium dicyanamide (EMIM-DCA) IL yielded stable dispersion of 1 wt% graphene via mechanical mixing and sonication, and it exhibited stability for more than two months [114]. The poly(1-vinyl-3-butylimidazolium chloride), a polymeric ionic liquid stabilized-graphene sheets furnished stable dispersion in the 1-butyl-3-methylimidazolium hexafluorophosphate (BMIM-PF_6) ionic liquid driven by π - π interaction between the imidazolium rings of the polymeric ionic liquid and the graphene lamellae [115]. The ionic liquid-based polyether, poly(1-glycidyl-3-methylimidazolium chloride) as stabilizers furnished the stable dispersion of graphene in the aqueous medium [120]. Table 2 summarizes the dispersion stability of GO, rGO, and graphene in a wide range of organic solvents and water.

Solvochemical reduction of GO in organic solvents is the most emergent chemical reduction to produce the stable dispersion of rGO in the processable solvents without the use of any stabilizer or surfactant [97,121,122]. The solvochemical reduction of GO in DMF yielded the $[\text{NH}_2(\text{CH}_2)_2]^+$ -stabilized graphene (STG), which is dispersible in the DMF. The dispersion of STG (0.3 mg mL^{-1}) in DMF was found to be stable for more than one year [97]. The STG could be re-dispersed (Fig. 9) in those organic solvents (NMP, DMSO, DMAc, and acetonitrile), which have a product of polarity and hydrogen bonding cohesive parameters in the range of 13–29. The solvochemical reduction of GO in NMP functionalized the rGO (STRG) by penary radicals being generated by solvochemical processing of NMP in the presence of air. The steric effect of functionalized groups on the surface of STRG sheets facilitates their dispersion in organic solvents via DMF, NMP, propylene carbonate, ethanol, and THF [121]. Karner et al. revealed that the combination of thermal and chemical deoxygenation events by NMP under the solvochemical processing produced the rGO (SRGO). The NMP molecules are grafted via hydrogen linkages with residual oxygen functionalities

of SRGO. The resultant SRGO exhibited good dispersibility in organic solvents like DMSO, ethyl acetate, THF, acetonitrile, DMF, and CHCl_3 , with minimal precipitation even after six weeks [122]. Table 3 summarizes the dispersion stability of chemically functionalized rGO, polymeric-stabilized rGO/graphene, and dispersant-assisted rGO/graphene in a wide range of organic solvents and water.

The γ -ray irradiation to GO has been established as an environmentally benign and cost-effective approach for preparing the rGO at a large-scale. Zhang et al. demonstrated a simple method to produce a stable dispersion of rGO by γ -ray irradiation of GO in DMF [142]. The electrons being formed by the interaction of high-energy γ -ray irradiation with DMF facilitate the reduction of GO. Furthermore, the formation of $\text{N}(\text{CH}_2)_2^+$ by radiolysis of DMF deposited on the surface of rGO sheets. As a result, rGO prepared by γ -ray irradiation could be re-dispersed in DMF, THF, isopropanol, Glycol, NMP, as shown in Fig. 10. These dispersions are noted to be stable for two weeks. The γ -ray-based radiolysis also functionalized the rGO with poly(ethylene)glycol (PEG-200) via hydrogen bonding network between the residual oxygen functionalities of rGO and the oxygen atom of PEG molecules. The PEG-functionalized rGO exhibited stable dispersibility [143].

Dispersibility of graphene-based materials in lube oils is one of the major prerequisites for furnishing intriguing low-frictional and antiwear properties. The high cohesive interaction driven by large surface area, structural defects, and π - π interaction between the graphene sheets leads to the formation of agglomerates. The larger size agglomerates of graphene adversely influenced the uninterrupted supply of graphene to contact interfaces. The highly disorganized arrangement of graphene sheets in such agglomerates damages the interplanar low shearing properties of graphene. Therefore, the intriguing low-frictional and antiwear properties of graphene-based materials can be achieved efficiently when they are thoroughly dispersed, and their fascinating tribological properties are not compromised.

The use of dispersants/surfactants and the chemical functionalization of graphene are recognized as simple and economic approaches to improve the dispersibility of graphene in lube-base oils. The use of dispersants and grafting of surface-active molecules over the graphene sheets are selected based on the nature of lube oils being used for dispersion of graphene-based materials. The grafting of long alkyl chains on GO and rGO increase the compatibility of resultant graphene-based materials with mineral and synthetic lube base oils, driven by the van der Waals interaction between the long alkyl chains of surface-active molecules grafted on graphene-based materials and the hydrocarbon content of lube base oil [144]. Furthermore, the chain length of grafted molecules also governs the degree of dispersibility. The dispersion stability of alkylated graphene in hydrocarbon solvents increased with increasing the chain length of alkyl groups grafted over the graphene's surface. The grafting of 3,5-di-*tert*-butyl-4-hydroxybenzaldehyde over

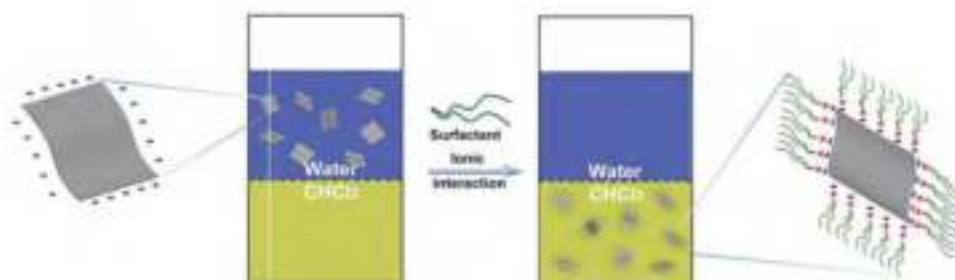


Fig. 8. Schematic illustration representing the transfer of graphene sheets from water to chloroform with the aid of surfactant based on ionic interactions. Reproduced with permission [103].

Table 2
Dispersion stability of GO, rGO, and graphene in a wide range of organic solvents and water.

No.	Sample description	Medium of dispersion	Dose	Dispersion stability	Reference
1	GO prepared by Hummer's method	NMP, DMF, THF, ethylene glycol	0.5 mg.mL ⁻¹	>3 Weeks	[88]
2	Ultrasound assisted GO dispersion	NMP, DMF, THF, Water, ethylene glycol	0.5 mg.mL ⁻¹	>24 hours	[90]
3	rGO prepared by hydrazine reduction of GO	1-Chloronaphthalene, NMP, o-chlorobenzene	0.5 mg.mL ⁻¹	>24 hours	[90]
4	rGO dispersion by reduction of GO in DMF and then top-up with targeted organic solvents	DMF, DMSO, THF, NMP, acetone, ethanol, acetonitrile	0.05 mg.mL ⁻¹	Stable dispersion	[91]
5	Graphene prepared by liquid-phase exfoliation of graphite	NMP	0.01 mg.mL ⁻¹	3 Months	[92]
6	Graphene prepared by liquid-phase exfoliation of graphite	o-DCB	0.01 mg.mL ⁻¹	–	[92]
7	Liquid-phase exfoliated graphene via rubbing approach	NMP	20–25 mg.mL ⁻¹	Indefinitely	[93]
8	Liquid-phase exfoliated graphene in low boiling point solvents	Chloroform, ethanol	0.5 mg.mL ⁻¹	80% content stable	[94]
9	Pristine graphene	THF	–	Stable dispersion	[95]
10	rGO prepared in organic media via chemical reduction of GO	NMP, DMF	–	2 Months	[96]
11	Graphene prepared by sonochemical reduction of GO	DMF	0.5 mg.mL ⁻¹	1 Year	[97]
12	Graphene prepared by liquid-phase exfoliation assisted by hyperbranched polyethylene stabilizer	THF	0.045 mg.mL ⁻¹	–	[98]
13	Graphene prepared via reduction of GO in green tea (polyphenols based stabilizer)	CHCl ₃	0.18 mg.mL ⁻¹	–	[99]
14	Graphene prepared via reduction of GO in green tea (polyphenols based stabilizer)	NMP, DMSO, DMF, ethanol, acetone	0.2 mg.mL ⁻¹	1 Week	[97]
14	Surfactant-stabilized graphene sheets	Water	0.75 mg.mL ⁻¹	4 Months	[101]
15	Graphene prepared by cation- π interaction induced exfoliation of graphite using polymeric dispersant	Water	0.50 mg.mL ⁻¹	6 Months	[102]
16	Graphene prepared by G-OH driven exfoliation of graphite without sonication and surfactant	Water	0.09 mg.mL ⁻¹	–	[103]
17	Solvent-processed exfoliated graphene nanoplatelets with the aid of 15% polyacrylate-based surfactant	Water	1%	6 Hours	[104]
18	Graphene platelets prepared by fluorinated surfactant-assisted ultrasonic exfoliation of graphite	Water	–	3 Weeks	[105]
19	Graphene prepared by sodium cholate surfactant-assisted exfoliation of graphite	Water	0.41 mg.mL ⁻¹	Stable dispersion	[106]
20	Graphene prepared by sodium decylsulfate surfactant-assisted exfoliation of graphite	Water	2.58 mg.mL ⁻¹	Stable dispersion	[106]
21	Graphene prepared by sodium chloride surfactant-assisted exfoliation of graphite	Water	6.3 mg.mL ⁻¹	Stable dispersion	[107]
22	Graphene prepared by sodium laurylsulfate surfactant-assisted exfoliation of graphite	Water	7.1 mg.mL ⁻¹	–	[108]
23	Graphene prepared by bis(hydroxy)phenylpropane (as exfoliant and dispersant)-assisted exfoliation of graphite	Water	0.26 mg.mL ⁻¹	2 Months	[109]
24	Solvent-processed exfoliated graphene with the aid of vinylimidazole-based polymer as a high-performance stabilizer	Water	3.25 mg.mL ⁻¹	–	[110]
25	Liquid phase production of graphene by sodium dodecylbenzene sulfonate surfactant-assisted exfoliation of graphite	Water	–	>4 Weeks	[111]
26	Liquid-phase exfoliated graphene in ionic liquid	EMBM-Ph ₃ ionic liquid	3.33 mg.mL ⁻¹	3 Weeks	[112]
27	Liquid-phase exfoliated graphene in ionic liquid	EMBM-Tf ₂ N ionic liquid	0.95 mg.mL ⁻¹	Stable and irreversible	[113]
28	Graphene dispersion via mechanical mixing and ionization	EMBM-DCA ionic liquid	1 wt%	2 Months	[114]
29	Polymeric ionic liquid-stabilized graphene	BMI-Ph ₃ ionic liquid	0.8 mg.mL ⁻¹	2 Months	[115]
30	Liquid-phase exfoliated graphene in levulinic acid	levulinic acid	0.005 mg.mL ⁻¹	–	[116]
31	Sonochemical free dispersion of GO using internal pressure from release of intercalated CO ₂	Ethanol, THF, DMF, NMP	1.0 mg.mL ⁻¹	Stable dispersion	[117]
32	Exfoliated graphene	Water	3.0 mg.mL ⁻¹	–	[118]

the rGO sheets facilitates its dispersion in SN-150 mineral lube oil. The tertiary butyl groups over the surface of chemically-functionalized rGO exhibit cohesive interaction with hydrocarbon chains of SN-150 oil driven by van der Waals interaction, resulting in good dispersion for enhancement of lubrication properties [126]. The graphene shows immiscibility with synthetic polyol lube base oil. The covalent grafting of octadecyl chains by using the organosilanes improved the dispersion stability. Furthermore, the grafting patterns of organosilanes over the surface of GO and rGO govern their dispersibility [145]. The modification of GO and rGO sheets with imidazolium ionic liquids (ILs) by simple mixing or covalent grafting improved their compatibility with various lube base oil via multialkylated cyclopentane and PEG 200 [146,147]. The variable strategies for synthesizing and functionalization of graphene, GO, rGO are designed considering the chemical nature and physico-chemical properties of lube base oil.

5. Tribological applications of graphene-based materials

5.1. Graphene, GO and rGO as lubricant additive

Water-based lubricants are widely used for several engineering applications such as oil extraction, cutting fluids, metal finishing, hydraulic fluids, metal forming processes, and so on. The low cost, abundant availability, good thermal conductivity, and environmental compatibility make water an excellent lubricating media. The natural systems use water as a lubricating fluid. In the biological systems, the presence of proteins, especially glycol-proteins, modify the interactive surfaces to make them lubricated in the presence of water-based fluids. The water without additives is a poor lubricant in most of the man-made engineering system. The high adhesion, formation of oxides/hydroxides, and corrosion-induced degradation limits its potential for metal surfaces. Therefore, water-miscible additives, which can protect the metal

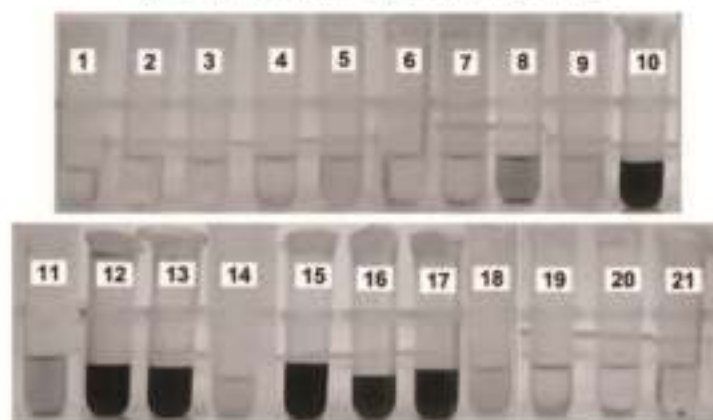


Fig. 9. Dispersion of $(\text{OH})_2(\text{CH}_3)_2^+$ -stabilized graphene (SG) in variable organic solvents and water. The number on each sample represents the solvents: (1) hexane, (2) heptane, (3) octane, (4) decane, (5) dodecane, (6) DCM, (7) chloroform, (8) ethyl acetate, (9) THF, (10) toluene, (11) acetone, (12) DMF, (13) N,N-dimethylacetamide (DMAc), (14) 1-isopropanol, (15) acetonitrile, (16) DMSO, (17) DMSO, (18) ethanol, (19) ethylene glycol, (20) water, and (21) methanol. Reproduced with permission [97].

surfaces and dissipate the heat, are gaining significant attention for aqueous-based lubricating systems.

The tribological system generates heat because of friction. The dissipation of heat from contact interfaces is the prime requirement for a good lubricant system [140]. The conductivity of graphene-blended lubricants is governed by intrinsic thermal property of graphene along with the interfacial thermal resistance between the graphene-based materials and the lube oil. The 2D planar structure, along with significantly high surface area, makes graphene-based materials highly promising candidates to take away the heat from contact zones when they are nicely dispersed in the lubricating medium. The higher degree of interfacial contacts between the GO/chemically-functionalized rGO and water, driven by excellent dispersibility, notably reduced the contact resistance and furnished the excellent thermal conductivity to water [140,150]. The inherently high thermal conductivity in the chemically-functionalized rGO provided excellent heat dissipation properties to lubricant system.

The aqueous dispersion of GO has been an excellent lubricating fluid to reduce friction and wear. The stable dispersion of single-layered GO in water (1 wt%) significantly improved the lubrication between the steel plate and tungsten carbide (WC) ball (Fig. 11). The average coefficient of friction was reduced to ~ 0.05 from 0.4 (for purified water) and showed no apparent wear even after 60,000 cyclic contacts. The elemental mapping results suggested the adsorption of GO sheets on the contact interfaces for the enhanced lubrication performance [151]. The aqueous dispersion of GO as a lubricating medium between the diamond-like carbon-coated steel disc and stainless steel ball decreased the coefficient of friction by 57% compared to water. The presence of GO inhibited the oxidative corrosion of contact interfaces. The lowering of friction was attributed to the formation of GO-based tribo-layer by the embedding of GO sheets on the contact interface and binding of water molecules on GO-based tribo-layer, which facilitates the lubrication [152].

The pH of GO dispersion influenced the chemical structure and nature of the charge. The increasing pH from acidic to alkaline not only breaks-down the sheets but also reduced them; consequently, significant changes were observed in the tribo-performance. The coefficient of friction and wear were increased at higher pH. The variable lubrication mechanisms at different pH were attributed to the physicochemical and morphological features of GO. The larger size of GO sheets at acidic pH tended to cover the contact interfaces by forming a protective tribo-

film, which reduced the wear and friction, effectively. The relatively smaller sheets of GO at alkaline pH couldn't form a good quality tribo-film, whereas they entered the contact area and caused the high density to scratch the sliding pairs. Consequently, high wear with alkaline dispersion of GO [153]. The efficacy of lubrication by GO-based dispersion can be improved using the chemically-functionalized engineering surfaces. The 3-aminopropyltriethoxysilane (APS) coated aluminum facilitates the deposition of GO sheets driven by the interaction between the oxygen functionalities of GO and amino groups of APS thin film, which results in the formation of durable and good quality tribo-film on the contact interface. The tribo-results revealed the significant reduction of friction (44%) and wear rate (80%) compared to the deionized water, and it was attributed to the synergistic effect of the amino-terminated surface of aluminum and GO additive [154]. The fluorination of GO enhances lubrication performance. The fluorination of GO at variable reaction conditions (temperature: 40 and 180 °C; F_2 concentration: 1 and 6%) yielded the oxygen-enriched GO (OFG) and fluorine-enriched GO (FGO). The difference in chemical composition, structural features, and dispersibility of GO, OFG, and FGO in water governs their tribo-performance. The aqueous dispersion of OFG (0.5 mg. mL^{-1}) decreased the wear rate between the steel tribo-pair by 47% and 31% compared to that of water and GO dispersion, respectively. However, FGO with poor dispersibility with water couldn't improve the tribo-performance of water. The ample oxygen functionalities make the OFG highly dispersible in the water, whereas the presence of fluorine in the OFG decreased the interlamellar-spacing, which facilitates the interplanar slips under the tribo-stress. The collective effects of good dispersibility and self-lubricating properties of OFG offered excellent tribo-performance for aqueous-based lubricant system [155].

The GO as an additive to water showed significantly improved friction-reducing and antiwear capability than the graphene. The superior tribological performance of GO nanofluids is attributed to highly hydrophilic oxygen-enriched functionalized groups (hydroxide, carboxylic, carbonyl, and so on) over the surface of GO. The high polarity of GO, driven by oxygen functionalities, facilitates the deposition on metal-based tribo-surfaces. The improved adhesion between the GO and the metal surface furnish long-term stability and load-bearing capacity to GO-based tribo film compared to the graphene [156]. Furthermore, the excellent dispersibility GO in the water ensures its uninterrupted supply to the contact area and form the protective layer on the interacting surfaces. Table 4 summarizes the various approaches for the preparation of GO dispersion in water and then their lubrication

Table 3

Dispersion stability of chemically functionalized rGO, polymeric-stabilized rGO/graphene, and dispersant-assisted rGO/graphene in a wide range of organic solvents and water.

No.	Sample description	Medium of dispersion	Dose	Dispersion stability	Reference
1	Graphene dispersion via solvothermal reduction of graphene oxide in DMF	DMF	0.3 mg mL ⁻¹	1 Year	[87]
2	Solvothermally-reduced and functionalized GO by NMP	NMP, DMSO, DMAc, acetonitrile	–	–	–
3	Solvothermally reduced graphene oxide (SRGO) by NMP	NMP	1.4 mg mL ⁻¹	1 Month	[121]
4	Chemically functionalized graphene by 1,3 dipolar cycloaddition of azorethynylide	DMF, ethanol, THF, propylene carbonate	–	–	–
5	Hydrazine-assisted stabilized graphene	DMSO, ethyl acetate, THF, acetonitrile	1 mg mL ⁻¹	6 Weeks	[122]
6	Ethylene diamine succinic acid functionalized rGO by cyclization and then reduction	DMF, ethanol, CHCl ₃	0.5 mg mL ⁻¹	> 30 Days	[123]
7	1,5-di (tert-butyl-4-hydroxyphenyl)ethyne grafted graphene (Gr-DDBHA)	Water	–	Few months	[124]
8	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[125]
9	1,5-di (tert-butyl-4-hydroxyphenyl)ethyne grafted graphene (Gr-DDBHA)	Water	–	–	–
10	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[126]
11	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[127]
12	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[128]
13	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[129]
14	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[130]
15	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[131]
16	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[132]
17	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[133]
18	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[134]
19	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[135]
20	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[136]
21	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[137]
22	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[138]
23	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[139]
24	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[140]
25	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[141]
26	Hydrazine-assisted stabilized graphene	Water	1 mg mL ⁻¹	3 Months	[142]

performance under variable conditions viz. applied load, speed, contact geometry, and frictional pair.

The incompatibility between the highly polar GO nanosheets and the non-polar mineral lube oil leads to poor dispersion. It has been a great challenge for their utilization in industrial lubricant applications. Recently, Wu et al. revealed that GO could be dispersed in the aviation lubricant with the aid of sonication. However, such dispersion showed poor stability for a long time. Nonetheless, GO as an additive (0.5 wt%) in aviation lubricant could improve the load-carrying capacity, friction, and wear-resistant by 18%, 15%, and 34%, respectively, between the Si₃N₄ and steel tribo-pair. The detailed studies of worn surfaces based on EDX and Raman spectroscopic measurements suggested that GO can easily enter the contact interfaces and formed a protective thin film, which reduced the friction and protected the contact interfaces against the wear [166]. The GO dispersion stabilized by polyisobutylsuccinic acid-polyamine ester dispersant in mineral oil has been investigated under the boundary and elastohydrodynamic regimes and showed significant improvement in lubrication performance between the steel tribo-pair. The formation of protective thin film by GO was accountable for the reduction in friction and wear [167].

The GO and graphene have been explored as potential additives for a diversified range of lubricating fluids to enhance the tribo-performance by reducing the friction and protecting the contact interfaces [159,168]. The chemically-exfoliated graphene suspended in the ethanol decreased the coefficient of friction and wear by factors of 6 and 4, respectively, between the steel tribo-pair. The Raman spectroscopic analyses of the graphene-lubricated worn track showed the suppressed signature of iron oxide, revealing the passivation of contact interfaces by a conformal protective coating of graphene layers. The tribo-induced deposition of graphene layers not only reduced the tribo-corrosion and wear but also facilitated the shearing, which results in a significant reduction of friction between the steel tribo-pair [168]. The dispersion of exfoliated graphene stabilized by Triton X-100 surfactant has been demonstrated as aqueous-based lubricants for the steel tribo-pair. An aqueous dispersion of graphene (110 µg mL⁻¹) decreased the friction by 81% and wear volume by two orders of magnitude owing to the formation of graphene-based tribo-layer on the sliding contact interfaces. Furthermore, the stabilized dispersion of graphene outperformed the GO dispersion under identical experimental conditions. The lubrication mechanism emphasized the role of surfactant, which facilitates the graphene deposition process on the contact interfaces by adjusting the



Fig. 10. Dispersion of rGO prepared by γ -ray irradiation-based reduction of GO in the DMF. The dispersion of rGO in (a) isopropanol, (b) glycol, (c) DMF, (d) THF, and (e) NMP solvent are shown after two weeks to demonstrate dispersion stability. Reproduced with permission [142].

dispersion wettability of the mating surfaces for superior tribo-performance [160]. The dispersion of 5 μ m-80 surfactant-stabilized graphene in the polyalphaolefin (PAO4) lube oil exhibited significantly improved lubrication properties for textured alloy cast iron surface. The 0.01 wt% dose of graphene in PAO4 could reduce the wear rate of flat and textured surfaces by 50% and 80%, respectively. The enhanced tribo-performance was attributed to the combined effect of micro-dimples on the textured surface and graphene nano-additive. The sheared graphene layers under the sliding contact stress formed a protective thin film of low shear strength. Furthermore, storing of graphene dispersion in the dimple accelerates thin film formation under the tribo-stress, consequently significantly enhanced tribo-performance by graphene dispersion for textured surfaces [169].

Graphene tends to form aggregates driven by cohesive interaction and settle down in the lubricants, which compromised the tribo-performance of graphene-based lubricants. The crumpled graphene balls prepared by rapidly evaporating of aerosol droplets of GO sheets exhibited excellent dispersibility in the PAO4 lube oil; whereas the agglomerations were seen in the dispersion of rGO, carbon black, and graphite particles in the PAO4 lube oil. The dispersion of crumpled graphene balls reduced the friction (30%) and wear coefficient (85%) of lube oil and outperformed the dispersions of rGO, carbon black,

graphite particles, and fully formulated 5W30 lubricant (Fig. 12). The self-dispersion, high mechanical strength, and nanoscale ball bearing-function collectively enhanced the lubrication performance of crumpled graphene balls-based lubricant [170]. The high mechanical strength and inertness of fluorinated graphene promise its potential for tribological applications. The fluorinated graphene prepared by liquid-phase exfoliation of fluorinated graphite as lubricant additive to PAO40 significantly improved the anti-wear property and exhibited better friction stability along with durability of lube oil [171]. The graphene quantum dots (GQDs) as an additive to PAO lube oil showed notably enhanced tribo-performance between the steel tribopair under the boundary lubrication by reducing the friction (80%) and wear (94%). The improved lubrication performance was attributed to the synergistic effect of a densely packed protective tribo thin film formed on the contact interfaces and presence of friction-induced graphene-like debris in the contact area [172]. Table 5 summarizes the various approaches for the preparation of GO, graphene, and rGO along with their application as additives to various lubricating media for enhancement of tribological properties.

The rGO prepared by chemical, thermal, radiation-induced reduction of GO showed excellent potential as a lubricant additive to improve the tribological properties between the engineering surfaces. The synthesis approaches influence the chemical and structural features of rGO. Ramaprabhu et al. have synthesized graphene by solar light-induced exfoliation of graphite oxide. The prepared graphene nano-sheets showed good hydrophobicity and excellent dispersibility in the engine oil. The graphene-based engine oil (0.025 mg mL⁻¹) decreased the friction and wear scar diameter by 80% and 30%, respectively, whereas extreme pressure properties increased by 40% compared to the engine oil. The enhancement of tribo-performance was attributed to topological structure, high mechanical strength of graphene, excellent dispersibility, and the formation of nano-bearing between the steel balls [175]. The direct thermal reduction of GO yielded a lot of wrinkles, folds, holes, and structural defects, which alter the intrinsic features of graphene, including shearing properties under the tribo-stress. The thermal reduction of GO in sulphuric acid produced the rGO (SA-rGO) lamellar structure without folds and wrinkles. The SA-rGO as an additive to PAO6 exhibited outstanding tribo-performance and decreased the coefficient of friction and wear rate by 30% and 75%, respectively [176]. The thermal reduction of graphite oxide in the presence of KOH yielded the highly exfoliated rGO (hrGO), and the

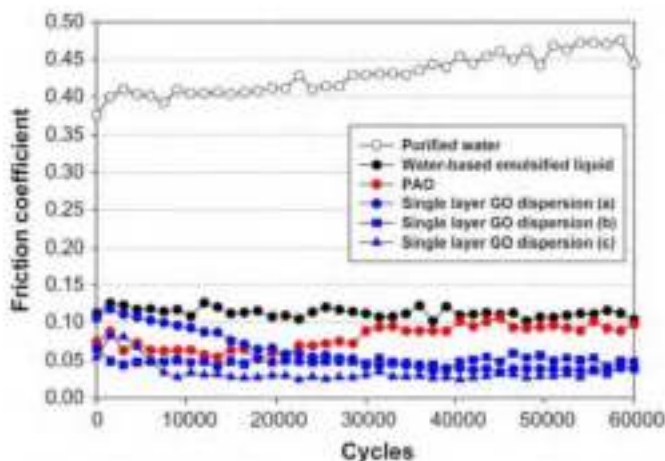


Fig. 11. Variation in the coefficient of friction between the WC ball and stainless steel plate in the presence of water-based lubricating fluids, including dispersion of GO. The friction measurements are carried out using purified water, water-based emulsified liquid, PAO, and three different samples of GO dispersion (a) (b) and (c) as lubricating mediums. Applied load: 1.0 N. Reproduced with permission [151].

Table 4
Preparation of GO dispersion by various approaches and their lubrication performance under variable working conditions viz. applied load, speed, contact geometry, and frictional pair.

No.	Preparation method/description	Lubricant medium	Frictional pair	Load	Contact geometry	Friction reduction	Wear reduction	Reference
1	GO Prepared by modified Hummer's method and microwave irradiation	Water	Steel/WC ball	1.88 N	Ball-on-disk	87.5%	–	[167]
2	GO Prepared by modified Hummer's method	Water	B4C/Steel	10 N	Ball-on-disk	57%	–	[168]
3	Commercially prepared GO, assessed the effect of pH (3.3–9.7)	Water	Steel/Steel	20 N	Ball-on-4-mm-plate	44.4%	17.3% (wear scar width)	[150]
4	Commercially prepared GO, used for anions-terminated self-assembled thin films on aluminum	Water	Steel/Aluminum	2 N	Ball-on-disk	40.6%	79.7% (wear rate)	[154]
5	Selective functionalization of GO, having a variable degree of oxygen functionalities and function	Water	Steel/Steel	20 N	Ball-on-plate	~22%	4.7% (wear rate)	[156]
6	GO Prepared by Hummer's method	Water	Steel/Steel	10 N	Ball-on-plate	~74%	64% (wear scar diameter)	[169]
7	Exfoliation of graphite in water with aid of Triton X-100 (non-ionic surfactant)	Water	Steel/Steel	2–15 N	Ball-on-plate	88.3%	61.4% (wear scar diameter)	[166]
8	Graphene oxide nanosheets with an ionic liquid	Water	Si ₃ N ₄ / Sapphire	2–4 N	Ball-on-disk	Superlubricity	–	[161]
9	Graphene oxide nanosheets and ethanol in aqueous solution	Water	Si ₃ N ₄ /SiO ₂	2–4 N	Ball-on-disk	Superlubricity	–	[162]
10	Phenolic graphene, functionalized graphene and graphene oxide nanosheets were deposited on SiO ₂ substrates to form graphene coatings	Water & glycerol	Si ₃ N ₄ /SiO ₂	1.5–4 N	Ball-on-disk	Superlubricity	–	[163]
11	Graphene oxide and onion-like carbon nano additive	Water	Steel/Steel	2–10 N	Ball-on-disk	~20%	–	[164]
12	Tribological performances of SiO ₂ /graphene combinations as water-based lubricant additives	Water	Steel/high alloy	5 N	Ball-on-disk	48.5%	78% Wear volume	[165]

microstructure of herGO is controlled by the ratio of graphite oxide and KOH. The herGO as an additive to PAO6 (0.5 wt%) decreased the wear scar depth and coefficient of friction by 90% and 44%. The enhanced tribo-performance was attributed to shearing by the book-like layered microstructure of herGO [177].

Besides the chemical structure, the microstructure of rGO sheets also monitors the lubrication properties. The rGO with regular edges as an additive to the hydraulic oil favoured the lubrication by reducing the coefficient of friction (28%) and wear-scar depth (14%), whereas the rGO with irregular edges and wrinkles showed no significant changes in tribo-performance of hydraulic oil. The graphene sheets with regular edges formed the thick and consistent tribo-film under the sliding stress, which avoided the asperities contacts and enhanced the

lubrication properties (Fig. 13). The graphene with irregular edges and wrinkles formed the thin film of discrete patches, leading to direct contact between the asperities of mating surfaces and provides high resistance to shear under the sliding stress; consequently, relatively higher friction and wear [178]. The rGO has been demonstrated as an additive to lubricants for improving the tribological properties by (a) reducing the frictional energy losses, (b) enhancing the lubricant drain time, (c) increasing the wear-resistivity of machinery parts, (d) improving the conductivity to take away the heat from contact interfaces, and (e) decreasing the carbon footprints by fuel conservation. Furthermore, the rGO exhibited no signatures of acute toxicity during in-vitro studies using prokaryotic and eukaryotic cells. These results suggest the safe use of rGO as a lubricant additive not only enhances the tribo-performance

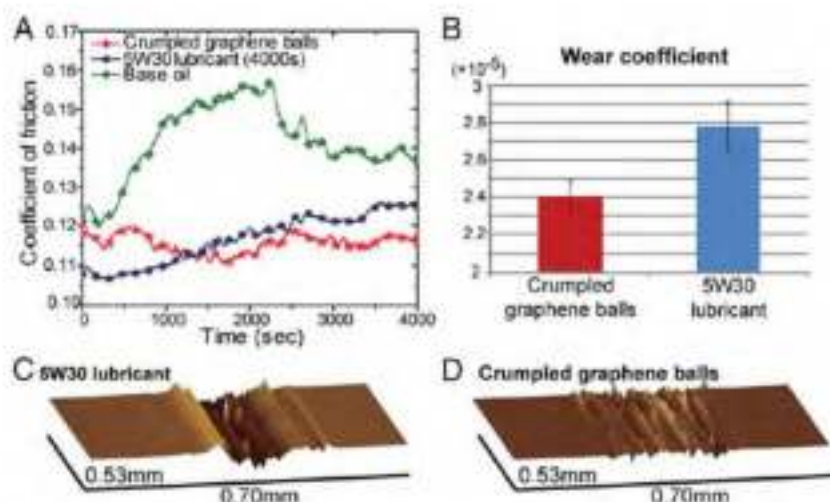


Fig. 12. Comparison of lubrication between fully formulated SW30 lubricant and crumpled graphene balls in PAO4 base oil. The 0.1 wt% dose of crumpled graphene balls in the PAO4 base oil surpassed the fully formulated SW30 lubricant. (A) Coefficient of friction, (B) wear coefficient, and (C and D) 3D profile images of the wear tracks using the SW30 lubricant and crumpled graphene balls in PAO4 base oil. Reproduced with permission [179].

Table 5

Lubrication enhancement by dispersion of GO, graphene, and rGO as additives in lubricating media. The contact geometry, design of graphene-based materials, and lubricating media are presented to understand their roles for enhancement of tribological properties.

No.	Preparation method/brief description	Additive	Lubric medium	Additive dosage	Contact geometry	Friction reduction	Wear reduction	Reference
1	Commercially procured GO nanosheets	GO	40/50 Aviation lubricant	0.25–1.0 wt%	Four ball	14.7%	34% (wear scar diameter)	[166]
2	Modified Hummers' method; polyisobutyroic acid polymerizer was used to prepare the GO dispersion in the oil	GO	SN-150	0.01 wt%	Ball-on-disk	14–18%	12–30% (wear scar diameter)	[167]
3	Chemical exfoliation of highly oriented pyrolytic graphite	Graphene	Ethanol	1.0 mg L ⁻¹	Ball-on-disk	64%	83% (wear volume)	[168]
4	Commercially procured graphene, dispersed in the PNO-4 lubricant with the assistance of Span-80 dispersing agent	Graphene	PNO-4 + 1 wt% Span-80	0.01 wt%	Ball plate (Tai and JSTARD)	~53%	52% (wear rate)	[169]
5	Crumpled graphene (also prepared by capillary compression in rapidly evaporating aerosol droplets of GO sheets)	Crumpled graphene	PNO-4	0.01–0.1 wt%	Ball-on-disk	20%	83% (wear rate)	[170]
6	Hummerized graphene prepared by ultrasound-assisted exfoliation of Hummerized graphite in NMP using EDTA as an intercalation agent	Hummerized graphene	PNO-40	0.1–0.4 mg mL ⁻¹	Ball-on-disk	–	~70% (wear rate)	[171]
7	Graphene quantum dots were prepared by acid treatment and chemical exfoliation of commercial pitch-based carbon fibers	GO/DS	Multialkylamine polypropylenes	2.0 wt%	Ball-on-disk	60%	93% (wear volume)	[172]
8	Graphene sheets were wrapped to form horn-shaped coats with a ball-fulcrum cap (30–50 nm length and 1–5 nm diameter)	Graphene-based nano-horns	Poly-alkylene glycol	0.04–1.0 wt%	Ball-on-disk	18%	30% (wear volume)	[173]
9	Commercially procured graphene	Graphene	Esterefied bio oil	0.5 wt%	Four ball	~35%	~37% (wear scar diameter)	[174]
10	Graphene was prepared by exfoliation of graphite oxide using focused solar light radiation	Graphene	Engine oil	0.012–0.062 mg mL ⁻¹	Four ball	80%	33% (wear scar diameter)	[175]
11	Thermal reduction of GO at 180 °C in sulphuric acid to prepare a lamellar structure of rGO without folds and wrinkles	SA-rGO	PNO-6	0.0–1.0 wt%	Ball-on-disk	30%	73% (wear rate)	[176]
12	Thermal reduction of graphite oxide with a variable mole ratio of EDTA at 700 °C	ED-rGO	PNO-6	0.5 wt%	Ball-on-disk	44%	88% (wear scar depth)	[177]
13	Reduced graphene oxide of different micro-morphologies having regular edges (RG), irregular edges (i-RG), and both irregular edges and wrinkles (i-RWG) prepared by ball-milling of commercially procured rGO	RG, i-RG, i-RWG	Hydraulic oil	0.1–5.0 wt%	Ball-on-disk	27.0%	14.1% (wear depth)	[178]
14	Commercially procured rGO	rGO	Base oil	–	Ball-on-disk	40%	–	[179]
15	Reduced graphene oxide with variable interlayer spacing prepared by chemical (RLG-L) and thermal (RLG-M) reduction of GO; Commercially procured highly oriented multilayer graphene (MLG-8)	RLG-L, RLG-M, MLG-8	PNO-6	0.1–2.0 wt%	Ball-on-disk	~50%	~83% (wear depth)	[180]
16	Mild thermal reduction of GO at 200 °C	MrGO	PNO-6	0.25–2.0 wt%	Ball-on-disk	30%	73.5% (wear depth)	[181]

but also reduces the environmental impact of transportation for sustainable growth of automotive sector [179].

The state near to vanish the friction is known as superlubricity. It is the most efficient way to reduce the losses of frictional energy and has attracted significant attention over the recent past. The origin of ultra-low friction lies in the incommensurability between the rotating graphite layers. The presence of graphite flake between the substrate and the tip elucidated the contact orientation-dependence friction. The two contact orientations governed by rotational angle furnished the high friction, where the graphite flake and the substrate lattices might have perfectly aligned and achieved the commensurate contact, whereas the rest of contact orientations in the intermediate rotational angle

established the incommensurate contact and furnished superlubricity [182]. The superlubricity can be achieved when the graphene is synergistically used with nanodiamond particles and diamond-like carbon. The graphene sheets at a sliding interface wrapped the nanodiamonds under the sliding tribo-stress and yielded the nanoscrolls of the reduced contact area. The slid of such nanoscrolls against the diamond-like carbon surface, achieved the incommensurate contact, and remarkably reduced the coefficient of friction to ~0.004, signifying the macroscopic superlubricity [183]. The graphene nanoscrolls over the graphite surface showed translational and rotational motions between commensurate initial and final states, as deduced from a scanning tunneling microscope. A tip-induced transition of graphene nanoscrolls with underlying

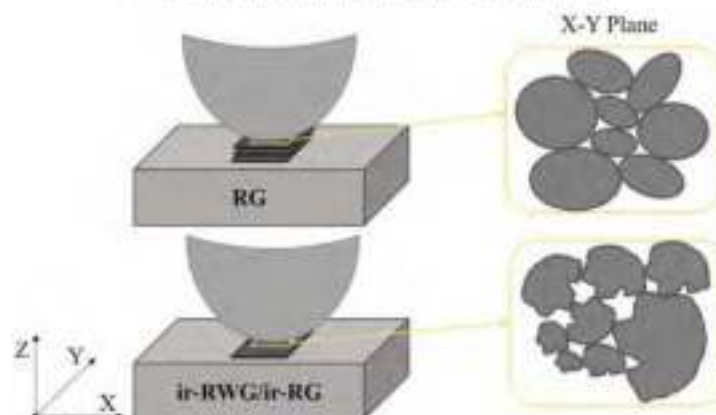


Fig. 11. Lubricating mechanism: emphasizing the role of micro-morphological features of graphene sheets (having regular or irregular edges) in the formation of a protective thin film on contact interface. RG: regular edges graphene; ir-RG: irregular edges graphene; ir-RWG: irregular edges and wrinkles graphene. Reproduced with permission [173].

graphene surface formed a commensurate to an incommensurate (the superlubric state) contact orientation, followed by rapid sliding until another commensurate position is reached [184]. The superlative state of graphene for remarkably low friction is further explored by molecular dynamics and new force field techniques. The unusual sliding mechanisms and temperature dependence of graphene in different contact orientations furnished the superlative state [185].

Recently, Luo group presented a novel method to achieve the liquid-superlubricity under extreme contact pressure by the synergistic effect of 2D materials and liquid molecules [161]. A robust macroscopic liquid-superlubricity ($\mu = 0.005$) under extreme pressure of 600 MPa was recorded by a combination of GO sheets with ionic liquid ([Li][EGD][PF₆]) for Si₃N₄/sapphire tribo-pair. Ionic liquid helps to form a boundary layer at the tribo-interfaces, while the GO sheets showed extremely low shear properties, and the combined effect led to liquid-superlubricity under extreme pressure. The macroscopic superlubricity ($\mu = 0.0037$) is also achieved by a combination of GO nanoflakes with ethylene glycol at the Si₃N₄/SiO₂ interface [162]. Most recently, the graphene coating on SiO₂ substrates was made by using pristine graphene (PG), flake-like graphene (FG), and GO materials for determination of superlubricity characteristics in the presence of an aqueous solution of glycerol [163]. It was noted that PG coating exhibited better tribological properties as compared to FG and GO coatings. Superlubricity ($\mu = 0.004$) was achieved by a combination of glycerol with PG coating was attributed to the formation of graphene nanoflakes tribofilm at the contact zone.

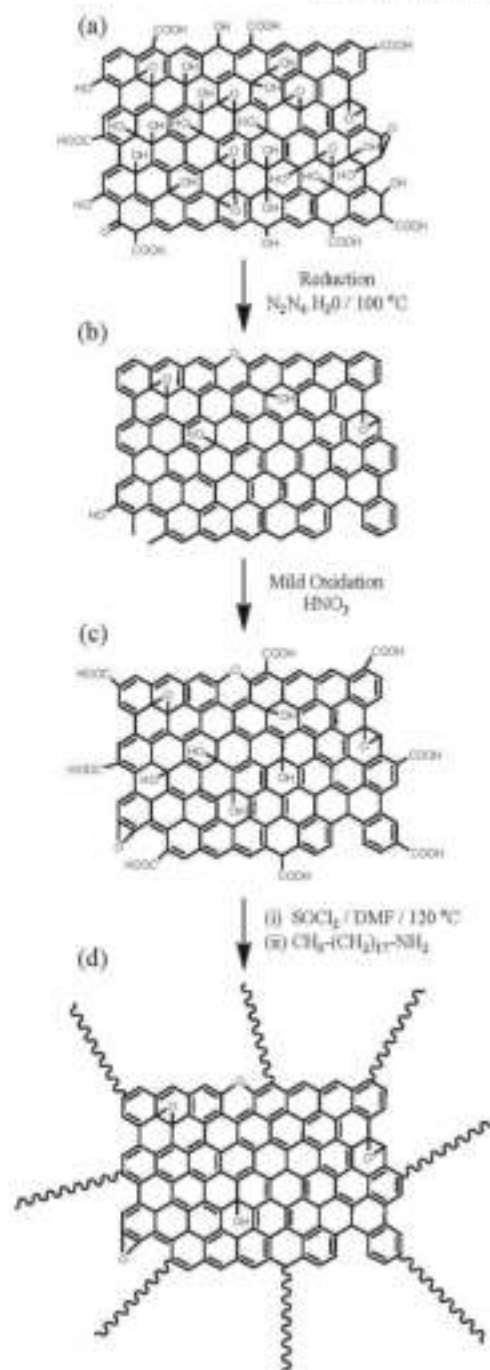
5.2. Chemically functionalized graphene, GO and rGO as lubricant additive

Graphene and rGO as additives to lubricants perform well when they are thoroughly dispersed in the lube oil and exhibited long-term dispersion stability. In the recent past, several approaches have been made to functionalize the GO and rGO to make them dispersible in lubricating oils. The GO, a non-stoichiometric material with ample oxygen functionalities, offers a platform for rich chemistry to occur within the interplanar gallery (basal plane) and along the edges of sheets [33,196,197]. The selective chemical functionalization targeting the specific type of oxygen functionality or reactions with multiple oxygen functionalities make the GO a potential material for several applications [186,187]. In the recent past, numerous types of functionalization based on covalent and non-covalent interactions driven by oxygen functionalities, π -electron-rich structure, and structural defects of GO have been demonstrated. The primary purpose of chemical functionalization of

GO and rGO is to furnish their long-term dispersibility in liquid media or solvents for expanding their potential for various applications, particularly for those domains, where the solution-processed graphene is needed [32,188–190]. Herein, functionalization of GO, rGO, and graphene is critically reviewed emphasizing their dispersion in the lubricating fluids and enhancement of tribological properties by graphene-based additives.

The carboxylic groups in GO were selectively targeted for grafting of alkyamines via amide linkage using the thionyl chloride as a coupling reagent. The long alkyl chains of octadecylamine-functionalized GO (GO-ODA) furnished cohesive interaction with hexadecane; consequently, the GO-ODA is nicely dispersed in hexadecane. The minute dose of GO-ODA (0.06 mg mL⁻¹) decreased the coefficient of friction and wear scar diameter of hexadecane by 26% and 98%, respectively. The enhanced tribo-performance was attributed to uninterrupted supply of GO-ODA to contact interfaces of steel tribo-pair [10]. The presence of oxygen functionalities viz. hydroxyl, ether, epoxy, etc., particularly in the basal plane of alkyated GO decreased the modulus and mechanical strength [32,161]. Thus, the GO-ODA as an additive couldn't show significant improvement in the wear (98% only).

The rGO prepared by chemical reduction exhibits the residual oxygen functionalities. The structural defects distributed across the sheet of rGO and the edges are prone to be oxidized into the carboxylic acid using mild nitric acid. The carboxylic groups in partially oxidized rGO were targeted for covalent coupling with ODA via an amide linkage to yield the alkyated-rGO (Fig. 14). The stable dispersion of rGO-ODA in the 10W40 engine oil significantly reduced the coefficient of friction (36%) and wear scar diameter (35%) between the steel balls. Furthermore, the tribo-performance of rGO-ODA was found to be better than the corresponding GO-ODA. The presence of oxygen functionalities in the GO-ODA diminished the shearing under the tribo-stress; consequently, poor performance compared to rGO-ODA [144]. The atomic force microscopic studies further revealed that the presence of oxygen functionalities in graphene oxide compromised the mechanical properties and the effective Young's modulus is estimated to be lower than the graphene [191]. Sohaei et al. have demonstrated simultaneous reduction of GO and grafting of ODA without using any reducing and coupling agent [192]. The surface functionalization of GO with ODA was facilitated by nucleophilic addition reaction between the amino group of ODA and epoxy group of GO [103]. The long alkyl chain of ODA grafted on the GO generates hydrophobicity and showed excellent compatibility with heavy paraffin oil. The 1 wt% dose of highly dispersible GO-ODA significantly decreased the coefficient of friction (~81%) compared to



that of graphene is the heavy paraffin oil under the mean Hertzian contact pressure of 0.9 MPa [192].

Recently, Khatri et al. revealed the participation of phenolic, carboxylic, epoxy, and other oxygen functionalities for interactions with alkylamines. The refluxing of aqueous dispersion of GO with ethanolic solution of alkylamine proficiently grafts the alkylamines via nucleophilic addition interaction with basal plane epoxy groups, hydrogen linkages with the oxygen functionalities, and columbic interactions with weak acidic sites like carboxylic and phenolic groups as demonstrated in Fig. 15 [194]. The facile approach for grafting of ODA in the basal plane and simultaneous reduction of oxygen functionalities in GO not only restore the graphitic characteristics but also make the GO-ODA nicely dispersible in the 10W40 lube oil (Fig. 16). The minute dosage (0.02 mg.mL⁻¹) of GO-ODA notably decreased the friction and wear of fully formulated 10W40 engine oil. The analysis of worn surfaces, particularly the Raman spectroscopic results suggested the deposition of sheared graphene lamellae on the contact interfaces under the sliding stress. The deposited graphene-based thin-film not only protects the tribo-pairs against the undesirable events of wear but also facilitates the shearing to reduce the friction [195]. The carboxylic groups in GO show immense potential for the covalent functionalization with variable organic moieties to convert water-miscible GO to oil-compatible graphene-based materials for liquid lubricant applications. The oleic diethanolamide borate (ODMB) has been selectively grafted on GO via an amide linkage. The resultant GO-ODMB exhibited excellent dispersibility in the SN 500 lube base oil. The coefficient of friction and wear scar diameter were decreased by 38.4% and 42%, respectively, using the 0.02 wt% dosage of GO-ODMB between the steel tribo-pair [196]. The dodecyl chains were introduced on the surface of GO via click chemistry-based multi-step covalent functionalization, which includes the preparation of alkyne-functionalized GO via amide linkage with carboxylic groups of GO and then click coupling between alkyne-grafted GO and azidodecane. The resultant material (GO-CuAAC) as an additive to petroleum lube oil enhanced the tribological properties by reducing the friction (16%) and wear (30%) [197].

The non-covalent functionalization of rGO with poly(ethylene glycol) via γ -radiolysis enhanced the dispersion of resultant material (rGO-PEG) in the PEG. The high energy γ -radiation exposure formed the PEG radicals, which are reacted with rGO through hydrogen bonding between the oxygen atoms of PEG molecules and residual hydroxyl groups of rGO [142]. The γ -radiolysis-based functionalization of rGO expanded the interlamellar spacing by intercalation of PEG molecules. The stable dispersion of rGO-PEG reduced the coefficient of friction and wear rate by 38% and 55%, respectively compared to the un-irradiated rGO sample in the PEG lubricating media. The rGO-PEG with expanded interlamellar spacing easily sheared under the sliding tribo-stress between the contact interfaces and reduced the friction [Fig. 17]. Furthermore, rGO nanosheets are adsorbed on the contact interfaces driven by mechano-chemical energy and formed the graphene-based tribo-film, which not only protected the contact interfaces but also acted as a solid lamellar lubricant to ease the sliding of contact interfaces under the tribo-stress [143]. The PEGylated graphene (Gr-PEG) having amino-terminal group has been explored as nano-additive for enhancement of tribological properties of aqueous-based lubricants. The amino-containing PEG molecules were grafted on carboxylic groups-enriched graphene via amidation process. The Gr-PEG exhibited good dispersibility in water and notably decreased the friction (39%) and wear rate (81%) compared to the pure water. The enhanced lubrication performance of Gr-PEG-based aqueous lubricant was attributed to the formation of fluid adhesive film and graphene-based protective tribo-

Fig. 14. Schematic models of (a) GO and (b) rGO, illustrating the distribution of various oxygen functionalities in their basal plane and along the edges. (c) The mild oxidation of rGO selectively introduced carboxylic groups at the edges and defects sites of rGO sheets. (d) ODA-functionalized rGO, prepared through amide linkage between the carboxylic sites of rGO and ODA. Reproduced with permission [144].

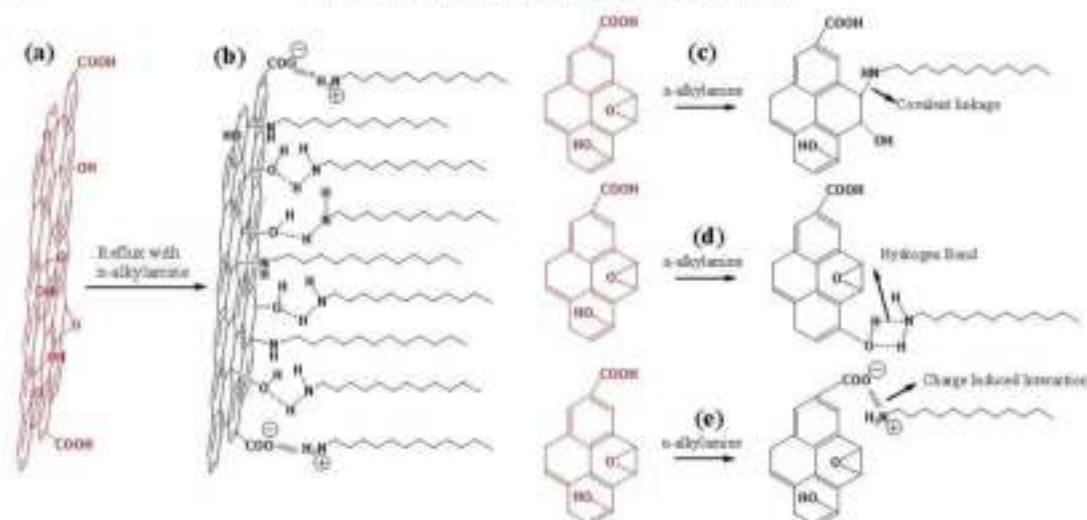


Fig. 15. (a) Schematic model of GO, (b) n-alkylamine grafted on the GO via multiple interaction pathways with basal plane oxygen functionalities, (c) Covalent linkage of n-alkylamine via an epoxy ring, (d) Hydrogen bonding between the hydroxyl groups of GO and the amino groups of n-alkylamines, and (e) charge-induced interaction between weakly acidic sites (carboxylic and phenolic) of GO and n-alkylamines. Reproduced with permission [104].



Fig. 16. Digital images of (a) 32W43 lube oil and (b–k) dispersion of GO-ODA in 32W43 lube oil (0.04 mg/mL) as a function of time. Time for each image is noted on the respective dispersion sample bottle. Reproduced with permission [106].

film, which prevented direct contact between the friction pairs [196]. The scope of graphene as an additive to water-based lubricant was further expanded by covalent functionalization of GO with amino-terminated block copolymer using the 3-(tri(hydroxysilyl))-1-propane sulfonic acid linker. The resultant liquid-like graphene showed excellent dispersibility in the water and as an additive it decreased the friction and wear rate by 53% and 91%, respectively compared to the water. The remarkable lubrication properties of functionalized graphene dispersion in the water were attributed to the formation of graphene-based tribo-film on the contact interfaces driven by electrostatic adsorption under the tribo-stress and molecular rearrangement of liquid-like graphene [199].

The increasing importance of environment-friendly green lubricants has attracted great attention towards novel additives based on green chemistry. In this context, ionic liquids have been identified as promising candidates as lubricants and additives to reduce the friction and wear efficiently, while keeping the environment clean and green [200–203]. The wide range of cations and anions makes ionic liquids highly designable molecules for targeted engineering surfaces and lubricants. In the recent past, the hybrids of graphene and ionic liquids have shown great potential as lubricant additives for enhancement of tribological properties [175–177]. The commercially procured graphene was sonicated and then stirred with 1-butyl-3-methylimidazolium iodide

[BMIM-I] ionic liquid to obtain the graphene-ionic liquid (Gr-IL) hybrid. The resultant Gr-IL hybrid served as an effective lubricant for aluminum-steel tribo pair and substantially reduced the friction and the wear [204]. The simultaneous reduction by KOH and functionalized of GO with imidazolium-based ionic liquids yielded the MGO-ILs via reaction of epoxy group of GO with imidazolium ionic liquids and the non-covalent linkages viz. π - π stacking, carbon- π , and van der Waals interactions [146]. The MGO-ILs exhibited good dispersibility in alkylated cyclopentane lube base oil and significantly enhanced the tribo-performance by reducing the friction (27%) and wear volume (74%). The worn surface analysis revealed the formation of ionic liquids-containing graphene-rich tribo-film, which prevented the direct contact between the sliding surfaces and improved the friction and wear properties [146].

Graphene-ionic liquids hybrid nanomaterials prepared by chemical grafting of imidazolium ring on the graphene nanosheets using organosilane as a bifunctional chemical linker has been a promising approach to prepare stable dispersion of graphene in PSG-200 synthetic lube base oil. Furthermore, this approach has a merit to replace the associated anion via metathesis reaction using bis(salicylate)borate (BSb), oleate (OL), and hexafluorophosphate (PF₆) anions. The Gr-BSb, Gr-OL, and Gr-PF₆ hybrid nanomaterials (Fig. 18) as additives to synthetic lube base oil remarkably improved the lubrication properties

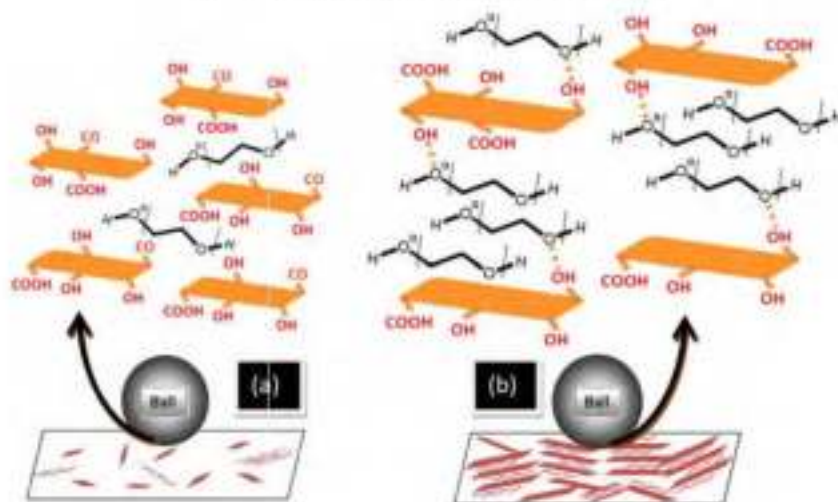


Fig. 12. Lubrication mechanism using (a) un-irradiated rGO with PEG, and (b) γ -irradiated rGO-PEG complex. Intercalated PEG molecules in rGO driven by hydrogen bonding facilitate the sliding between steel tribo-pair under tribo-stress. Reproduced with permission [143].

by reducing the friction and wear between steel tribo-pair. Besides the graphene, the anionic structure of ionic liquids also played a significant role and governed the lubrication properties. A tribo-induced thin film composed of Cr-ionic liquids and its tribo-chemical products with contact interfaces lubricated the sliding interfaces. The [Bsc] anion-based Cr-ionic liquid hybrid exhibited the lowest friction, whereas oleate analogous showed the smallest wear [147]. Table 6 summarizes the preparation of chemically functionalized GO, rGO, and graphene along with their application as additives to various lubricating media for enhancement of tribological properties.

The intriguing properties of graphene-based materials, particularly high surface area, good thermal conductivity, low shear, and excellent mechanical strength, which are governed by their chemical and structural features, make them lubricious additives to liquid lubricants. The ultralow thickness of graphene-based materials, along with their good dispersibility, facilitates their uninterrupted supply to tribo-interfaces and forms the graphene-based thin film, which reduced the friction and wear. The high surface area of graphene covers the larger contact area of tribo-interfaces by stress-induced thin film formation. The graphene thin film prevents the direct contact of the sliding surfaces and eases the sliding because of interplanar low shear strength [211]. The graphene lamellae are surface-active, particularly under the tribo-stress, and deposited on the contact interfaces of tribo-pair [212]. The deposited graphene sheets not only facilitate the sliding along the basal plane of the graphene but also the deposited sheets furnish the slip with graphene lamellae in the liquid dispersion; as a result, it reduced the friction and protected the tribo-pair against the wear. The structural defects, wrinkles, surface functionalization, number of lamellae in graphene, roughness, and mechanical properties of tribo-interfaces, and interlamellar spacing in graphene-based materials influence the frictional properties [144,213–215]. Graphene effectively offers excellent anti-friction and antiwear properties. However, graphene is prone to agglomerate in lube oils because of cohesive interaction between their sheets, which adversely influences the frictional properties. The use of dispersant or chemical functionalization of graphene makes them nicely dispersed in variable lube base oil and effectively offers the inherent low frictional properties between the tribo-pair. The uninterrupted supply of chemically functionalized graphene to contact interfaces and formation of tribo-induced thin film facilitates the sliding

because of low shear strength and avoiding direct contact between the tribo-interfaces, which result in significant improvement in lubrication properties.

The formation of graphene-based tribo thin-film governs by several factors viz. affinity of graphene with tribo-interfaces, applied load, the dosage of graphene in the lubricating fluid, dispersion stability, effect of other surface-active additives in the lube, structure and chemical composition of graphene, and so on. The wearing of materials under the tribo-stress forms the pristine surface of the metal, which is considered to attract the graphene nanosheets. The delaminated graphene sheets under the sheared contact are gradually deposited on the sliding interfaces. As the tribo-sliding goes on, the graphene nanosheets in the lubricants enter the contact interfaces and easily sheared with deposited graphene-based tribo thin-film and reduced the friction [195]. Biswas et al. have revealed that deposition and removal of molecular lamellae of such nanostructural layered materials-based tribo thin film on the sliding interfaces are continuous processes [216].

The microscopic mapping of contact interfaces by Raman spectroscopy suggested the heterogeneous nature of graphene-based tribo thin film on the contact interfaces. Fig. 19 explicitly revealed the discrete patches of the graphene-based thin film on the contact interfaces and also suggested the heterogeneity of graphene thin film within the individual patches, and it was attributed to the roughness of the tribo-interfaces and load distribution [126].

5.3. Graphene-based nanocomposites and hybrid materials as lubricant additive

Graphene-based nanocomposites and hybrid materials show rapid progress for their applications as a lubricant additive for enhancement of tribological properties. The synergistic effect among two or more components in the nanocomposites and hybrid materials along with their compatibility with lubricating media make them promising materials for lubricant applications. Chen et al. synthesized the supercritical CO_2 -assisted metal nanoparticles (Au, Ni, Cu)-decorated-GO nanocomposites (Sc-M/GO) with regular size distribution (Au and Cu: 5–10 nm; Ni: <5 nm) of metal nanoparticles [217–219]. The Sc-Au/GO nanocomposite (0.3 wt%) as an additive to PAD lube base oil reduced the coefficient

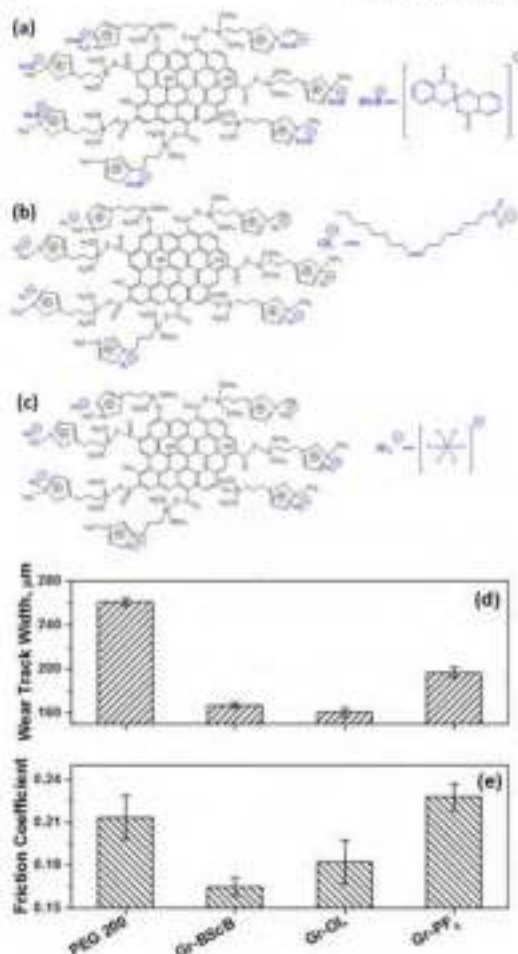


Fig. 18. Representative chemical structures of (a) Gr-B5O₈, (b) Gr-OL, and (c) Gr-PF₆ hybrid nanomaterials. (d) Wear track width and (e) average coefficient of friction of steel disc lubricated with PEO 200 and Gr-B5 hybrid nanomaterials (Gr-B5O₈, Gr-OL, and Gr-PF₆) tested in the PEO 200 (Dose: 0.02 mg/cm²). Reproduced with permission [147].

of friction (33.8%) and wear rate (73%) between steel tribo-pair. The Se-Au/GO nanocomposite (in lubricating oil) is believed to enter the contact interfaces and could eventually accumulate in pits/grooves; consequently, the contact surfaces became smooth and flat. The deposited Se-Au/GO nanocomposite gradually covered the mating surfaces (Fig. 20) and avoided the direct contact between the friction pair, which is attributed for a significant reduction in friction and enhanced anti-wear ability. The GO nanosheets are exfoliated by Au nanoparticles under the tribo-stress and ease the interlamellar sliding, which can effectively reduce the friction in the boundary lubrication regime [217].

Recently, graphene-based composites with metal and metal oxide nanomaterials have attracted considerable attention because of their improved tribological properties, particularly under the boundary lubrication regime. The Cu and CuO as spherical nanoparticles and nanorods of variable aspect ratio, respectively, act as nano-bearings between the mating surfaces and furnish rolling-based mechanism to enhance the tribological properties [220,221]. The rGO-Cu nanocomposite with

well-distributed Cu nanoparticles of 20 nm size as an additive to paraffin oil showed low friction and improved the load-bearing and antiwear properties compared to oil mixed with a blend of individual Cu nanoparticles and rGO nanosheets [222]. The microscopic results based on worn surface analysis revealed the formation of a composite thin film which could prevent not only the direct contact between the mating surfaces but also bear the load. Moreover, the formation of nano-bearings between the contact interfaces contributes a significant role in the reduction of friction and wear. The oleic acid capped Cu/rGO nanocomposites as an additive (0.5 wt%) to PAO oil decreased the friction (45%) and wear (53%) between steel tribo-pair. The EDS results of lubricated surfaces revealed the formation of Cu/rGO nanocomposites-based tribo film, which improved the tribological properties [223]. The α -Fe₂O₃/GO nanocomposite could easily enter the contact zone between the mating surfaces and yielded a physical tribo thin film, which could bear the load and improve the antiwear property [224]. Therefore, α -Fe₂O₃/GO nanocomposite having α -Fe₂O₃ nanorods of 3–5 nm diameter and 15–30 nm length as an additive to paraffin oil exhibited a remarkable reduction in friction (64%) and improved the antiwear properties (76%). The enhanced tribological features were attributed to the combination of rolling effects and the formation of tribo thin films on the mating surfaces by α -Fe₂O₃/GO nanocomposite. The graphene decorated with ceria nanoparticles (CeO₂/Gr) as an additive to paraffin oil offers considerably improved antiwear properties under a high load condition [225]. The improved tribological properties by CeO₂/Gr nanocomposite were attributed to synergistic antiwear effect and friction reduction by CeO₂ nanoparticles and graphene sheets. The CeO₂ nanoparticles not only prevented the tribo-interfaces against the wear but also inhibited the restacking of graphene. Simultaneously graphene provides an ideal carrier to load the CeO₂ nanoparticles without aggregation. The LaF₃-GO hybrid as a lubricant additive to water enhanced the tribological properties by forming the protective tribochemical thin film over the contact interfaces of steel tribo-pair [226]. Table 7 summarizes various types of graphene-based nanocomposites and hybrid materials as additives to lubricating media for enhancement of tribological properties. The contact geometry, the dosage of graphene-based nanocomposites, and lubricating media are outlined to understand their roles for improvement of tribological properties.

The MX₂ (M = Mo, W; X = S, Se), the dichalcogenides exhibit molecular lamellar structure, where the weak van der Waals interaction connects the MX₂ lamellae and furnish the intracrystalline slip under the sliding stress. As a result, MX₂ nanostructured layered materials are gaining increasing attention as lubricating materials for tribological applications [21,244,245]. The synergistic effects between MoS₂/MoSe₂ and graphene in their nanocomposites significantly improved the lubrication properties under boundary lubrication [231–233]. The nano-sized MoS₂ deposited on graphene could enter the contact zone of mating surfaces and formed the stable and durable boundary thin film, which reduced the friction and wear. The XPS of worn surfaces revealed the formation of tribo-induced thin film based on MoS₂/GO nanocomposites [233]. The FeS₂/pyrrole/rGO nanocomposite prepared by hydrothermal method increased the load-bearing capacity of tribo-pair. The FeS₂/rGO as an additive to paraffin oil decreased the friction by ~63%. The improved tribological properties were attributed to the synergistic effects of rGO and FeS₂ layers (Fig. 21). The FeS₂/rGO composites not only filled the micro-gaps of mating surfaces but also formed a homogeneous lubricating film under the high sliding contact pressure, which bears the high load, reduces the friction, and prevents the direct contacts between tribo-interfaces. Moreover, the delaminated FeS₂ sheets under the tribo-stress facilitate the sliding because of the planar structure of rGO sheets between the mating surfaces [234]. The grafting of polyacrylamide, polyacrylates, and polyaniline on GO not only improved the dispersibility in lubricating media but also enhanced the tribological properties [237–239]. Polyacrylamide-functionalized GO (rGO-PAM) prepared by microwave-assisted surface-initiated redox polymerization

Table 4

Lubricative enhancement by dispersion of chemically functionalized GO, rGO, and graphene as additives in various lubricating media. Contact geometry, dosage of graphene-based materials, and lubricating media are presented to understand their roles for enhancement of tribological properties.

No.	Preparation method/ brief description	Additive	Lube medium	Additive dosage	Contact geometry	Friction reduction	Wear reduction	Reference
1	Selective grafting of allylamine ($C_n = 8, 12, 18$) targeting carboxylic groups of GO via amide linkage using thionyl chloride as a coupling agent	GO-ODA	Dimedureane	0.04–0.06 mg ml^{-1}	Bear ball	30%	95 (wear scar diameter)	[166]
2	Functionalization of carboxylic groups-enriched rGO with octadecylamine via amide linkage using thionyl chloride as a coupling agent	rGO-ODA	10W40 oil	0.01–0.05 mg ml^{-1}	Bear ball	30%	35% (wear scar diameter)	[144]
3	Simultaneous chemical reduction and surface functionalization of GO with octadecylamine targeting basal plane epoxide functionalities	GO-ODA	Paraffin oil	1 wt%	Pin/Ball on-disk	~61%	–	[163]
4	Functionalization of basal plane functionalities (epoxide, hydroxyl) of GO surface with ODA	GO-ODA	10W40 oil	0.01–0.06 mg ml^{-1}	Ball-on-disk	23%	23% (wear track width)	[185]
5	Selective grafting of oleic diethanolamine borate on carboxylic groups of GO surface using thionyl chloride as a coupling agent	GO-ODAB	SH-500 oil	0.01–0.04 wt%	Bear ball	38.4%	42.0% (wear scar diameter)	[186]
6	Surface functionalization of carboxylic groups with azidoacetate via click chemistry using thionyl chloride as a coupling agent	GO-CuAAC	800 N oil	0.005–0.01% wt %	Bear ball	16%	30% (wear scar diameter)	[167]
7	γ -Radiolysis-based functionalization of rGO with poly (ethylene glycol)	rGO-PEG	PEG-200	0.02–2.0 mg ml^{-1}	Ball-on-disk	38%	35% (wear scar)	[143]
8	Covalent grafting of amino containing PEG onto the surface of carboxylated graphene using a volatile 2,4-diaminotoluene bridging agent	Gr-PEG	Water	0.005–0.1 wt%	Ball-on-disk	54.2%	81.2% (wear rate)	[188]
9	The sulfonation of GO surface using 3-(trihydroxyethyl)-1-propane sulfonic acid followed by grafting of amino terminated block copolymer	GO-F	Water	10–100 mg ml^{-1}	Ball-on-disk	53%	91% (wear rate)	[189]
10	Graphene-ionic liquid-based hybrid prepared by exfoliation and stirring of graphene with ionic liquid	Gr-ILs	Ionic liquid	–	Ball-on-disk	~67%	–	[204]
11	Modified GO obtained by simultaneous reduction and functionalization of GO with amide-ionic liquids in presence of KOH	MGD-ILs	Multiple allylated cyclopentane	0.1 wt%	Ball-on-disk	17%	74% (wear volume)	[146]
12	Commercially prepared graphene was thoroughly blended with ionic liquid (with 1-methylimidazolium triafluoroborate)	Gr-IL	10W40 oil	0.005 wt%	Ball-on-disk	73%	–	[205]
13	Carboxylated rGO prepared by mild oxidation of rGO was used for selective grafting of ionic liquids based on covalent interaction	Gr-ILs	PEG-200	0.01–0.05 mg ml^{-1}	Ball-on-disk	23%	38.3% (wear track width)	[147]
14	GO simultaneously reduced and functionalized with 3,3'-di-(tri-butyl-4-hydroxyphenyl)ethyne using 3-aminopropyl trimethoxysilane as a chemical linker	Gr-DHBM	SH-150	6.2–0.6 mg ml^{-1}	Bear ball	40%	17% (wear scar diameter)	[188]
15	Chemically rGO functionalized with oleic acid via heating the blend of rGO and oleic acid	rGO-OA	PWD 9	0.01–0.10 wt%	Bear ball	17%	14% (wear scar diameter)	[206]
16	Graphene reduced with a mixture of oleic and oleic acid (mole ratio 1:3)	Gr-OA/SA	SH-330 oil	0.015–0.10% wt %	Bear ball	~33%	~64%	[207]
17	Surface functionalization of rGO with PEG-200	rGO-PEG	PEG-200	0.02–1.0 mg ml^{-1}	Ball-on-disk	78%	90%	[208]
18	Commercially prepared graphene modified with Nitrogen and ammonia plasma	Gr- N_2 , Gr- NH_3	Polysiloxane oil	0.05 wt%	Cylinder-on-glass	–	94% coefficient; 62% specimens	[209]
19	Superhigh exfoliation reduced graphene oxide prepared by thermal reduction of GO with the assistance of KOH	SRGO	Base oil	0.1–1.5 wt%	Ball-on-disk	70%	60% wear volume	[310]

of acrylamide with functionalized of GO showed excellent dispersibility in water, and substantially improved the antiwear properties (37%) and decreased the friction (55%) [207].

The composite of GO with carboxylic-functionalized carbon nanotube (GO/MWCNTs–COOH), as an aqueous-based additive improved the lubrication performance between the steel tribopair [246]. As demonstrated in Fig. 22, the GO lamellae could easily delaminate because of shear force between the mating surfaces and transferred to contact interfaces. The deposited GO nanosheets prevent the direct contact between the steel balls and bear the load to reduce the friction. Besides, the MWCNTs–COOH in composite material functions as micro-bearing to convert sliding friction into rolling friction between the mating bodies. The synergistic effects extended by GO/MWCNTs–COOH composite under the tribo-stress promises its potential not only for aqueous-based lubrication but also opens new directions for designing of novel hybrid materials and composites for tribological applications. The ZnO-decorated rGO/MoS₂ (Gr-MS-Zn) hybrid as an additive to engine oil

improved the tribological performance by significant reduction of wear volume (87%) and the friction (37%). The enhanced lubrication performance was attributed to low shear strength, arising from weak interfacial interaction between the incommensurately stacked graphene and MoS₂ and their uninterrupted supply to tribo-interfaces because of stable dispersion [247].

6. Summary and outlook

Remarkable mechanical, electronic, optical, and structural properties of graphene has propelled enormous interest for fundamental studies to a diversified range of applications. The weak van der Waals interaction between atomic-thick lamellae of graphene, excellent mechanical strength, remarkable thermal conductivity, and high surface area make graphene a potential candidate for tribological applications to reduce the friction and wear of engineering surfaces. Over the recent past, the use of graphene, chemically-functionalized graphene, and

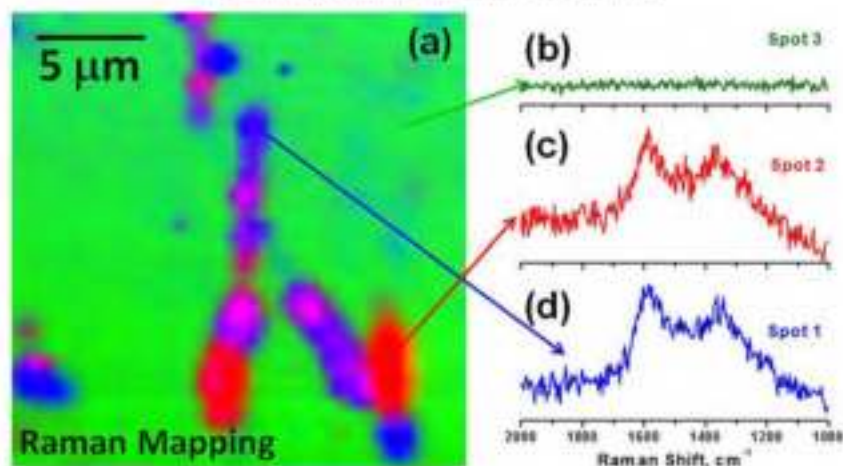


Fig. 19. (a) Raman mapping of the worn scar on the steel ball after the lubrication test using ultrasonically functionalized graphene blended in the 50–150 mineral lube base oil. The Raman spectra corresponding to different color pixels in the Raman map revealed the heterogeneity of graphene-based thin film deposition on the worn scar. (b) Raman spectra shows no signature of D and G bands. (c)–(d) The red and blue patches exhibit D and G bands, signifying the deposition of graphene. Reproduced with permission [136]. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

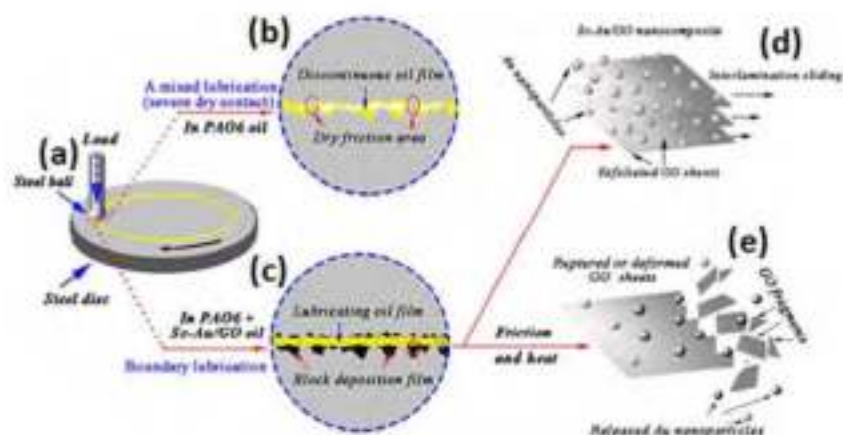


Fig. 20. Schematic demonstration of (a) sliding contact geometry and lubrication model using (b) PAO6 oil and (c) Si-Au/GO nanocomposite in PAO6 lubricating oil. (d) The asperitic sliding by Si-Au/GO nanocomposite under the ultra-stress improved tribological properties between the coating surfaces. (e) Some of the GO sheets may captured by friction force and the Au nanoparticles are exposed and released. Reproduced with permission [137].

graphene-based composites as additives to variable lubricating medium has increased exponentially. Although graphene-based materials furnish significantly improved tribological properties of variable lubricants, however, their long-term dispersibility has been an important challenge for applications in the mainstream of industrial lubricants. Herein, a thorough and critical review on preparation, structural models, and dispersibility of graphene, GO, rGO, chemically-functionalized graphene, and graphene-based composites are presented. The functionalisation and stabilisation of GO, rGO, and graphene by variable chemical and physical approaches are discussed for enhancement of their dispersion in water, organic solvents, and variable lube oils. The dispersion of graphene-based materials in aqueous,

organic solvents and lubricating oils are found to be governed by variable interactive forces viz, the van der Waals interaction, π - π interaction, charge induced interaction, Coulombic interaction, and so on.

The graphene, GO, rGO, chemically-functionalized graphene, and graphene-based composites as additive to lubricating media including water and industrial lube oils furnish significantly improved tribological properties by reducing the friction, protecting the contact interfaces against the undesirable events of wear, dissipating the heat from tribo-interfaces, and mitigating the corrosion by forming the protecting thin film. The weak van der Waals interaction between graphene lamellae provide low-resistance to shear and facilitate the sliding events, consequently lowering of the

Table 2

Lubricative enhancement by graphene-based nanocomposites as additives in various lubricating media. The contact geometry, dosage, and lubricating media are presented in underlined and their roles for enhancement of tribological properties.

No.	Preparation method/ brief description	Additive	Lube medium	Additive dosage	Contact geometry	Friction reduction	Wear reduction	Reference
1	Supercritical CO ₂ -assisted deposition of Au nanoparticles on GO nanosheets	Se-Au/GO	PAG6	0.1 wt%	Ball-on-disk	33.6%	72.6% wear rate	[217]
2	Supercritical CO ₂ -assisted deposition of Ni nanoparticles on GO nanosheets	Se-Ni/GO	Paraffin oil	0.08 wt%	Four-ball	32%	43% wear scar diameter	[218]
3	Supercritical CO ₂ -assisted deposition of Cu nanoparticles on GO nanosheets	Se-Cu/GO	Paraffin oil	0.05 wt%	Four-ball	27%	52.7% wear scar diameter	[219]
4	rGO-Cu nanocomposite prepared by chemical reduction of CuSO ₄ and GO dispersion	rGO-Cu	Liquid paraffin	0.5 wt%	Ball plate	~50%	78% wear scar diameter	[220]
5	Oxalic acid capped Cu/GO composite prepared by single-step chemical reduction of CuSO ₄ and GO using hydrazine hydrate	Cu/GO	PAG	0.5 wt%	Four-ball	40%	53% wear scar diameter	[221]
6	Decorative of α -Fe ₂ O ₃ nanosheet on GO nanosheets by a facile hydrolysis route	α -Fe ₂ O ₃ /GO composite	Paraffin oil	0.5 wt%	Ball plate	~40%	~70% wear scar diameter	[224]
7	Carbon nanotube reinforced graphene prepared by hydrothermal reduction of GO and Gd(NO ₃) ₃	CNTs/G	Paraffin oil	0.06 wt%	Ball disk	47.6%	98.3% wear rate	[225]
8	LaF ₃ nanoparticles-dispersed GO prepared by a solution processed chemical approach using ammonium fluoride and lithium fluoride in GO dispersion	LaF ₃ /GO	Water	1.5 wt%	Four-ball	34%	16% wear scar diameter	[226]
9	rGO/ZnO nanocomposite prepared by hydrothermal reduction of ZnOCl ₂ and GO using hydrazine hydrate reducing agent	rGO/ZnO	Paraffin oil	0.06 wt%	Ball disk	50%	66% wear rate	[227]
10	Biohybrid GO nano-hybrid prepared by covalent coupling between GO and 3-glycidyloxypropyltrimethoxysilane functionalized boehmite	GO-GPTS-AIOOH	VHVO Lube oil	0.03 mg mL ⁻¹	Ball disk Four-ball	14% (Ball-disk)	28% wear scar diameter	[228]
11	Cu impregnated multi-layer graphene prepared by ultrasound-assisted processing of ammonia dispersion of GO with [Cu(NO ₃) ₂ ·6H ₂ O] followed by calcination of resultant product at 400 °C	MG-Cu	Engine oil	0.5–1.0 wt%	Block ring	43%	63% wear volume	[229]
12	Calcium borate/GO composite synthesized by hydrothermal processing of borax, calcium nitrate, and graphene oxide	CB/GO	PAG	0.5–3.0 wt%	Four-ball	46%	52% wear scar diameter	[230]
13	Nanocomposite of chemically functionalized GO with MoS ₂	FeGO-MoS ₂	Group II SBO N oil	0.8 wt%	Four-ball	15.8%	26% wear scar diameter	[231]
14	Hydrothermal synthesis of MoS ₂ nanoflowers on rGO	MoS ₂ /rGO	Paraffin oil	0.1–5.0 wt%	Ball plate	~40%	75% wear width	[232]
15	Nanitized MoS ₂ deposited graphene prepared by chemical processing of (NH ₄) ₂ MoS ₄ and graphene dispersion followed by annealing at 800 °C	MoS ₂ /G	PAG	0.1–0.3 wt%	Ball disk	70%	54% wear volume	[233]
16	Fe ₃ O ₄ /poly(rGO) prepared by hydrothermal method using the solution-processed product of FeCl ₃ , NaOH, polyvinylpyrrolidone, and sulfur powder in GO dispersion	Fe ₃ O ₄ /GO	Paraffin oil	7.0 wt%	Ball-disk	~67%	–	[234]
17	Phosphorus-graphene hybrid prepared by ball milling of graphene and red phosphorus	P-G	PAG	1.0 wt%	Ball disk	12%	86% wear volume	[235]
18	TiO ₂ -reinforced boron and nitrogen co-doped microwave-induced reduced graphene oxide	TiO ₂ -B-N-MRG	Paraffin oil	0.150 wt%	Four-ball	25.4%	50.8% wear scar diameter	[236]
19	Polyacrylamide-grafted functionalized GO prepared by microwave-assisted surface-initiated radical polymerization of acrylamide with functionalized GO	FGO-PAM	Water	0.8 wt%	Four-ball	53%	37% wear scar diameter	[237]
20	GO-poly(L-lysine) nanocomposite prepared by surface-initiated atom transfer radical polymerization	GOPLA	Polylol	0.04 mg mL ⁻¹	Four-ball	42%	14% wear scar diameter	[238]
21	PANI-GO hybrid prepared by peroxide-assisted processing of chemical processing of aniline and GO	PANI-GO	Paraffin oil	0.01 wt%	Four-ball	44%	18% wear scar diameter	[239]
22	Zinc/graphene core-shell prepared by coating of graphene on ZnO nanoparticle via ionic linker	ZnO@graphene	Spindle oil base oil	0.25–2 wt%	Four-ball	55%	40% wear scar diameter	[240]
23	Ag/graphene nanocomposite prepared by one-step laser irradiation strategy	1-Ag/GO	Liquid paraffin	0.1 wt%	Four-ball	40%	16% wear scar diameter	[241]
24	Tributylmethylphosphonium hexafluorophosphate modified graphene gel prepared by a simple mechanical grinding method	[TBMH ⁺][PF ₆ ⁻]-G	ISO N Base oil S	0.25–0.75 wt%	Ball-plate	–	85% wear volume	[242]
25	Ultrasound-assisted blending of 2D & 0D particles and the 3D graphene nanofiber	2D & 0D/3D graphene	PAGSA oil	0.01–3 wt%	Ball-Sheet	32%	63% wear rate	[243]

friction. The microscopic and spectroscopic analyses of lubricated surfaces and worn scars revealed the formation of a graphene-based protective thin film on the contact interfaces under the tribo-stress, which minimizes the wear. The formation of

graphene-based tribo-film governs by several factors viz. affinity of graphene with tribo-interfaces, applied load, the dosage of graphene in the lubricating fluid, dispersion stability, effect of

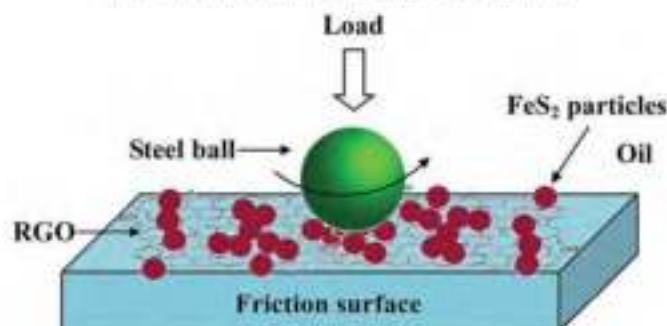


Fig. 21. Lubrication mechanism demonstrating the role of FeS_2/RGO heterojunction under the tribo-stress for a reduction in friction and wear. Reproduced with permission [234].

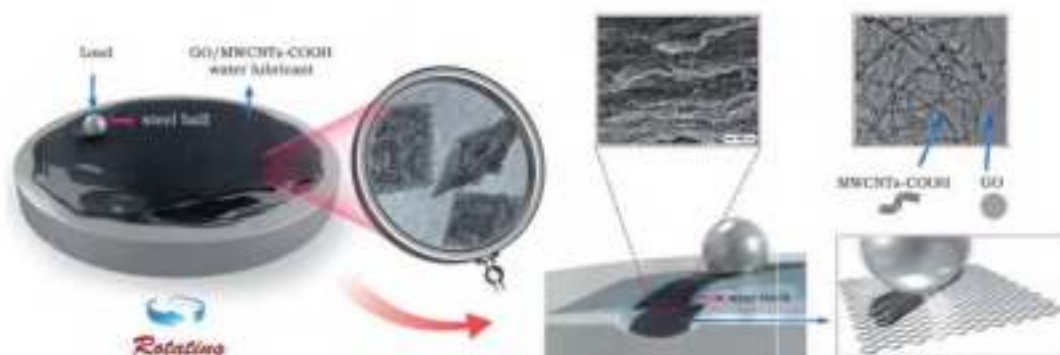


Fig. 22. Lubrication mechanism of GO/MWCNTs-COOH hybrid as an additive to water under the sliding contact between the steel tribopair. Reproduced with permission [246].

other surface-active additives in the lube, structure and chemical composition of graphene, and so on.

The fundamental and laboratory-scale experiments promise the potential of graphene-based materials as new generation additive for enhancement of tribological properties of industrial lubricants such as engine oils, cutting fluids, metal-finishing, and hydraulic oils. Nonetheless, it carries several challenges before graphene-based additives can be used for mainstream industrial lubricants. Over the last decade, several efforts have been directed for functionalization and stabilization of graphene-based materials to achieve long-term stable dispersion in the lubricating media. However, the poor dispersibility of graphene-based materials, particularly, under the high temperature and pressure (unavoidable under tribo-experiments) promotes the agglomeration and compromises the tribo-performance. The role of graphene in the formation of the tribo-film under variable tribo-stress and the lubrication mechanism including the interaction of graphene with variable tribo-interfaces are yet to be understood precisely, which is very important to design the contact interfaces and dosage of graphene in industrial lubes. The findings to these major challenges through scientific interventions can make graphene-based materials as novel additives for industrial lubricants.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Generation of cold white light by using energy transfer process in single phase Ce³⁺/Tb³⁺ co-doped CaSrAl₂SiO₇ phosphor: Dr. S. S. Sharma



1. Introduction

Phosphors that are the luminescent materials are widely utilized in our daily life, some of the best applications of phosphors are color television screen, fluorescent lamps, scintillators, dosimeters, X-ray storage, screen intensifying phosphors, sensors, LEDs, watch dials, laser materials etc. [1]. The white light sources based on light emitting diode have so many valuable advantages as compare to conventional incandescent lamps. White LEDs have longer lifetime, better reliability, environmentally characteristics and higher efficiency which provide significant contribution in power consumption and pollution from fossil fuel power plants [2]. In recent years, researchers have been concentrating on investigation of single composition white-light-emitting phosphors that are excited by UV-LED to prevent some difficulties like the cross-color, instability of color, inequity, and expensive cost problem [3]. The process of co-doping of sensitizer and activator into one host matrix is one of the best ways to fabricate a single-phased white light-emitting material by utilizing the principle of energy transfer from sensitizer to activator. Now a days the white light can be obtained from co-doping of divalent and trivalent rare earth elements in a single phase host. White light emission was investigated in Eu²⁺, Mn²⁺ co-doped Ca₂MgSi₂(PO₄)₆ [4], Ce³⁺, Tb³⁺ co-doped Ba₂LaBO₃Cl [5] and

Gd and Y [6], Ce³⁺, Dy³⁺ co-doped Ca₂(P₂O₇)₂ and Dy³⁺, Sm³⁺ co-doped La₂GaO₄ [7] systems [8]. Due to predominant ⁵D₃ → ⁷F₅ transition [9,10] and of Tb³⁺ ion it is the best candidate for green luminescence among all rare earth ions. However, within the 4f configuration of the Tb³⁺ ion, the electric dipole transition is both spin and parity forbidden, which results in the weak absorption intensity in the near UV region and the narrow width. Therefore a suitable sensitizer is need for the Tb³⁺ activated phosphors. From the very beginning to recent years, Ce³⁺ ion is known to be an excellent sensitizer for Tb³⁺ ion. Ce³⁺ ion transfer a fraction of its energy to Tb³⁺ ion depending upon its lowest 5d electronic state and lowest absorption and emission levels associated with 4f → 5d transitions [9,10,11].

In the present work we synthesized a novel single phase CaSrAl₂SiO₇:Ce³⁺,Tb³⁺ phosphor by solid state reaction (SSR) method for generation of cool white-light emission. Photoluminescence (PL) behaviour shows that the present co-doped phosphor covers the entire range of visible region which can create cool white emission which was resulted from the energy transfer from Ce³⁺ to Tb³⁺ ions. To the best of our knowledge, luminescence properties and energy transfer between Ce³⁺ and Tb³⁺ in CaSrAl₂SiO₇ host lattice have not been reported so far. PL spectrum of Ce³⁺,Tb³⁺ co-doped sample was compared with PL spectra Ce³⁺ and Tb³⁺ single doped sample. Preparation of powder samples was

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S. Sharma et al.

Optics and Laser Technology 135 (2021) 106602

confirmed by X-ray diffraction (XRD) analysis and average particle size was estimated from transmission electron microscopy (TEM).

2. Synthesis and experimental

In the present investigation powder samples $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ were synthesized by SSR method at 1300°C . The preferred raw materials were calcium carbonate (CaCO_3 , analytical pure 99.90%), strontium carbonate (SrCO_3 , analytical pure 99.90%), aluminum oxide (Al_2O_3 , analytical pure ~ 98%), silicon dioxide (SiO_2 , analytical pure 99.99%), ceric oxide (CeO_2 , analytical pure 99.99%) and terbium oxide (Tb_2O_3 , analytical pure 99.99%). The synthesis route can be understood from the flow chart as shown in Fig. 1.

The luminescent properties of prepared phosphors were analysed by using a RF-5301PC Shimadzu spectrofluorometer equipped with a xenon lamp (150 W) as an excitation source. XRD data was obtained with Cu-K α radiation using a D2 phase Bruker diffractometer. Average particle size was determined by a JEOL/JEM 2100 transmission electron microscope using LaB_6 as electron source which is available at SAIL Kochi, India.

3. Results and discussion

3.1. XRD analysis

The phase composition and purity of the synthesized samples were measured by XRD method. The XRD patterns of $\text{Ce}^{3+}/\text{Tb}^{3+}$ single or co-doped $\text{CaSrAl}_2\text{SiO}_7$ samples are depicted in Fig. 2. All the reflections of as-prepared samples are in good agreement with the $\text{CaSrAl}_2\text{SiO}_7$ host and can be indexed to the JCPDS card (No. 26-0327). XRD result of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (1.0 mol%), $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ (1.0 mol%), Tb^{3+} (5.0 mol%) phosphors indicates formation of single phase as no additional peaks from other phases were observed, which shows that the doping of small amount of $\text{Ce}^{3+}/\text{Tb}^{3+}$ ions does not cause any significant influence on the crystal structure of $\text{CaSrAl}_2\text{SiO}_7$ host. $\text{CaSrAl}_2\text{SiO}_7$ has a tetragonal crystal structure with a space group of $I4_1/a$.

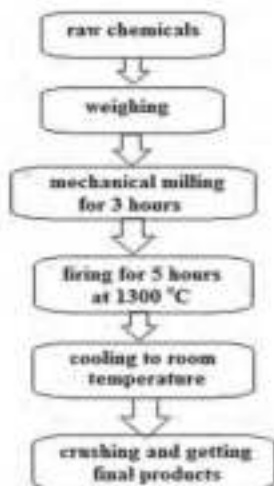


Fig. 1. Synthesis route of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}/\text{Tb}^{3+}$ phosphors by SSR method.

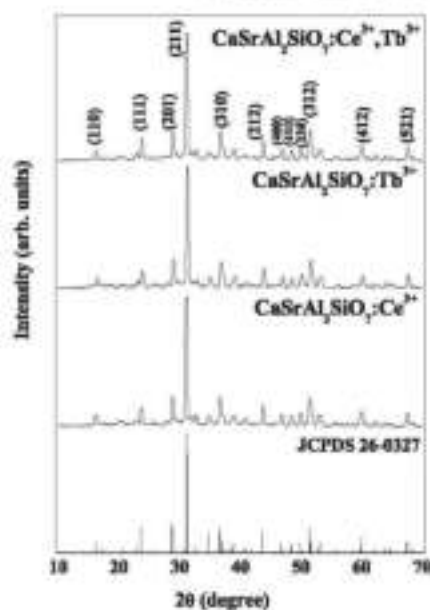


Fig. 2. XRD patterns of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}/\text{Tb}^{3+}$ phosphors.

3.2. TEM analysis

A TEM utilizes energetic electrons to provide topographical, morphological, compositional and crystallographic information on samples. The TEM images allow observers to view samples on a molecular level, making possible to analyse structure and texture. The TEM images of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (1.0 mol%), $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ (1.0 mol%), Tb^{3+} (5.0 mol%) phosphors at the magnifications 100 nm are shown in Fig. 3 (a), 3 (b) and 3 (c), respectively along with their Selected Area Electron Diffraction (SAED) patterns. The agglomeration of powder particles was observed in TEM images this was because of high temperature treatment. The average particle size of entire phosphors is found in nanometre range between 40 and 100 nm. The main diffraction rings in SAED patterns are associated with the diffraction planes in XRD pattern.

3.3. Photoluminescence (PL) spectroscopy

3.3.1. PL of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphor

Trivalent Cerium (Ce^{3+}) and terbium (Tb^{3+}) doped phosphors are attractive materials for a broad range of applications. Cerium ions can act as sensitizer in most of luminescent materials. Influence of cerium and terbium concentration on photoluminescence of singly doped $\text{CaSrAl}_2\text{SiO}_7$ phosphor was first seen. Figs. 4a and 4b exhibit PL excitation (PLE) and PL emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphor for various concentrations of Ce^{3+} ions, i.e., $x = 0.1, 0.3, 0.5, 1.0, 2.0, 3.0$ and 4.0 mol%, respectively. Optimum luminescence was observed for 1.0 mol% of Ce^{3+} ions and then quenching of emission arises. Three excitation peaks were observed with 415 nm emission for entire series; the excitation peaks were due to $4f \rightarrow 5d$ transition and situated at 250, 290 and 345 nm as shown in inset of Fig. 4a. The strongest excitation at 345 nm was particularly selected to record emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphors. Concentration dependent PL emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ sample is displayed in Fig. 4b. There was no change in position of PL emission peaks for various concentrations of Ce^{3+} ions.

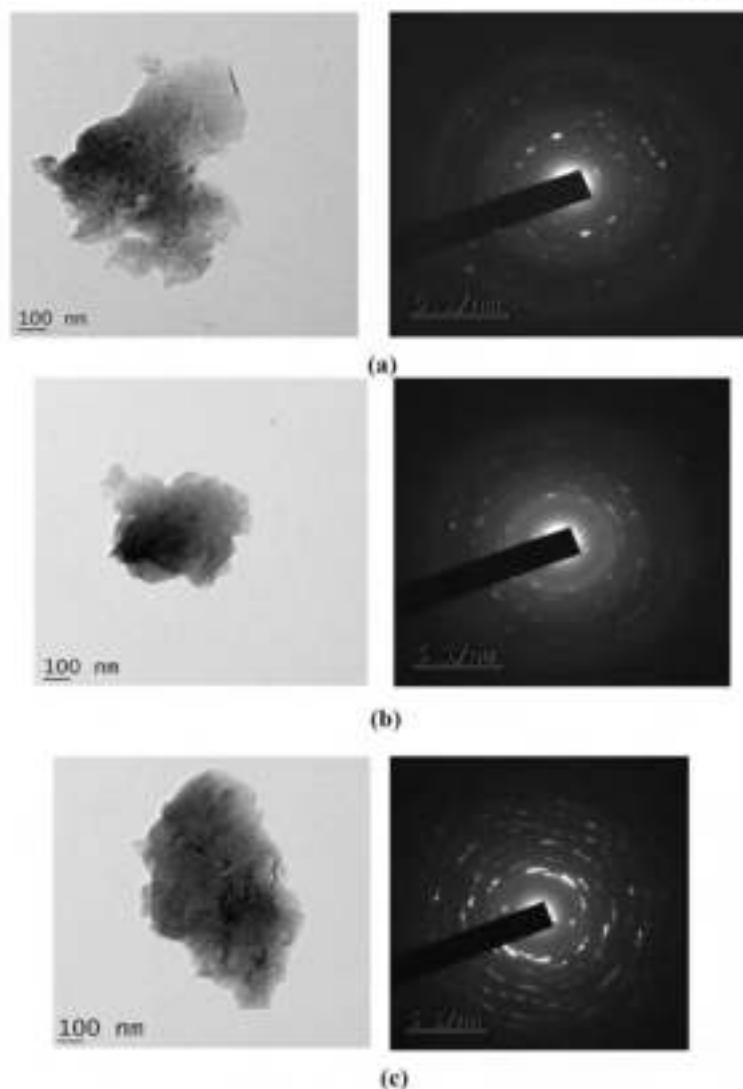


Fig. 3. TEM images of (a) $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$, (b) $\text{CaSrAl}_2\text{SiO}_7:\text{Th}^{3+}$ and (c) $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Th}^{3+}$ phosphors.

As prepared $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ phosphors show a broad emission peak in the blue wavelength region located at around 415 nm which is ascribed to $5d(^1\text{D}_{3/2}) \rightarrow 4f(^1\text{F}_3)$ transition of Ce^{3+} . A graph between Ce^{3+} concentration and luminescence intensity is shown in the inset of Fig. 4b, which shows PL emission intensity increases with rise in Ce^{3+} concentration and maximum intensity is observed at 1.0 mol% of Ce^{3+} after that quenching in luminescence emission occurs.

3.3.2. PL of $\text{CaSrAl}_2\text{SiO}_7:\text{Th}^{3+}$ phosphor

Fig. 5a displays PLE spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Th}^{3+}$ phosphor doped with various Th^{3+} concentrations such as 1.0, 3.0, 5.0, 7.0 and 10.0 mol %. Optimum excitation spectrum was monitored for $\text{CaSrAl}_2\text{SiO}_7:\text{Th}^{3+}$ (5.0 mol%) phosphor that is represented in the inset of Fig. 5a. PLE

spectra for entire series (monitored with 545 nm emission wavelength) have similar fashion containing a broad absorption band in 220–280 nm region, centred at around 240 nm and with three small excitation peaks at around 253 nm, 275 nm and 483 nm that are due to $^7\text{F}_6 \rightarrow ^5\text{D}_3$, $^7\text{F}_6 \rightarrow ^5\text{L}_{10}$ and $^7\text{F}_6 \rightarrow ^5\text{D}_4$ transitions, respectively [12]. The absorption band at 240 nm is assigned to $4f^8(^7\text{F}_6) \rightarrow 4f^85d^1$ transition of Th^{3+} ions in the $\text{CaSrAl}_2\text{SiO}_7$ host composition, which is a spin-allowed transition in accordance with Laporte's rule. Th^{3+} -doped materials necessarily have strong absorption band in the 200–300 nm range due to $4f \rightarrow 5d$ transition. Emission spectra of $\text{CaSrAl}_2\text{SiO}_7$ doped with $\times$ mol% of Th^{3+} (where $\times$ equals to 1.0–10.0 mol%) are shown in Fig. 5b. A series of characteristic luminescence signals of Th^{3+} was appeared in the emission spectra within the range 250–600 nm when samples were excited at

S. Sharma et al.

Optics and Laser Technology 135 (2021) 106602

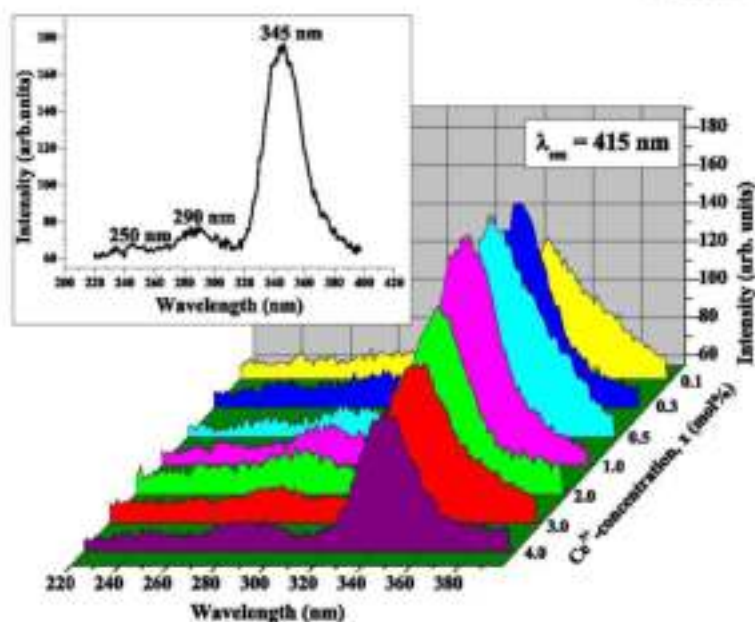


Fig. 4a. PL excitation spectra of $\text{Ca}_{0.9}\text{Al}_2\text{SiO}_5:\text{Ce}^{3+}$ phosphor at different mol% concentration of Ce^{3+} (a.u.). Inset shows PL excitation spectrum of $\text{Ca}_{0.9}\text{Al}_2\text{SiO}_5:\text{Ce}^{3+}$ (1.0 mol%) phosphor.

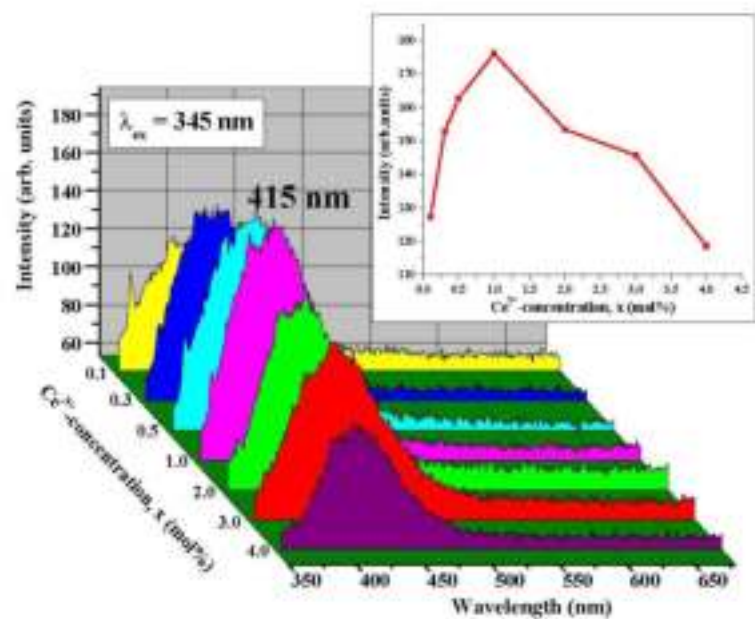


Fig. 4b. PL emission spectra of $\text{Ca}_{0.9}\text{Al}_2\text{SiO}_5:\text{Ce}^{3+}$ phosphor at different mol% concentration of Ce^{3+} (a.u.). Inset shows emission intensity as a function of Ce^{3+} concentration, x.

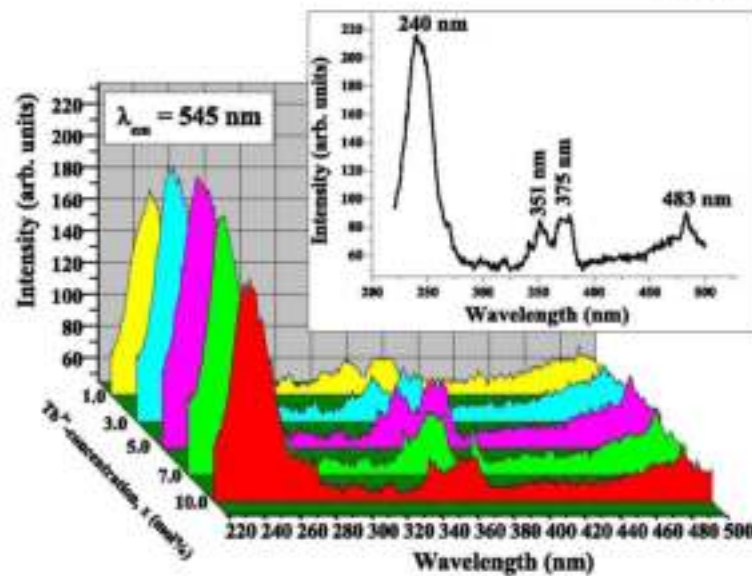


Fig. 5a. PL excitation spectra of $\text{GaInAl}_3\text{SiO}_7:\text{Th}^{3+}$ phosphor at different mol% concentration of Th^{3+} ions. Inset shows PL excitation spectrum of $\text{GaInAl}_3\text{ACl}_7:\text{Th}^{3+}$ (5.0 mol%) phosphor.

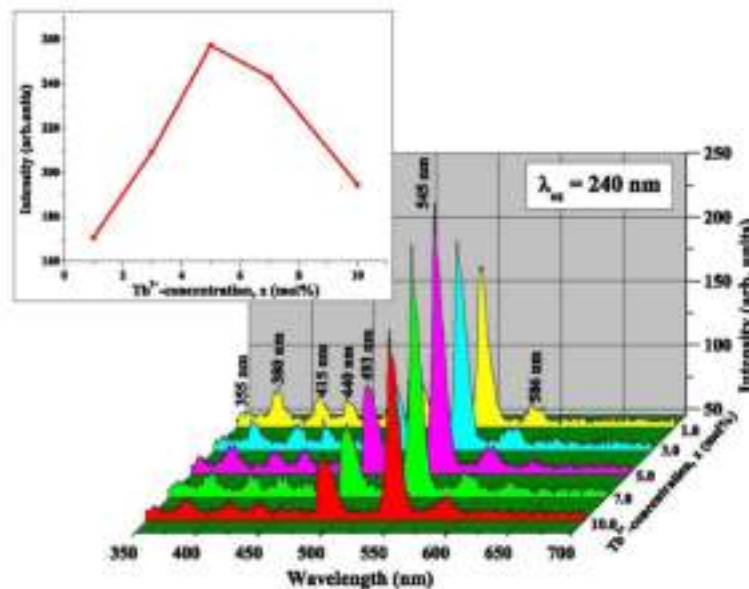


Fig. 5b. PL emission spectra of $\text{GaInAl}_3\text{SiO}_7:\text{Th}^{3+}$ phosphor at different mol% concentration of Th^{3+} ions. Inset shows emission intensity (545 nm) as a function of Th^{3+} -concentration x .

240 nm. The emission spectra showed few sharp emission bands that correspond to the $^5\text{D}_4 \rightarrow ^7\text{F}_J$ intraconfigurational transitions of Th^{3+} ions (Th green luminescence) [13]. The emission spectrum of the Th^{3+} activator normally presents two typical sets of intraconfigurational transitions from the $^5\text{D}_4 \rightarrow ^7\text{F}_J$ ($J = 3 - 6$, 620 - 465 nm) and $^5\text{D}_3 \rightarrow ^7\text{F}_J$ ($J = 3 - 6$, 465 -

375 nm) transitions, which result in green and blue emissions, respectively. The dominant set usually corresponds to $^5\text{D}_4 \rightarrow ^7\text{F}_J$ transitions (green set), whereas the $^5\text{D}_3 \rightarrow ^7\text{F}_J$ transitions (blue set) are difficult to obtain as a primary emission because of the depopulation of the $^5\text{D}_3$ state [14]. The emission of $^5\text{D}_3 \rightarrow ^7\text{F}_J$ transitions are obtained in the

present case, although their intensity was weak but that could be identified. An examination of luminescence spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Th}^{3+}$ phosphor notified that it comprises of two UV emission bands (at 235 and 280 nm), three blue emission bands (at 415, 440 and 493 nm) and one green emission band (at 545 nm) and one yellow band (at 586 nm). The sharp emission peaks originated from $4f \rightarrow 4f$ transition of Th^{3+} ions. The emission at 355 nm is due to host emission. The emission band at 380 nm and blue set of emitting peaks monitored at 415/440 nm are because of $^3\text{D}_3 \rightarrow ^3\text{F}_{6,5,4}$ transition; emission band at 493 nm (blue), 545 nm (green) and 586 nm (yellow) represents the transitions $^3\text{D}_4 \rightarrow ^3\text{F}_{6,5,4}$ respectively [15]. The transition $^3\text{D}_4 \rightarrow ^3\text{F}_6$ (493 nm) is an allowed electric dipole transition since $\Delta J = 2$ while $^3\text{D}_4 \rightarrow ^3\text{F}_5$ (545 nm) is allowed magnetic dipole transition as $\Delta J = 1$. Most intense peak was situated 545 nm which is responsible for green luminescence under UV (240 nm) excitation. Inset in Fig. 5b indicates that PL emission intensity enhanced with increase in Th^{3+} concentration and reached an optimum value at a Th^{3+} -concentration of 5.0 mol%. For inclusion exceeds 5.0 mol%, the luminescence was distributed, this behaviour comes from concentration quenching phenomenon. Concentration quenching originates from non-radiative energy transfer between dopant ions. This behaviour implies that the Th^{3+} aggregates may be formed at high concentration of Th^{3+} , these aggregates act as the trapping centers and dissipate absorbed energy non-radiatively and reduces PL emission [16]. The increase in concentration of Th^{3+} ion enhances the interaction between them. The cross relaxation processes are likely to occur in $^3\text{D}_4 \rightarrow ^3\text{D}_3$ and $^3\text{F}_6 \rightarrow ^3\text{F}_5$ or $^3\text{D}_4 \rightarrow ^3\text{D}_3$ and $^3\text{F}_6 \rightarrow ^3\text{F}_5$ and then the luminescence emission is quenched [17].

3.3.3. PL of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Th}^{3+}$ phosphor

A series of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Th}^{3+}$ phosphor was prepared by conventional solid method by fixing Ce^{3+} -concentration x at 1.0 mol% and changing Th^{3+} -concentration y ($y = 1.0, 3.0, 5.0, 7.0$ and 10.0 mol%). Fig. 4a shows comparative PLE spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(1.0 \text{ mol}\%), \text{Th}^{3+}(y \text{ mol}\%)$ with varying Th^{3+} -concentration. Excitation spectra were measured with constant 545 nm emission of Th^{3+} ions. Inset in the same figure shows excitation spectrum of the optimized $\text{Ce}^{3+}, \text{Th}^{3+}$ co-doped phosphor, i.e., $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(1.0 \text{ mol}\%), \text{Th}^{3+}(5.0 \text{ mol}\%)$. This co-

doped phosphor has first excitation peak at 235 nm, second and the most intense peak at around 345 nm and third very weak peak at 482 nm; the third peak could not be observed for low Th^{3+} -concentration that means for $y = 1.0$ and 3.0 mol%. The presence of weak blue peak at 482 nm signifies that $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(1.0 \text{ mol}\%), \text{Th}^{3+}(y \text{ mol}\%)$ phosphors with $y \geq 5.0$ mol% can weakly excited by blue light. Excitation bands at around 345 nm is the characteristic of $4f \rightarrow 5d$ transition of Ce^{3+} and a peak around 235 nm and 482 nm were seen for Th^{3+} singly doped sample (see Fig. 5a). The absorption band at 235 and 482 nm are due to $4f^0(^1\text{F}_3) \rightarrow 4f^15d^1(^3\text{I}, ^3\text{I}')$ and $^3\text{F}_6 \rightarrow ^3\text{D}_4$ [19] transition of Th^{3+} ions. It is to be noted that spectral distribution of excitation spectra of $\text{Ce}^{3+}, \text{Th}^{3+}$ co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphors was unchanged while changing Th^{3+} concentration y .

Illustration of PL emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(1.0 \text{ mol}\%), \text{Th}^{3+}(y = 1.0, 3.0, 5.0, 7.0 \text{ and } 10.0 \text{ mol}\%)$ phosphors is shown in Fig. 4b. Upon excitation of Ce^{3+} band at 345 nm, the PL spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(1.0 \text{ mol}\%), \text{Th}^{3+}$ consist of the $5d(^3\text{D}_{3,2}) \rightarrow 4f(^3\text{F}_{6,5})$ blue emission broad band centred at 410 nm assigned to Ce^{3+} ions while the blue emissions at 484 and 494 nm ($^3\text{D}_4 \rightarrow ^3\text{F}_6$); and green and yellow emissions situated at 545 and 589 nm ($^3\text{D}_4 \rightarrow ^3\text{F}_{5,4}$) corresponding to Th^{3+} ions. Inset in Fig. 4b shows the variation of the emission intensities of Ce^{3+} and Th^{3+} ions in co-doped sample, versus Th^{3+} concentration. Although the amount of the Ce^{3+} ions is fixed, their emission intensity gradually decreases along with the increase of the concentration of the Th^{3+} ions. The overall emission intensity decreases with increasing Th^{3+} concentration while the yellow emission intensities increases firstly and then reaches a maximum at the Th^{3+} concentration of 5.0 mol% and decreased gradually with a further increase of Th^{3+} concentration. It is clear that the addition of Th^{3+} ions in the 1.0 mol% Ce^{3+} activated $\text{CaSrAl}_2\text{SiO}_7$ sample results a decrease of the overall Ce^{3+} emission [9]. The result indicates that a lot of Th^{3+} ions as acceptors accelerate energy diffusion of donors (Ce^{3+}) before they can fluoresce, which speeds up the average transfer rate of $\text{Ce}^{3+} \rightarrow \text{Th}^{3+}$ [10,20]. According to Dexter's energy transfer theory, the calculation of efficiency principally based on the spectral overlap between the emission spectra of the sensitizers and the absorption spectra of the activators [21]. Co-doped phosphor possesses two types of energy transfer pathways: nonradiative and

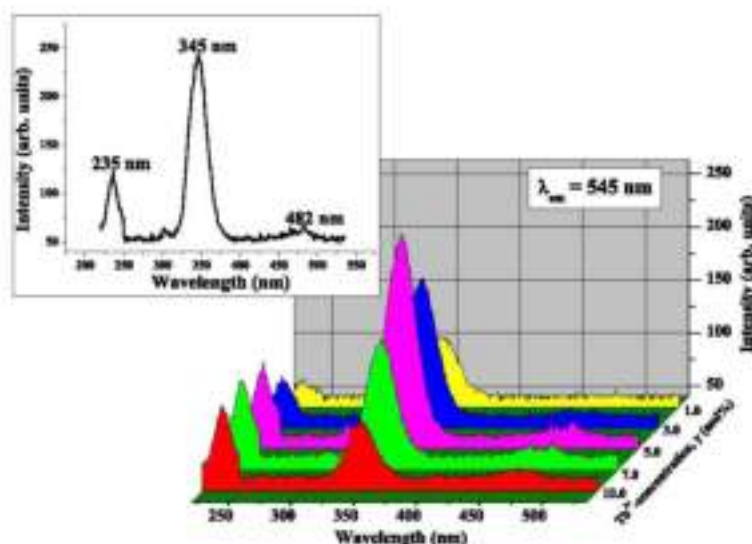


Fig. 4a. PL excitation spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(1.0 \text{ mol}\%), \text{Th}^{3+y}$ phosphor at different mol% concentration of Th^{3+} ions. Inset shows PL excitation spectrum of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(1.0 \text{ mol}\%), \text{Th}^{3+}(5.0 \text{ mol}\%)$ phosphor.

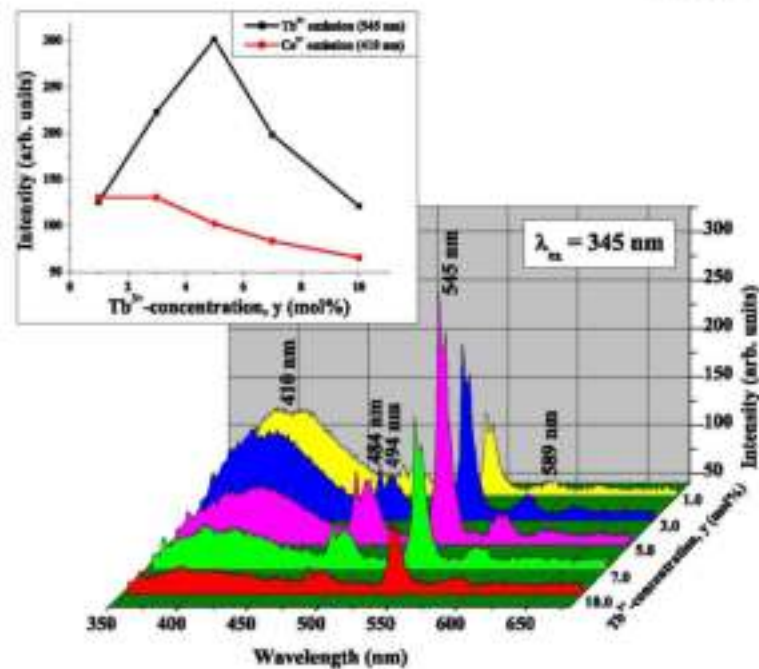


Fig. 6b. PL emission spectra of $\text{CaSrAl}_2\text{SiO}_6:\text{Ce}^{3+}(1.0 \text{ mol}\%)\text{Tb}^{3+}$ phosphor at different mol% concentration of Tb^{3+} ion. Inset shows emission intensity (410 nm and 545 nm) as a function of Tb^{3+} concentration y .

radiative. The nonradiative transfer mechanism is complex because of the unknown multipolar interaction and exchange interaction [22]. Furthermore, in the radiative energy transfer process, the broad

emission ($5d \rightarrow 4f$) of the sensitizers, which is allowed by the Laporte parity selection rules, is affected greatly by the crystal field and the covalency of many inorganic hosts [23]. Therefore the sensitized

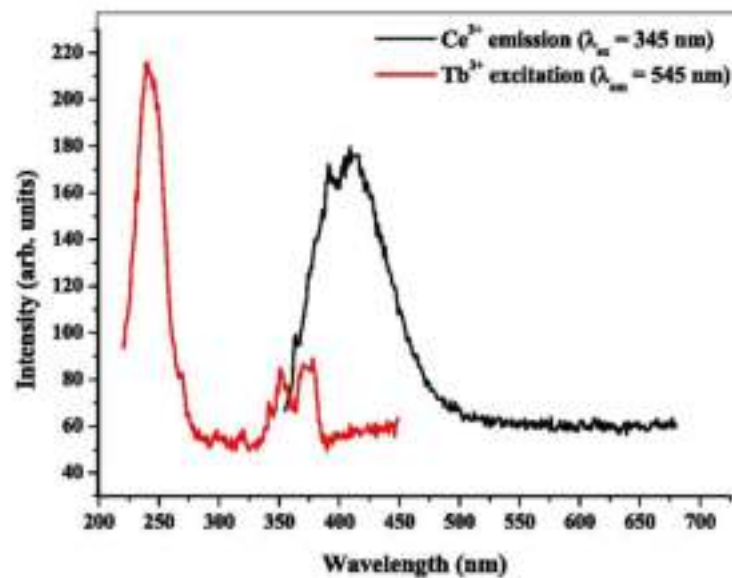


Fig. 7. Overlapping between luminescence spectrum of Ce^{3+} and absorption spectrum of Tb^{3+} in $\text{CaSrAl}_2\text{SiO}_6$ phosphor.

luminescence of the Tb^{3+} ion by the Ce^{3+} ion can be verified from the Fig. 7, where it was noted that emission spectrum of Ce^{3+} ion overlaps with absorption spectra of Tb^{3+} ion.

The origin of energy transfer process from Ce^{3+} to Tb^{3+} was determined, by using the method based on Dexter's energy transfer formula of multipolar interaction and Reisfeld's approximation:

$$\frac{I_0}{I} = (C)^n$$

where I_0 and I are the emission intensities of the Ce^{3+} ion without and with the presence of Tb^{3+} , respectively and C is the total dopant concentration of Ce^{3+} and Tb^{3+} ion. The plots of I_0/I versus $C^{1/3}$ for Ce^{3+} : Tb^{3+} (1.0:1.0, 3.0, 5.0, 7.0, 10.0 mol%) co-doped $\text{CaSrAl}_2\text{SiO}_7$ samples, excited at 345 nm with the luminescence monitored at 410 nm, are shown in Fig. 8. The best linear fitting was seen for $n = 6$ ($R^2 = 0.9800$), indicating that the energy transfer from Ce^{3+} to Tb^{3+} ions occurs via electric dipole-dipole interaction [24]. Fig. 9 represents the schematic of the energy level system describing energy transfer in the case of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor.

A comparison between PL emission of $\text{Ce}^{3+}/\text{Tb}^{3+}$ singly doped and co-doped samples is shown in Fig. 10, from which it can be seen that in the co-doped sample cerium emission intensity is decreased while terbium emission intensity is enhanced. This result suggested that co-doping of small amount (1.0 mol%) of Ce^{3+} ion in $\text{CaSrAl}_2\text{SiO}_7/\text{Tb}^{3+}$ enhanced luminescence emission of Tb^{3+} ion. Thus the energy transfer took place from $\text{Ce}^{3+} \rightarrow \text{Tb}^{3+}$ in $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor. Hence Ce^{3+} acted as sensitizer and Tb^{3+} acted as activator.

The color of luminescence emitted from $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (1.0 mol%), $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (1.0 mol%), Tb^{3+} (5.0 mol%) phosphor was determined by Commission International de l'Éclairage (CIE) chromaticity diagram as shown in Fig. 11. CIE chromaticity co-ordinates, calculated correlated color temperature (CCT) value and color purity for three phosphors are examined and represented in table 1. CIE co-ordinates of Tb^{3+} doped and $\text{Ce}^{3+}, \text{Tb}^{3+}$ co-doped samples are very close to equal energy point (0.3333, 0.3333) and highlight emission in near white region, calculated CCT value of these phosphors suggested that these phosphors is considered as cool in appearance. The low value of the color purity indicates the purity for white-light emission [25]. As $\text{Ce}^{3+}, \text{Tb}^{3+}$ co-doped phosphor has low value of color purity as compare to Tb^{3+} doped phosphor, hence co-

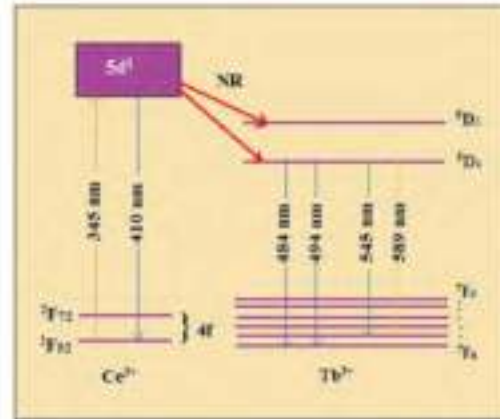


Fig. 9. Scheme of energy level and energy-transfer pathways in $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor.

doping of Ce^{3+} ion sensitizes the terbium emission and lowers the value of color purity of Tb^{3+} doped $\text{CaSrAl}_2\text{SiO}_7$ sample. Thus co-doped sample may be more appropriately useful for white light generation.

4. Conclusions

Tetragonal phase $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}/\text{Tb}^{3+}$ white-light emitting phosphors were successfully prepared by using the solid-state reaction method. The crystallinity and purity of the phase of as-prepared phosphors were measured by XRD technique. $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor showed characteristic luminescence bands of Ce^{3+} (410 nm) and Tb^{3+} (most intense band is at 545 nm) ion. The combination of these emission bands give rise to white-light emission and the chromaticity coordinates for $\text{Ce}^{3+}, \text{Tb}^{3+}$ co-doped phosphor are ($x = 0.3007$, $y = 0.2660$) with color purity 9.19×10^{-2} and a CCT value of 6954 K, which are close to the standard cool white region. Although $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphor also emitted near white light whose CIE chromaticity co-

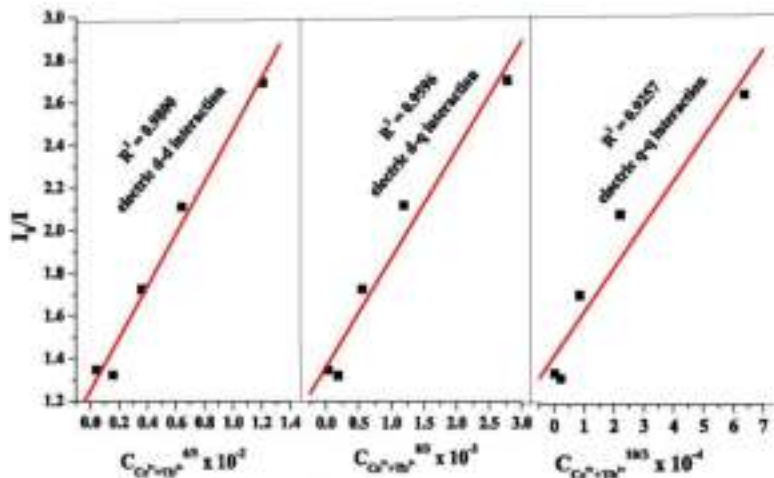


Fig. 8. Plots of I_0/I versus $C_{\text{Ce}^{3+}, \text{Tb}^{3+}}^{1/3}$ (1.0, 3.0 and 5.0) for $\text{Ce}^{3+}, \text{Tb}^{3+}$ co-doped $\text{CaSrAl}_2\text{SiO}_7$ samples excited at 345 nm and with the luminescence monitored at 410 nm.

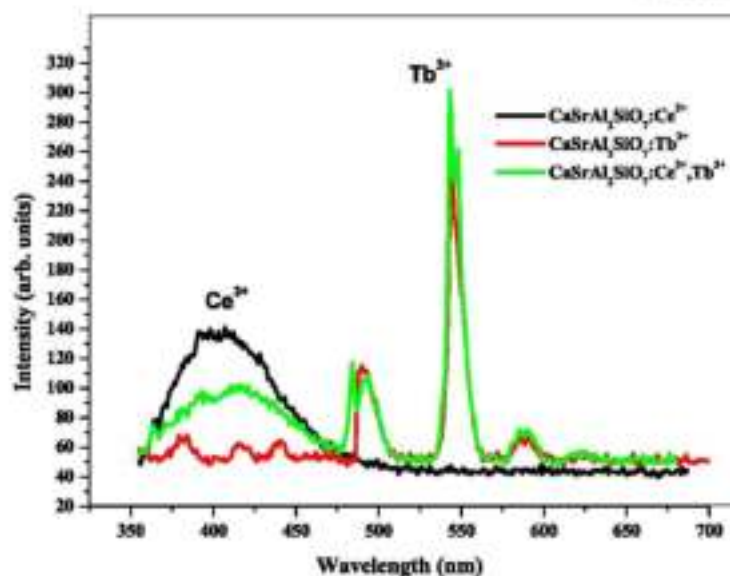


Fig. 10. Comparison between PL emission spectra of $\text{Ce}^{3+}/\text{Tb}^{3+}$ singly doped and co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphor.

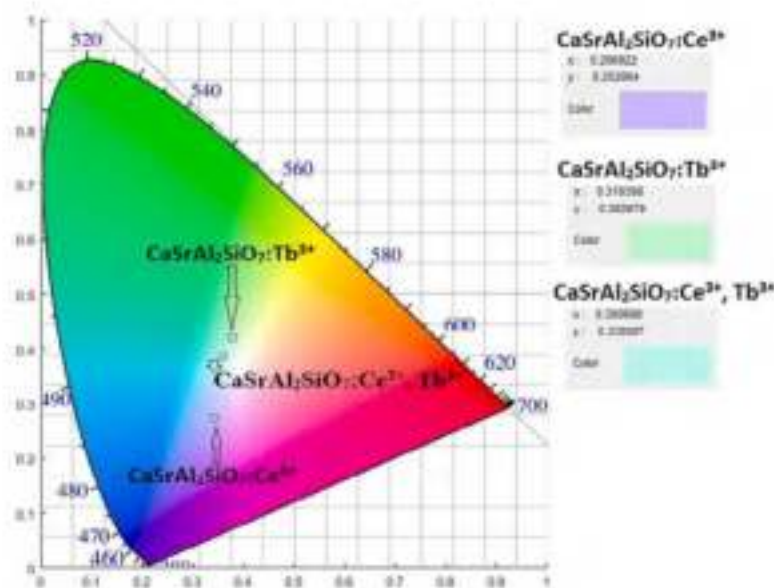


Fig. 11. CIE chromaticity of $\text{Ce}^{3+}/\text{Tb}^{3+}$ singly doped and co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphors.

Table 1

CIE, CCT and color purity values of optimized phosphors.

Phosphor	CIE co-ordinates	CCT	Color purity
$\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$	(0.2060, 0.2321)	11928 K	25.47 ± 10^{-2}
$\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$	(0.2194, 0.3830)	9972 K	15.05 ± 10^{-2}
$\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$	(0.2060, 0.2580)	6054 K	9.19 ± 10^{-2}

ordinates are (0.2194, 0.3830), color purity is 15.05×10^{-2} . As the low value of the color purity indicates the purity for white-light emission, hence we can say that inclusion of small amount (1.0 mol%) of Ce^{3+} ions enhanced the purity for white luminescence of Tb^{3+} doped $\text{CaSrAl}_2\text{SiO}_7$ phosphor. Therefore $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphor can be considered as a potential candidate for fabrication of white LEDs for outdoor illuminations.

5. Compliance with ethical standards

Authors have no conflict of interest.

Declaration of Competing Interest

The authors declared that there is no conflict of interest.

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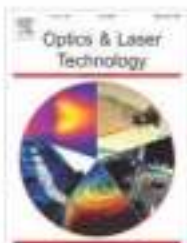


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Investigation of structural and thermal response of Sm³⁺ doped Sr₃MgSi₂O₈ phosphors

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Abstract

Present study deals with the effect of Sm³⁺ doping on crystal and optical behaviour of Sr₃MgSi₂O₈. For these studies prepared phosphor was characterized by using X-ray powder diffraction and luminescence spectra by recording excitation and emissions spectra. Emission spectra have emission wave lengths centered at 490 nm and 545 nm. Effect of Sm³⁺ ion concentration shows dipole-dipole interaction was responsible for the quenching shown by phosphor. The thermoluminescence curves were used to define the ability of the trap to trapping the carriers for Sm³⁺ doped Sr₃MgSi₂O₈ phosphor.

Keywords Sr₃MgSi₂O₈:Sm³⁺ · Solid state reaction method · Luminescence

1 Introduction

Rare earth doped Sr₃MgSi₂O₈ phosphors have been broadly contemplated by scientists inferable from their interesting physical and optical properties. These nanomaterials have been used in device fabrication, enthusiasm for examination of their conceivable use of use in various fields of gadget applications. They may take jobs in part of gadgets, for example, opto-electronics, sensors, identifiers, im-petuses, luminescent and bio-medical gadgets. A few kinds of silicate-based phosphor have been found and examined (Talwar et al. 2009; Klassen et al. 1957; Barry 1968; Blasse et al. 1968; Liu et al. 2014; Pan et al. 2011).

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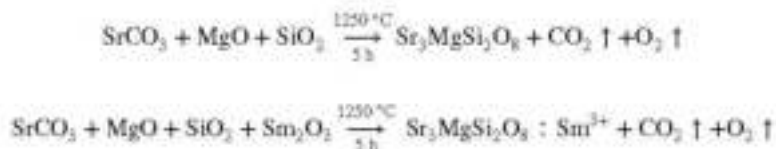


In this type of material synthesis temperature play a major role to produce pure phases materials, its luminescent brightness, brilliance and luminescence characteristics. Recently, silicate based materials have been extensively studied as host lattices for rare earth doping, which show un-usual, interesting luminescence properties (Pan et al. 2011; Zhang et al. 2016; Wang et al. 2018; Jung and Seo 2006).

Previously, optical and thermal response was investigations were achieved and reported for $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphor (Pan et al. 2011). In present study, synthesis, crystal structure, photoluminescence and thermoluminescence properties of Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor. The phase purity and structure was characterized by XRD, morphology and particle size determination was performed by SEM and TEM respectively. The excitation and emission spectra of title phosphor were measured and the mechanism of CQ was discussed. The thermoluminescence properties were also studied in detail for Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor. The trapping parameters of glow curves were also calculated by CGCD curve fitting method (Dewangan et al. 2018, 2019a, b).

2 Experimental

The pure and Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor was prepared by solid state reaction method. The raw materials for pure phosphor were $[\text{SrCO}_3$ (99.90%)], $[\text{MgO}$ (99.90%)] and $[\text{SiO}_2$ (99.99%)] and the raw materials for Sm^{3+} doped phosphor were same except the addition of small amount of Sm_2O_3 (99.99%). All the raw materials are of analytical grade. These raw chemicals were weighted and grinded into a fine powder by agate mortar and pestle for 2 h, then transferred in to a crucible, and calcined at 1250 °C for 5 h. All samples were observed when furnace-cooled to room temperature. Amount of raw ingredients extracted from the following chemical reactions:



3 Results and discussion

3.1 X-ray diffraction (XRD)

Phase compositions of un-doped and Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphors were examined by XRD and their diffraction patterns obtained (Fig. 1). JCPDS cards of $\text{Sr}_3\text{MgSi}_2\text{O}_8$ (no. 10-0075) is also illustrated in Fig. 1 as a comparison. The diffraction peaks well match with the standard pattern, suggesting that obtained samples have pure monoclinic phase $\text{Sr}_3\text{MgSi}_2\text{O}_8$. By small amounts of Sm^{3+} ions did not made any structural change which confirms proper incorporation of Sm^{3+} ion in crystal lattice, it maintained monoclinic phase (Dewangan et al. 2018, 2019a, b; Chen et al. 2016). The $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor samples exhibit obvious agglomeration and irregular shape without any porous structure. It is noted that the morphology of the samples did not significantly change when the dopant introduced into it.

Table 1. Different Parameter Calculated from XRD of $\text{Sr}_3\text{MgSi}_2\text{O}_8$ and $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphors

Sample's	2 θ	FWHM	Particle size	Strain	Dislocation Density
$\text{Sr}_3\text{MgSi}_2\text{O}_8$ Phosphors	22.62	0.12	67.52	0.00261	0.00021
	26.77	0.13	62.82	0.00238	0.00025
	38.91	0.19	44.35	0.00234	0.00050
	46.39	0.18	48.02	0.00183	0.00043
Sm^{3+} Doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ Phosphors	22.62	0.13	62.33	0.00283	0.00025
	26.77	0.14	58.33	0.00257	0.00029
	38.91	0.17	49.56	0.00209	0.00040
	46.39	0.16	54.02	0.00163	0.00034

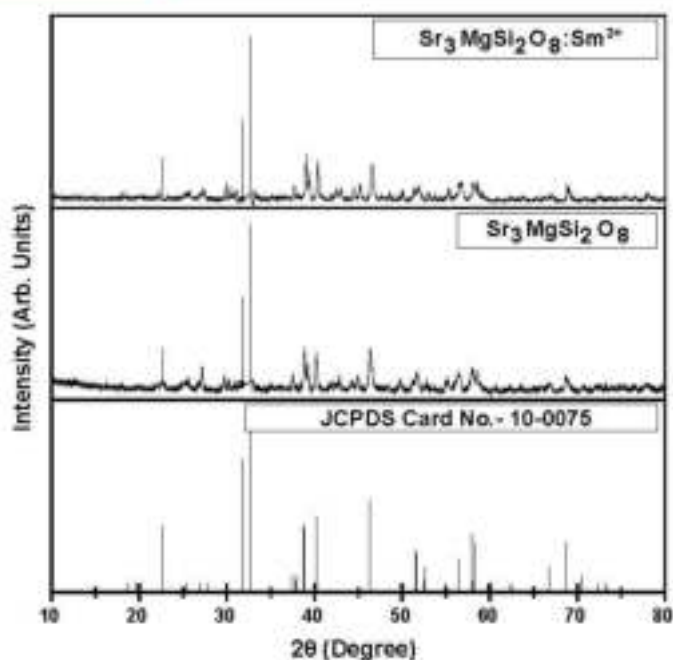


Fig. 1 X-ray diffraction (XRD) patterns of $\text{Sr}_3\text{MgSi}_2\text{O}_8$ and $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphors

3.2 Transmission electron microscopy (TEM)

The morphologies of single phase un-doped and Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphors were observed by TEM and micrographs are displayed in Fig. 2 shown that quasi-spherical morphology. SEAD pattern also displayed.

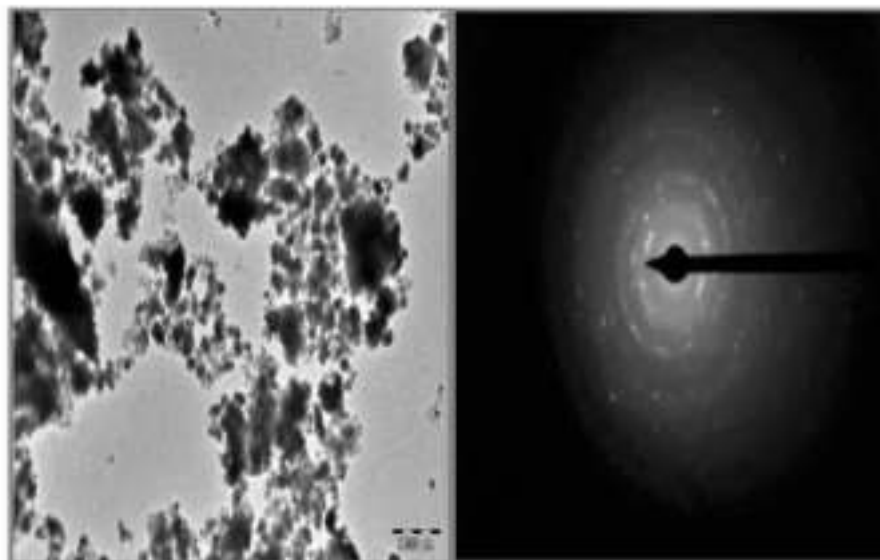


Fig. 2 TEM images of Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor

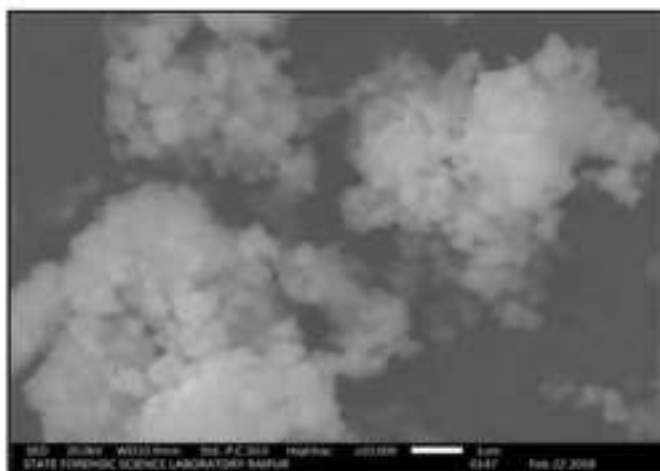


Fig. 3 SEM images of Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor

3.3 Scanning electron microscopy (SEM)

The grain morphologies of single phase pure and Sm^{3+} rare earth doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphors were done by field FESEM and the micrographs are displayed in Figs. 3 and 4, respectively.

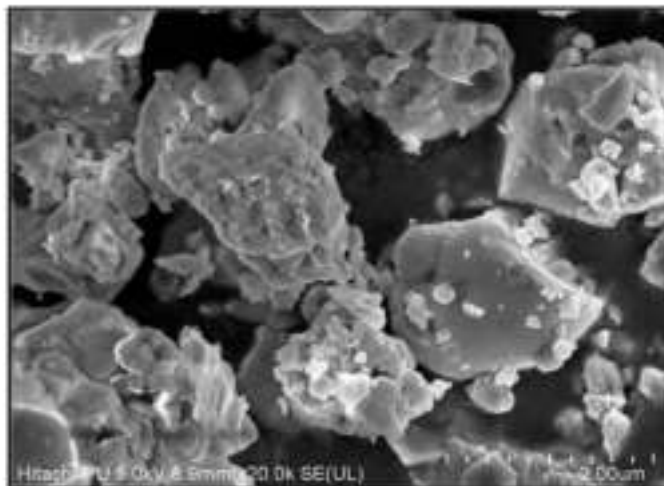


Fig. 4 FESEM images of Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor

Table 1 Chemical composition of Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor

S. no.	Element	Weight%	Atomic%
1.	Sr K	38.68	12.77
2.	Mg K	4.29	5.11
3.	Si K	9.20	9.47
4.	O K	39.26	70.99
5.	Sm K	8.57	1.65
Total		100	100

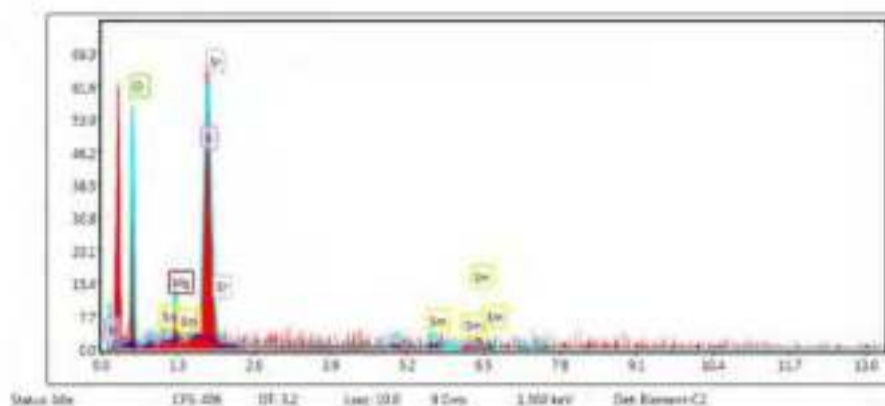


Fig. 5 EDS spectrum of Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor

3.4 Energy dispersive spectroscopy (EDS)

Elemental compositions of the prepared sample were determined using EDS. There were no emissions of other element in the EDS, other than those from doped Sr, Mg, Si, O, and Sm for Sm^{3+} doped $\text{Sr}_2\text{MgSi}_2\text{O}_8$ phosphors. Table 1 shows the compositional elements of rare earth doped $\text{Sr}_2\text{MgSi}_2\text{O}_8$ phosphors (Fig. 5).

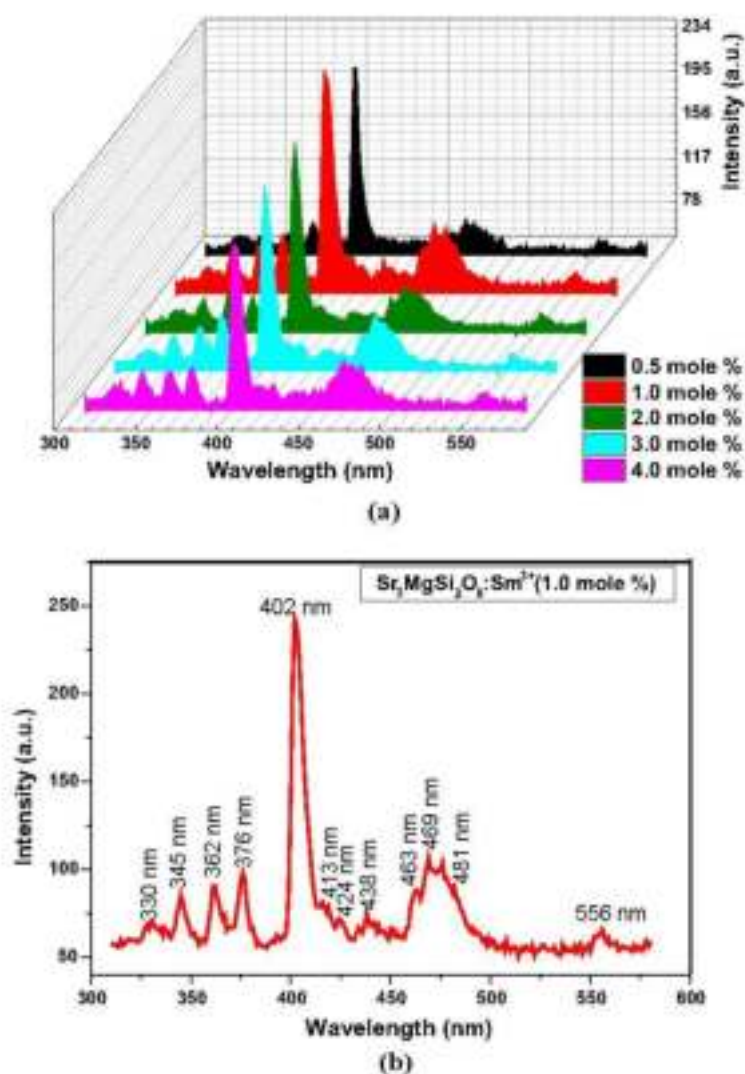


Fig. 6 Photoluminescence excitation (PL) spectra of **a** $\text{Sr}_2\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphors with variable Sm^{3+} concentration **b** $\text{Sr}_2\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ (1.0 mol%) phosphor

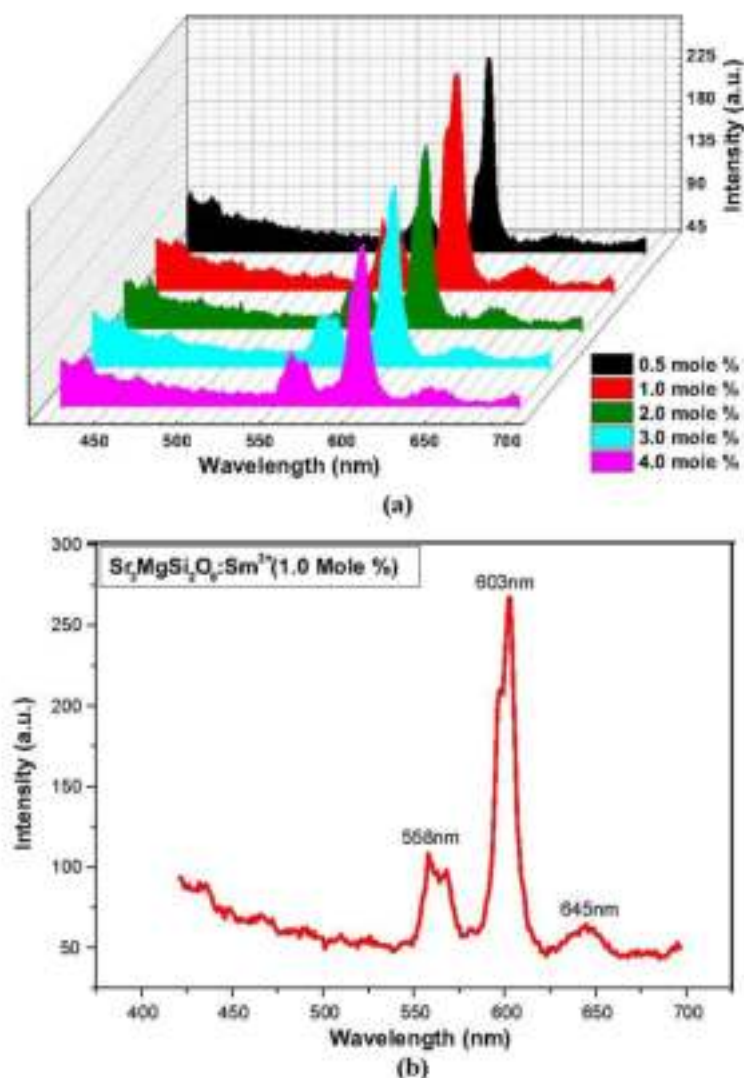


Fig. 7 Photoluminescence emission spectra of **a** $\text{Sr}_3\text{MgSi}_2\text{O}_8:x\text{Sm}^{3+}$ ($x = 0.5, 1.0, 2.0, 3.0$ and 4.0 mol%) phosphors and **b** $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ (1.0 mol%) phosphor, under 402 nm excitation

3.5 Photoluminescence (PL)

For PL study of $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$, its luminescence spectra were recorded (Fig. 6a, b). Under 603 nm wavelength excitation spectra were recorded and shows peak in UV to visible region. Excitation peaks are located at 330 nm ($^6\text{H}_{5/2} \rightarrow ^2\text{L}_{15/2}$), 345 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{H}_{9/2}$), 362 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{D}_{3/2}$), 376 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{D}_{1/2}$), 402 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{F}_{7/2}$), 413 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{M}_{15/2} + (^6\text{P}, ^6\text{P})_{5/2}$), 424 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{P}_{3/2}$), 438 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{G}_{9/2}$), 463 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{I}_{13/2}$), 469 nm and 481 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{I}_{11/2} + ^4\text{M}_{15/2}$ and 556 nm ($^6\text{H}_{5/2} \rightarrow ^4\text{G}_{3/2}$) (Jha

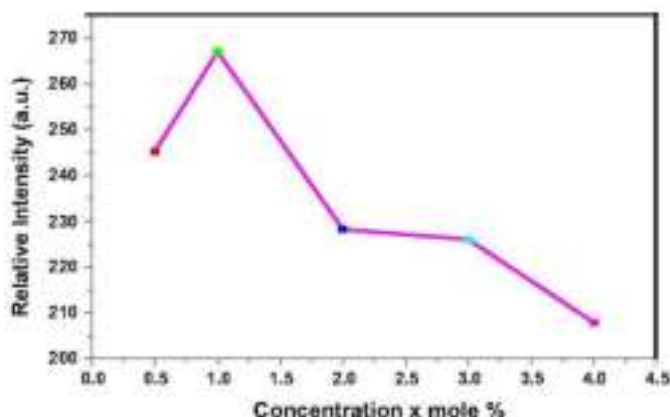


Fig. 8 Dependence of emission intensity of Sm^{3+} concentration for $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphor

et al. 2017; Sun et al. 2016; Lai et al. 2017). At 402 nm corresponds to $^6\text{H}_{5/2} \rightarrow ^4\text{F}_{3/2}$ transition, is the strongest one so used to record emission spectra (Ikhechi et al. 2015a, b, c; Ikhechi and Kaleji 2016).

Figure 7a, b shows emission spectra of $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ ($x=0.5$ to 4.0 mol%) phosphors recorded under 402 nm excitation. The emission peaks centred at yellow-orange-red region at 558 nm, 603 nm and 645 nm.

The emission band located at 558 nm is due to transition $^6\text{G}_{5/2} \rightarrow ^6\text{H}_{5/2}$, 603 nm and 645 nm are due to $^6\text{G}_{5/2} \rightarrow ^4\text{H}_{1/2}$ and $^6\text{G}_{5/2} \rightarrow ^6\text{H}_{9/2}$, respectively (Yu et al. 2012). Unaffected peak positions remains with variation in Sm^{3+} molar percentage but emission intensity alters (Fig. 8).

With increasing Sm^{3+} concentration intensity increase up to 1.0 mol% further increase in concentration results in concentration quenching (CQ) due to non radiative energy transfer between neighbouring Sm^{3+} ions. It is depends on critical distance (R_c) and calculated by using the Blasse equation in Eq. (1) (Blasse 1969; Dewangan et al. 2020): by using critical concentration x_c , number of cations N and volume of unite cell V

$$R_c = 2 \left(\frac{3V}{4\pi x_c N} \right)^{1/3} \quad (1)$$

For $\text{Sr}_3\text{MgSi}_2\text{O}_8$ $N=4$, $V=715.9 \text{ \AA}^3$, and $x_c=0.01$. R_c was found around 33 Å. For R_c shorted than 5 Å quenching is due to exchange above this multipolar interaction. Therefore, energy transfer mechanism in $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphor has multipolar interaction.

Here emission intensity per doped ion is followed by the Eq. (2):

$$\log \frac{I}{x} = -Q/3 \log x + \log K - \log \beta \quad (2)$$

It is a straight line equation. From slope of $\log(I/x)$ Vs $\log(x)$ plot we find out Q value (Fig. 9). The Q value is 3.336 which is closer to 4 as compare to 8. Hence energy transfers mechanism between Sm^{3+} ions in $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphor follows the dipole-dipole interaction. Corresponding energy level diagram for luminescence emission

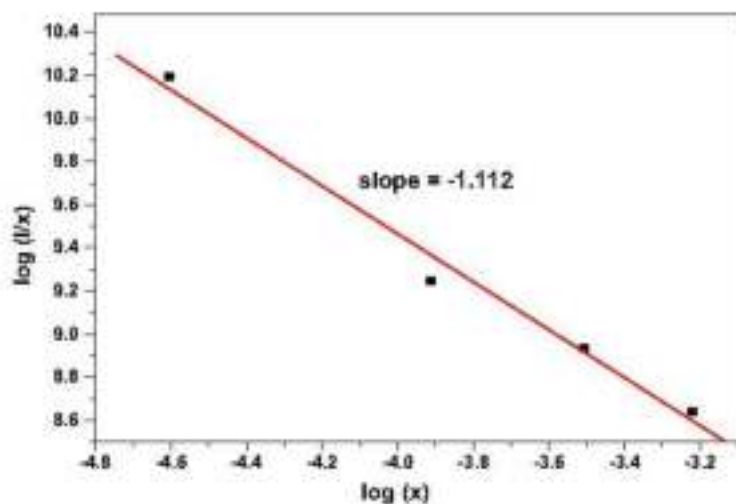


Fig. 9 Plot between $\log(I/I_0)$ and $\log(x)$ for $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Sm}^{2+}$ phosphor

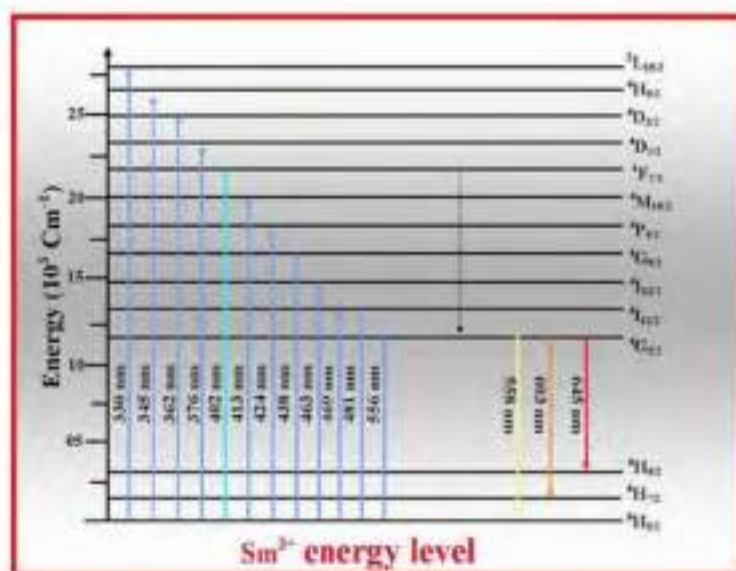


Fig. 10 Energy level diagram of Sm^{3+} ions

from $\text{Sr}_3\text{MgSi}_2\text{O}_8\cdot\text{Sm}^{2+}$ phosphor is represented in Fig. 10 (Dexter and Schulman 1954; Uiterl 1967).

The luminescence color of Sm^{3+} doped $\text{Sr}_3\text{MgSi}_2\text{O}_8$ phosphor is identified by using CIE coordinates placed at $X=0.417$ and $Y=0.420$ that very close to the orange light emission in the chromaticity diagram (Fig. 11) (CIE, International Commission on Illumination, Publication CIE, no. 15 1931).

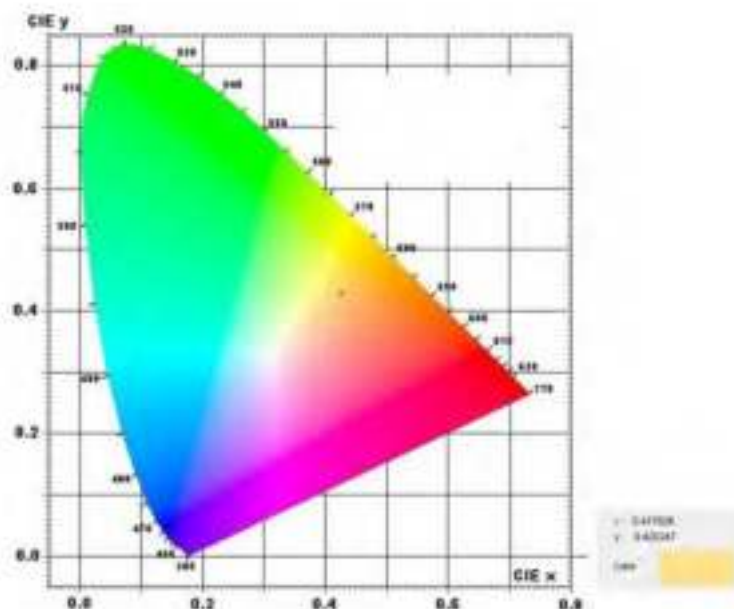


Fig. 11 CIE chromaticity diagram for $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Sm}^{3+}$ (1.0 mol%) phosphor excited at 402 nm

3.6 Thermoluminescence (TL)

Thermally stimulated luminescence glow curves were recorded at room temperature on a thermoluminescent dosimetric material reader TL1009I supplied by Nucleonix Systems Pvt. Ltd. Hyderabad. The phosphor obtained under thermoluminescent examination was given ultraviolet (UV) radiation from a 365 nm source.

TL of prepared phosphors was recorded by giving 365 nm UV source irradiation for different time. Every time 0.5 mg weighed powdered phosphor was taken for TL measurement. The heating rate used for TL measurements was 3°C s^{-1} , 4°C s^{-1} , 5°C s^{-1} , 6°C s^{-1} , 7°C s^{-1} and 8°C s^{-1} (Chen and Kirsh 1981).

TL glow curve of the Sm^{3+} doped sample was obtained and it was observed that the glow curve has two peaks at 131°C and 233°C . To optimize the heating rate TL glow curve was recorded with different heating rate from 3 to 8°C s^{-1} . A slight variation in peak position along with intensity was observed. Optimization of heating rate is necessary to overcome the effect of thermal quenching. Maximum intense peak was obtained for 5°C s^{-1} heating rate therefore it was used as optimal heating rate for the further study (Fig. 12).

Concentration was optimized by recording the TL glow peak for different concentration of Sm^{3+} . The intensity of both the glow peak increases with increasing Sm^{3+} intensity up to 2.0 mol % after that the glow peak intensity starts to fall due to the CQ (Fig. 13). Another factor such as the optimization of UV exposure time was further determined. As shown in Figs. 14 and 15 the glow peak intensity increases up to 70 min of UV exposure time after its decrease due to destroy of the traps. The corresponding activation energy (E) values were calculated by the use of formulas modified by Chen

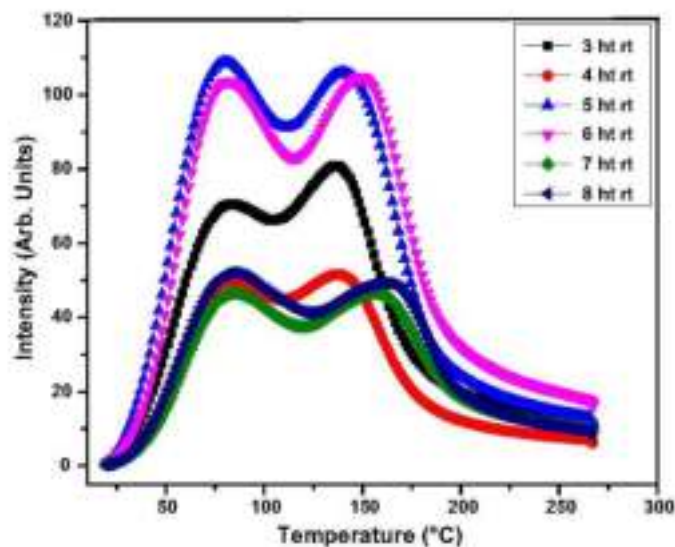


Fig. 12 TL glow curves of $\text{Sr}_3\text{MgSi}_3\text{O}_{12}:\text{Sm}^{3+}$ (2.0 mol%) for different heating rates

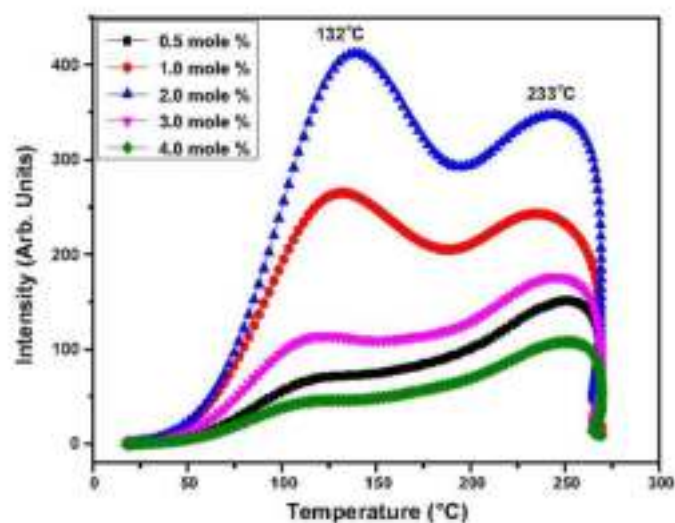


Fig. 13 Sm^{3+} concentration dependent TL glow curves of $\text{Sr}_3\text{MgSi}_3\text{O}_{12}:\text{Sm}^{3+}$ phosphor for 70 min UV exposure time

and others (Chen and Kirsh 1981; Chen and Mc Keever 1997; Chen and Pagonis 2011; Chen 1969a).

TL glow peak of the sample was deconvoluted by using computerized glow curve deconvolution process. The sample shows two deconvolution peaks as shown in Fig. 16. Peak shape method were used to calculated TL parameters (Dewangan et al. 2018;

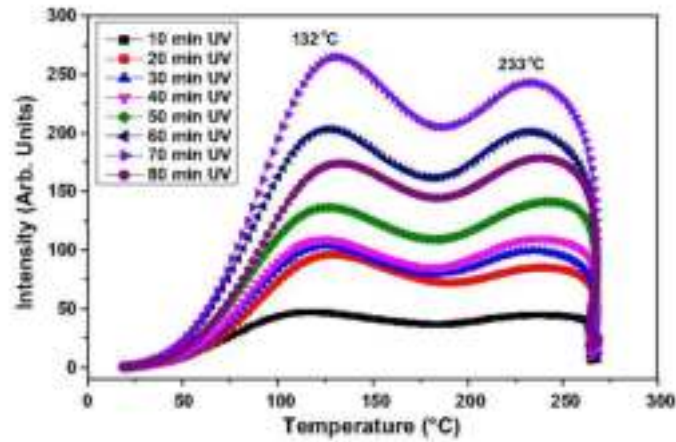


Fig. 14 TL glow curves of $\text{Sr}_3\text{MgSi}_2\text{O}_{8.8m^{3+}}$ (2.0 mol%) phosphor for different UV exposure time

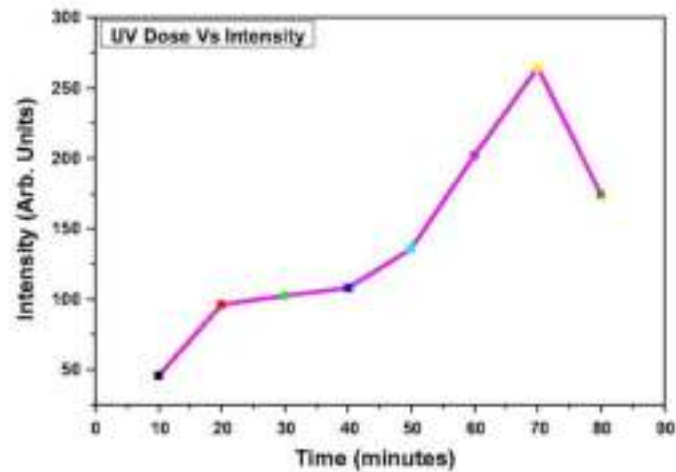


Fig. 15 TL intensity V/S UV exposure time for $\text{Sr}_3\text{MgSi}_2\text{O}_{8.8m^{3+}}$ (2.0 mol%) phosphor

Pagonis et al. 2006; Tamrakar and Upadhyay 2017). The relationship between the frequency factor's' and the activation energy 'E' is given by the Eq. (3)

$$\frac{\beta E}{kT_m^2} = z \left[1 + (b-1) \frac{2kT_m}{E} \right] \exp(E/KT_m) \quad (3)$$

where, k=Boltzmann constant, E=activation energy, b=order of kinetics, T_m =temperature of higher peak position and b=order of kinetics.

In the present work β 5 °C/s. For Trap depth calculation for 2nd order kinetics is using the Eq. (4) (Chen 1969b).

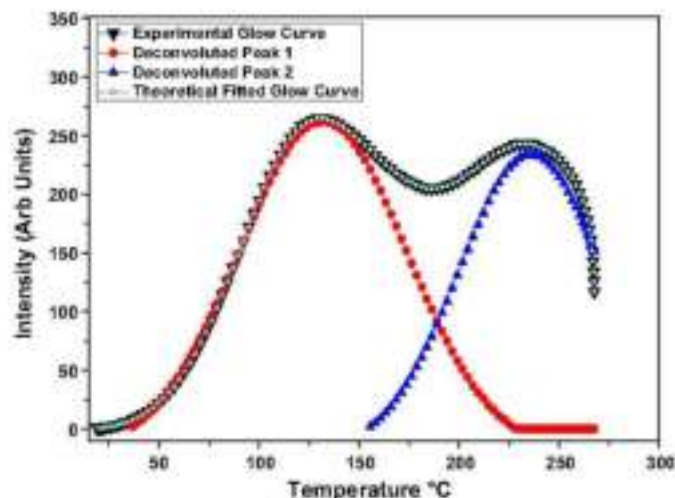


Fig. 16 Peak deconvolution of TL glow curve of 70 min UV exposed $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ (2.0 mol%) phosphor

$$E = 2kT_m \left(1.76 \frac{T_m}{\theta} - 1 \right) \quad (4)$$

The calculated kinetic parameters of $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphor by the peak shape method is given in Table 2. In our case, the value of shape factor has been calculated to be ≈ 0.51 for both the peak, which indicates that it is a case of second order kinetics. The activation energies for the prepared $\text{Sr}_3\text{MgSi}_2\text{O}_8:\text{Sm}^{3+}$ phosphor were estimated to be 0.49 and 1.53 eV for 1st and 2nd peak respectively (Dewangan et al. 2019a; Tamrakar et al. 2017, 2019; Tamrakar and Upadhyay 2016; Tamrakar 2013; Tamrakar and Bisen 2015).

4 Conclusion

The Sm^{3+} doped phosphor was obtained by solid state reaction method. Phosphor have crystallizes in a monoclinic phase confirmed by XRD analysis. The XRD patterns confirmed single phase, monoclinic structure of the sample. The particle size is in nano range confirmed by TEM. All the component elements in the desired samples have been detected by employing the EDS spectrum. FESEM results confirmed agglomerated morphology of the sample.

Under 402 nm excitation, characteristic photoluminescence (PL) peaks recorded. The emission peaks centred at yellow-orange-red region at 558 nm, 603 nm and 645 nm for Sm^{3+} doped phosphor. Further, the phosphor showed excellent CIE chromaticity coordinates (X,Y) in white region and CCT observed in orange light emission as a result. The mechanism of CQ was discussed well which due to dipole-dipole interactions. The phosphor emits in yellow-orange-red region.

Table 2 Analysis of glow curves by CGCD curve fitting and trapping parameters of $\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Sm}^{2+}$ (2.0 mol%) phosphor

Phosphor name	Peaks	T_1 (°C)	T_2 (°C)	T_m (°C)	τ (°C)	β (°C)	α (°C)	$\eta_g = \beta/\alpha$	E (eV)	S (s^{-1})
$\text{Sr}_2\text{MgSi}_2\text{O}_7:\text{Sm}^{2+}$	Peak 1	89.64	173.74	131.79	42.15	41.95	84.10	0.50	0.49	9.9×10^6
	Peak 2	212.16	269.16	235.33	23.17	24.83	48.00	0.51	1.53	3.2×10^{16}

The TL curve was recorded under UV exposure. The glow peak has two peaks that may be due to formation of two different trap centres. The optimized factors for TL glow curve were obtained as 5°C s^{-1} heating rate, 2.0 mol % of Sm^{3+} concentration and 70 min UV exposure time. The computerized glow curve deconvolution study showed the presence of two traps having activation energies of 0.49 eV and 1.53 eV for the lower temperature and higher temperature peaks respectively which confirms the presence of shallow traps and deep traps respectively.

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Adaptation of an indigenous insect cell line DZNU-Bm-1 to Mitsuhashi and Maramorosch (MM) Culture Medium supplemented with 3% Foetal Bovine Serum: S. O. Qureshi

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Adaptation of an Indigenous Insect Cell Line DZNU-Bm-1 to Mitsuhashi and Maramorosch (MM) Culture Medium Supplemented With 3% Foetal Bovine Serum

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ABSTRACT

Although utility of insect cell lines for production of proteins of human interest has been scaled up from experiment-level to industrial production level, use of vertebrate sera is discouraged during industrial production of therapeutics. DZNU-Bm-1, a larval ovarian cell line is highly susceptible to BmNPV in MGM-440 medium supplemented with 10% Foetal Bovine Serum and can be used as IC-BEVS platform for foreign gene expression. The high cost of this medium is a serious bottleneck for use of DZNU-Bm-1 in production of therapeutics. This study reports reduction in serum dependence of this cell line and its successful adaptation to a low-cost Mitsuhashi and Maramorosch (MM) medium supplemented with 3% FBS to bring down the cost of maintaining the cell line. Adaptation of MM medium was achieved by decreasing the volume of original medium by 20% and increasing the volume of the new medium to which the cell line was being adapted by 30% at each passage. The initial cell density, final cell density, number of days taken to attain final cell density and population doubling time were noted at each passage. The cell line was slow to reach sufficient cell density during initial steps but showed reduced population doubling time during subsequent passages.

KEY WORDS: DZNU-Bm-1, INSECT CELL LINE, ADAPTATION, POPULATION DOUBLING TIME.

INTRODUCTION

Insect cell lines have found application in a wide array of fields including cell biology, genetics, virology and agriculture. Insect cells have many advantages over mammalian cells in culture, being able to

tolerate a broad range of environmental conditions, to proliferate and maintain physiological function in variable pH, temperature, and oxygen conditions (Akiyama et al., 2013). Their ability to grow without carbon dioxide supplementation has further simplified culture systems and has made them an economically viable alternative to mammalian cells (Irons et al., 2018). Insect cells exhibit many additional desirable characteristics like easy adaptation to suspension and serum-free culture as well as low media requirements. These qualities point to possibility of their large-scale culture (Neumann and Wagner, 1999; Donaldson and Shuler, 1998; Lynn, 2001; Ikumoto et al., 2002; Mitsuhashi and Goodwin, 2003).

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842

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Recently, Rubio et al. (2019) have suggested that pursuing development of insect cell-based foods may lead to new sustainable food products thereby accelerating the field of cellular agriculture. Trager's (1935) success in using inorganic salts, maltose, digested egg albumin and *B. mori* haemolymph to maintain ovarian tissues of *B. mori* spawned a wave of experiments to formulate growth media for culture of insect cells. Mitsuhashi (1982, 1984) successfully modified Grace's medium several times for cultivation of different cell lines. Supplementation with vertebrate sera like foetal bovine serum (FBS) or other proteinaceous materials has been successfully used to get high growth rate (Weiss and Vaughn, 1986). Gradually with increase in interest of researchers in insect cell culture, many serum-free media have been developed (Jlink, 1991; Donaldson et al., 1998). Caron et al. (1990) reported high growth rate by increasing total Pluronic F-68 concentration to 0.3% with IPL/41 serum-free medium. Lepidopteran cell lines are now being viewed as an attractive alternative to mammalian cell lines for biomanufacturing of proteins of human interest, biopesticides and vaccines (Kost et al., 2005; Drugmand et al., 2012; Aïrenne et al., 2013; Van Oers et al., 2015).

Over the years the utility of insect cell lines for production of proteins of human interest has been scaled up from experiment-level to industrial production level (Elias, 2007). Use of vertebrate sera is discouraged during industrial production of therapeutics because of inherent potential risk for transmission of infectious agents as well as the heterogeneity and lack of reliability (Grillberger et al., 2009). Insect cell lines that have high susceptibility to a baculovirus can be used to develop efficient Insect Cell-Baculovirus Expression Vector Systems (IC-BEVS) (Agathos, 2010). An efficient IC-BEVS production platform can be used for foreign gene expression, production of therapeutic compounds and vaccines. DZNU-Bm-1, a larval ovarian cell line has been shown to be highly susceptible to BmNPV in MGM-448 medium (Khurad et al., 2006) and can be used as IC-BEVS platform for foreign gene expression (Khurad et al., 2013).

However, the high cost of MGM-448 medium with supplementation of 10% Foetal Bovine Serum (FBS) is a serious bottleneck for its use in production of therapeutics or other proteins of interest. The present study describes successful reduction in the serum dependence of the cell line DZNU-Bm-1 by first adapting it to MGM 448 growth medium supplemented with 3% FBS and then to a low-cost Mitsuhashi and Maramorosch (MM) medium supplemented with 3% FBS to reduce the cost of maintaining this cell line.

MATERIAL AND METHODS

Culture Media: Modified Grace's medium MGM-448 (Mitsuhashi, 1984) and Mitsuhashi and Maramorosch medium (Mitsuhashi and Maramorosch, 1964) were prepared in laboratory. Modified Grace's Medium (MGM-448): MGM-448 is a complex medium composed of six

salts, twenty amino acids, three sugars, four organic acids, ten vitamins and four additives, inosine, cytochrome-c, fetuin and bovine plasma albumin fraction-V. Five stock solutions viz. MGM O.S.A., GMA-salt-mix-A, GMA-salt-mix-B, GMA-Vita-mix-1A, GMA-Vita-mix-1B were prepared as described by Mitsuhashi (1984).

In order to prepare 250 ml of complete MGM-448 medium, required quantities of respective stock solutions were mixed and the double distilled water was added to make up the volume. To this solution bovine plasma albumin fraction-V, fetuin, cytochrome-c, inosine were added. The medium was supplemented with required quantity (3% or 10%) of FBS. The pH was adjusted to 6.37-6.40 with saturated KOH. The medium was sterilized by passing through 0.2 µm pore size Millipore membrane filter using negative pressure through Sartorius filter unit. No antibiotics were added to the medium.

Mitsuhashi and Maramorosch medium (MM Medium): It is among the simplest insect tissue culture media. It is a mixture of salts used in Carlson's Balanced Salt Solution (CBSS), lactalbumin hydrolysate, TC-yeastolate and glucose supplemented with 3% Foetal Bovine Serum (FBS). For preparation of this medium, stock solution A and stock solution B of CBSS were prepared. 25 ml of each stock solution was mixed. The additives were added and the volume was made up to 250 ml by adding double distilled water. The medium was supplemented with 3% FBS. Sterilization was carried out by passing through 0.2 µm pore size Millipore membrane filters using negative pressure through Sartorius filter unit. No antibiotics were added to the medium.

Maintenance of cell line and subculturing: DZNU-Bm-1 a larval ovarian cell line from *B. mori* was kindly provided by Dr. A. M. Khurad. The cell line was being cultured in MGM-448 medium supplemented with 10% FBS. The cell line was maintained in the laboratory in glass tissue culture flasks, incubated at 25±1°C and passaged regularly. After some time, the cell line could be subcultured regularly by splitting the cultures in a ratio of 1:2 at an interval of 4-5 days.

Adaptation to MGM-448 medium supplemented with 3% FBS: The cell line was first adapted to MGM-448 supplemented with 3% FBS to reduce its serum dependence as described by Mitsuhashi and Grace (1969) through passaging of the cells. This was done by decreasing the volume of original medium (MGM-448 supplemented with 10% FBS) by 20% and increasing the volume of MGM-448 medium supplemented with 3% FBS by 20% at each passage. The initial cell density, final cell density, number of days taken to attain final cell density and population doubling time were noted at each passage.

Adaptation to MM medium supplemented with 3% FBS: The cell line was subsequently adapted to MM medium supplemented with 3% FBS. The volume of MGM-448 supplemented with 3% FBS was reduced while the volume of MM medium supplemented with 3% FBS

was increased through regular passaging. During each passage, the volume of original medium was reduced by 20% while the volume of MM medium supplemented with 3% FBS was increased by 20%. The process was repeated till MGM-448 supplemented with 3% FBS was completely replaced with MM medium supplemented with 3% FBS. The initial cell density, final cell density, number of days taken to attain final cell density and population doubling time were noted at each passage.

Population Doubling Time: Cell suspension was sampled from culture flasks at the time of seeding the culture and on attainment of final cell density. The cell suspension was allowed to flow in the chambers of Neubauer haemocytometer chamber and cells were counted in four large corner squares each of which is divided into 16 small squares. The volume of one large square is 0.1 mm³. Therefore, average cell count was multiplied by 104 to give number of cells per ml. The cell number was determined as an average of readings from two culture flasks. The population doubling time (PDT) was calculated using the exponential formula given by Hayflick (1973).

$$r = 3.32 \{ \log x_2 / x_1 \} / t_2 - t_1$$

where,

r = Multiplication rate,

x₁ = Initial cell number at selected time t₁,

x₂ = Final cell number at selected time t₂.

Generation time (g) = 1/r

Cell Morphology: The cell cultures were observed under MAGNUS INVI inverted phase contrast microscope and photographed regularly after they were adapted to MM medium supplemented with 3% FBS. Cell population comprised of different cell types. The cell size of each type was determined by ocular micrometer in the population taking about 50-100 readings. The relative percentage of each type in population was determined by counting different cell types in optical fields under the inverted microscope.

Karyotype Analysis: Karyotypic studies were carried out in healthy cell cultures during exponential cell growth phase after their successful adaptation to MM medium supplemented with 3% FBS. Demicolcine (Sigma) having a concentration of 50 µg/ml was added to culture flasks to arrest the cell division at metaphase. Final concentration of demicolcine in the culture flask was 1 µg/ml of medium. The cultures were allowed to stand at 25°C for 24-72 hours. The cells were harvested by centrifugation and the supernatant was removed. The pellet was resuspended in hypotonic solution (0.6% KCl) for 15-20 minutes. The cells were again centrifuged and the cell pellet was fixed in 50% glacial acetic acid for 10-15 minutes. The fixed cells were spread on glass slides and allowed to air dry. The slides were washed in distilled water, transferred to Acetocorn stain for 20 minutes, washed briefly in running tap water, air-dried for 12 hours, cleared in two changes of xylene and mounted in DPX. About 50 chromosome spreads were

counted and spreads were photographed to determine the range of chromosomes.

Table 1. Population Doubling Time of DZNU-Bm-1 during adaptation from MGM-448 medium supplemented with 10% FBS (A) to MGM-448 medium supplemented with 3% FBS (B).

No. of Days after Seeding of Cultures	Step	Medium Composition	Population Doubling Time in Hours
06	a	80% A + 20% B	52
07	b	60% A + 40% B	68
09	c	40% A + 60% B	83
09	d	20% A + 80% B	81
08	e	100% B	70

(A) = MGM-448 medium supplemented with 10% FBS

(B) = MGM-448 medium supplemented with 3% FBS

Figure 1. Graph showing adaptation of DZNU-Bm-1 from MGM-448 medium supplemented with 10% FBS (A) to MGM-448 medium supplemented with 3% FBS (B). a- 80% A + 20% B; b- 60% A + 40% B; c- 40% A + 60% B; d- 20% A + 80% B; e- 100% B



RESULTS AND DISCUSSION

In the first stage of experiment, DZNU-Bm-1 cell line originally cultured in MGM-448 medium supplemented with 10% FBS was successfully adapted to MGM-448 medium supplemented with 3% FBS through several passages. The cell line took about 39 days for adaptation (Fig. 1). DZNU-Bm-1 has been reported to take more than 10 months for adaptation to haemolymph-free MGM-448 (Babekar, 2018). The cells remained freely suspended in culture medium. Cell clumping reported in earlier studies (Babekar and Qureshi, 2013) was not observed. During step b of adaptation the cells showed reduced growth (Fig. 1). The growth of cells during step c and step d was slowest (Fig. 1). During each of these steps cells required 9 days to reach sufficient cell density for subculturing. The population doubling time was also highest during these steps showing a peak value of about 83 hours (Table 1). Bellonck et al. (1990) have reported slow

Qureshi

growth rate and other difficulties during adaptation. In the present study, by step-e the population doubling time was reduced to about 70 hours. After further passages in the same medium population doubling time was reduced to about 49 hours.

The cell line was later adapted to more cost-effective MM culture medium supplemented with 3% FBS. The cell line was easily adapted to MM medium taking about 32 days for adaptation (Fig. 2). Growth of cells was slow during step b and c requiring about 7 days each for attaining sufficient cell density for subculture. Population doubling time was maximum (about 81 hours) during step b (Table 2). During next subcultures the cell line could be passaged by day 6 with population doubling time being reduced to about 54 hours. There was no significant change in cell morphology after adaptation to MM medium supplemented with 3% FBS. The population of DZNU-Bm-1 cell line is heterogeneous and can be differentiated into four cell types- large round, small round, giant cells and spindle shape - on the basis of their shape and size.

Table 2. Population Doubling Time of DZNU-Bm-1 during adaptation from MGM-448 medium supplemented with 3% FBS to MM medium supplemented with 3% FBS (II)

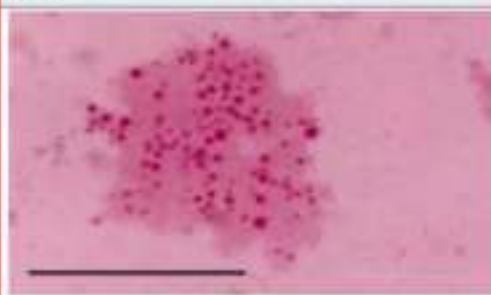
No. of Days after Seeding of cultures	Step-designation	Medium composition	Population Doubling time in Hours
06	a	80% A + 20% B	48
07	b	60% A + 40% B	81
07	c	40% A + 60% B	70
06	d	20% A + 80% B	53
06	e	100% B	54

(A)= MGM-448 medium supplemented with 3% FBS
(B)= MM medium supplemented with 3% FBS

Figure 2: Graph showing adaptation of DZNU-Bm-1 from MGM-448 medium supplemented with 3% FBS (A) to MM medium supplemented with 3% FBS (B). a- 80% A + 20% B; b- 60% A + 40% B; c- 40% A + 60% B; d- 20% A + 80% B; e- 100% B



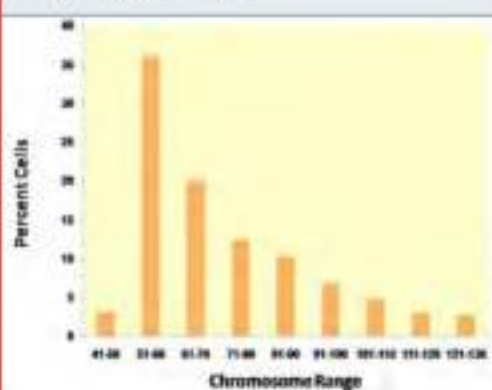
Figure 3: Chromosomes of DZNU-Bm-1 cell line grown in MM culture medium supplemented with 3% FBS. Bar= 30 μ m



Large round cells: They are round, measuring about $31 \pm 0.62 \mu$ m in diameter. Their relative percentage in the population is about 30.2%. Initially, they showed partial attachment to the surface of culture flask immediately after subculture.

Small round cells: The small round cells measure about $13.74 \pm 0.16 \mu$ m. They form 53.4% of the total cell population.

Figure 4: Graph showing distribution of chromosome number in DZNU-Bm-1 cells.



Giant cells: These are very large round cells having a diameter of about $69 \pm 1.22 \mu$ m. They form 4.7% of the total cell population.

Spindle-shape cells: These cells have protoplasmic processes on the sides giving them appearance of a spindle. Their size is about $51 \pm 3.42 \mu$ m $\times$ $31 \pm 0.51 \mu$ m. Their percentage in cell population is 11.7%.

The chromosome spreads of cells exhibited numerous microchromosomes that resembled the chromosomes of DZNU-Bm-12 cell line (Khurad et al., 2009). The chromosomes appeared as numerous dot-like, darkly stained bodies (Fig. 3). The diploid number of *B. mori* chromosomes has been known to be 56. The chromosome

number in the cell line ranged from 45 to 126 with a mode towards diploidy. A few cells also possessed more than 100 chromosomes indicating the presence of polyploid cells (Fig. 4). Grace (1967) has reported more than 100 chromosomes in the cells of *B. mori* ovarian cell line. Chromosome range of 35 to 150 in larval ovarian cell line and 60 to 180 in the pupal ovarian cell line of *B. mori* have also been reported (Sudheep et al., 2002).

CONCLUSION

In the present study DZNU-Bm-1 cell line being cultured in MGM-448 medium supplemented with 10% FBS was first adapted to MGM-448 medium supplemented with 3% FBS. The cell line was slow to reach sufficient cell density during initial steps but showed reduced population doubling time during subsequent passages. The cell line was then successfully adapted to cost effective MM medium supplemented with 3% FBS. The morphological and cultural characteristics as well as the chromosome range of the cells did not exhibit any significant change from those cultured in MGM-448 (30% FBS). DZNU-Bm-1 has earlier been shown to be able to produce foreign proteins. The current study establishes its reduced serum dependence and ability to grow in low-cost culture media raising hopes of adapting it to other serum-free media.

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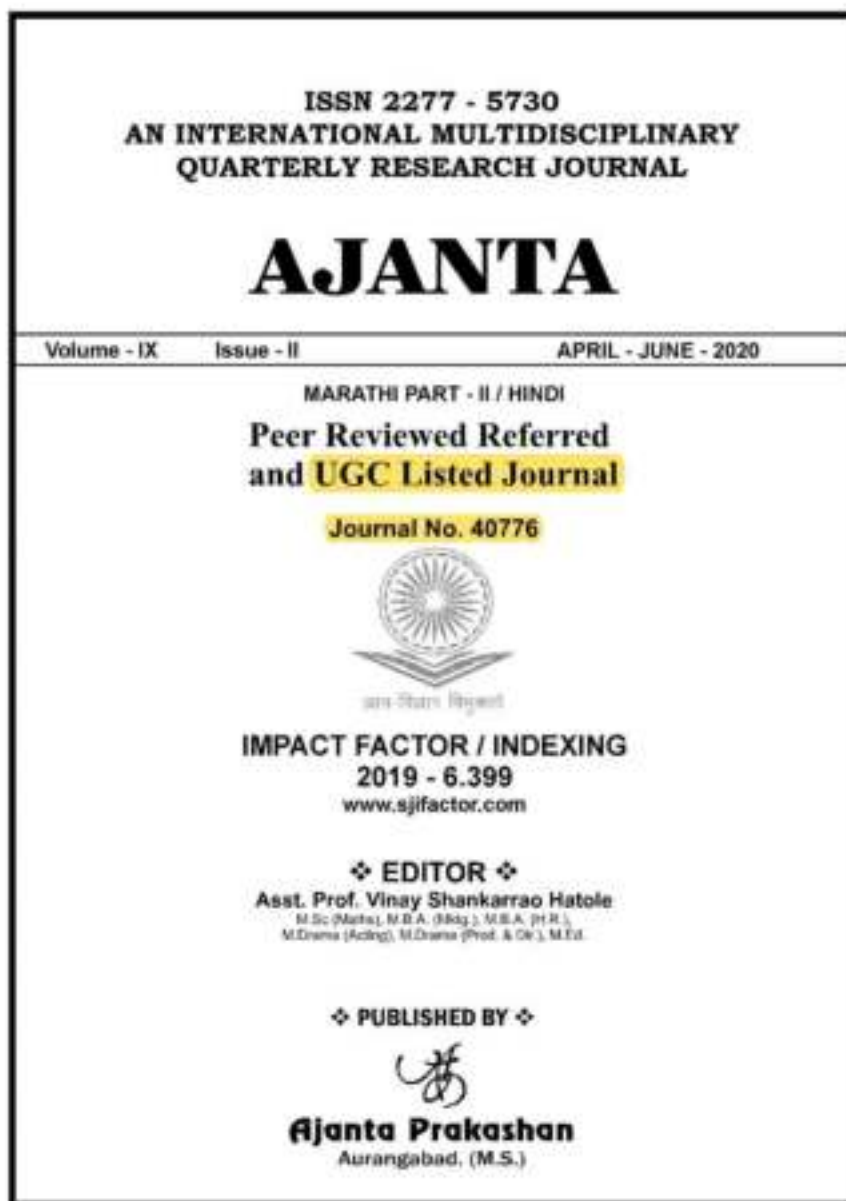
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Qureshi

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VOLUME - IX, ISSUE - II - APRIL - JUNE - 2020

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**CONTENTS OF HINDI**

अ.क्र.	लेख आर्षि लेखकाचे नाव	पृष्ठ क्र.
१	साम्यवाद का प्रधानस्रोत :- "ब्रीमद्वयवद्गीता" डॉ. अजित अशोक देव	१-५
२	अंबिकाब रावेन्द्र मिश्र के कथा साहित्य में पौराणिक शास्त्रीय ज्ञान डॉ. अशोक कैवर शिखावत	६-७
३	चिकित्सा और स्वास्थ्य का पारम्परिक ज्ञान कविता शर्मा	८-१४
४	मानवकल्याणाय संस्कृतज्ञानस्रोत डॉ. प्रशान्त कुमार सेठी	१५-१९
५	कौटिलीयवार्थशस्त्रे वर्णित समाजचित्रम् दुम्पा जान	२०-२५
६	कात्यायन श्रौतसूत्र में प्रतिपादित सामाजिक-समरसता डॉ. अरुणिमा	२६-३१
७	साहित्यशास्त्रीय परम्परा में भानुदत्त का योगदान डॉ. वन्सी केशवराव लव्हाडे	३२-३८
८	रामकालीन अर्द्धजन्मस्था - अर्द्धजन्मस्था की परंपरा के संदर्भ में डॉ. गीता भुल	३९-४२
९	सर्वदर्शन के प्रति आचार्य हरिचंद्र का योगदान श्रीलेश दत्तात्रय पवार	४३-४९
१०	अनुवाद के संदर्भ में संस्कृत शास्त्रों की भाष्य एवं टीका परम्परा का अवलोकन शुभम उनीयाल	५०-५४
११	भारतीय संगीत के मौलिक ग्रंथों में संस्कृत भाषा का महत्त्व डॉ. वैजाली संतोष देशमुख	५५-५८
१२	हिमालयस्थ परिस्थितिकारण दिव्योद्भवः सौरभ कण्ठवालः	५९-६३
१३	उपनिषद्की ज्ञानपरंपरा, निर्मित विचार तथा औपनिषदिक ज्ञानसे प्राप्त अंतर्दृष्टि कु. विष्णुषा पुरुषोत्तम नाईक	६४-७१

VOLUME - IX, ISSUE - II - APRIL - JUNE - 2020

AJANTA - ISSN 2277 - 5730 - IMPACT FACTOR - 6.399 (www.sjifactor.com)

❧ CONTENTS OF MARATHI PART - II ❧

अ.क्र.	लेख आणि लेखकाचे नाव	पृष्ठ क्र.
१	इसदृशिशिरोमनोषु स्वास्थ्यलक्षणम् । प्रा. डॉ. गणेशधर धर्मेष्ठरराव कऱ्हाळेकर	१-४
२	मानव जालीसाठी संस्कृतची ज्ञान संसाधने प्रा. जयश्री मुराजीलाल बनगे	५-१०
३	'विवेकानन्दविजयम्' महानाटकात वर्णित तत्कालीन सामाजिक परिस्थिती डॉ. भीमल श्रीगिरीवार	११-१६
४	गरुडपुराणे सामाजिक व्यवस्था - आचारः Dr. N. Shashidhar	१७-२३
५	संस्कृत साहित्यात 'अच्युतराव मोडक' यांचे योगदान डॉ. राधेश रंगनाथराव कुलकर्णी	२४-२७
६	वसुधैवकुटुंबम् प्रतिबिम्बितं वेदान्तचिन्तनम् Dr. Thiagarajan S. Dr. Vinod M.	२८-३३
७	सधुर्गतयेने संस्कृतसाहित्यज्ञानम् R. Rajalakshmi	३४-३८
८	वैदिक ज्ञान परंपरेतून मानवीय विकासधर्मे प्राप्ता झालेले वार्धक्य ज्ञान Dr. Poonam M. Gahukar	३९-४२
९	भारतीय ज्ञानपरम्परायाः ईश्वरविचारः (सांख्यदर्शनानुसारम्) बनश्री बेरा	४३-४९

८. वैदिक ज्ञान परंपरेतून मानवीय विकासाचे प्राप्त झालेले यथार्थ ज्ञान

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प्रस्तावना

भारतीय विचारसरणीनुसार वेद नित्य आहेत, अपौरुषेय आहेत, शाश्वत आहेत आणि म्हणूनच सर्व ज्ञानाचे व धर्माचे उगम स्थान आहेत. भारतीयोंची अशी धारणा आहे की सर्व ज्ञान हे वेदपासूनच उत्पन्न झाले – “सर्वज्ञानमयि हि सः।” वेद म्हणजे ज्ञान व ज्ञान सर्व शास्त्र, कला, विज्ञान, संगीत, आयुर्वेद, शिल्प वेदपासूनच उत्पन्न झाले. असे मानणे भाग पडतात. म्हणूनच जिज्ञासू संशोधनाची कीर्तने शोधण्यासाठी वैदिक साहित्याचा अभ्यास करतात व प्रमाण शोधतात. वैद्यक, गणित, साहित्य, ज्योतिष्य, फार काय तर स्वापत्य कला, पदार्थ विज्ञान, रसायन इत्यादी भौतिक शास्त्रे आपली परास्मिक अवस्था वेदा मध्ये निहित आहे असे सांगतात.

“विदन्ति ज्ञानन्ति विदन्ते भवन्ति विचारयन्ति सर्वे मनुष्याः सत्यं विद्याम यै वा तदा विद्वांसस्त भवन्ति ते वेदाः।” – अर्थात ज्याच्या द्वारे संपूर्ण सत्य विद्या गृह्य व परत्पत् केली जाते व ज्यामुळे विद्वानांना ज्ञान प्राप्त होते त्याला वेद असे म्हणतात. वैदिक साहित्याचा सर्व प्रथम वेद होय, वेदाचा अर्थ आहे – ‘ज्ञान !’ भारतीय संस्कृतीमध्ये याचे महत्वाचे व गौरव पूर्ण स्थान आहे. वेद शब्द विद धातुला घट्ट प्रत्यय जोडून बनलेला आहे.

“विदन्ते ज्ञानन्ते सभ्यन्ते वा धर्मादि पुरुषार्थ एभिर्विद वेदाः।” – अर्थात ज्याच्या द्वारे धर्म-अर्थ-काम-मोक्ष पुरुषार्थ जागणे व प्राप्त करणे हे यथार्थ ज्ञान सांगणारे ‘वेद’ होत.

वेदांचे महत्त्व

सृष्टी उत्पत्तीच्या वेळी धार्मिक, नैतिक व आध्यात्मिक ज्ञानासाठी याचा प्रादुर्भाव झाला. हे मानव जातीचे पचीनत धर्मग्रंथ होय म्हणूनच वेद त्याकाळातील संस्कृती, सभ्यता, धार्मिक मान्यता व परंपरा इत्यादींना जाणव्याचे एकमात्र स्रोत होय. वेद धर्माचे आधार स्तंभ आहे.

इहलौकिक व पारलौकिक दोन्ही प्रकारचे सुखप्राप्तीचे स्थान वेद आहे. हे उचित व अनुचित याचे निर्देशक आहे, कर्तव्याचे उद्देशक आहे. सुख शांतीचे साधन आहे. ज्ञान लोका चे व निराशेचे विनाशक वेदच आहे. भारतीय संस्कृती प्रमाणे वेद निंदकाला ‘नास्तिक’ म्हणतो अर्थात ‘नास्तिको वेद निंदकः ।’

वेद साहित्य दृष्टीनोमुद्रा अत्यंत महत्त्वपूर्ण आहे. महाकाव्य, गीतिकाव्य, मधकाव्य, माटक, अष्टाध्याय, साहित्य इ. काव्यांच्या सर्व विधा उत्पत्ति वेदाचे सक्रिय योगदान राहिले आहे.

वनस्पती विज्ञान असोपा सृष्टीविषयक वर्ता असो वा किंवा गीका निर्माण संबंधी कथा असो. सर्वांचे अंकुर वेदांमध्ये विद्यमान आहे.

नाथा विज्ञानाच्या दृष्टीने सुद्धा वेद अत्यंत महत्त्वपूर्ण आहे. वैदिक नाथाविना युरोपीय भाषांचे अध्ययन संभव नाही. वेदांचे उपदेश सार्वकालीन व सार्वभौमिक आहे.

मनुष्याच्या उन्नतीचे सर्वोत्तम साधन आहे ते भारतीयांचे आधारस्तंभ आहे.

वैदिक परंपरा व मानवीय विकास

वैदिक अध्ययन संदर्भात आजही विद्यार्थ्यांमध्ये प्राचीन धरोहर सुरक्षित आहे. एक विशाल यांचे स्मरण किंवा आर्य संस्कृती व प्राचीन, ऐतिहासिक, धार्मिक व लौकिक रूप ऋग्वेदातील विविध दैवत कथा प्राप्त होतात. आपली राष्ट्रीय एकता, अखंडता व नैतिक मूल्य यासाठी आजही सुद्धा ह्या देवकथा प्रसंगिक आहे. वैदिक दैवतकथा अर्जुनकालीन, अवतरकथा आहे. त्या आजही प्रभावशाली आहेत. वैदिक व लौकिक परंपरा या देवांचे नाव व त्यांची कार्यक्षेत्रां आपल्याला जरी भिन्न पसंतीत होत असले तरी उद्देश केवळ एकच लोककल्याण, देवत्व शक्ती मनुष्यासाठी उपादेय तत्त्व आहे.

“यदा यदा हि धर्मस्य ग्लानिर्भवति भारत ।

अभ्युत्थानमधर्मस्य तदात्मानं सृजाम्यहम् ।।”

या गीता वाक्यानुसार हे देव भविष्यात सुद्धा युग युगत जन्म घेतीलच, ज्यांच्या विचाराने संपूर्ण विश्व संस्कृती पुन्हा होईल व सर्वांची आत्मा शुभ संस्कारांनी संपन्न होत राहिल कारण संस्कारांच्या अभावी संस्कृती नाही. संस्कृती हि राष्ट्रचाच आत्मा होय.

आपल्या प्राचीन परंपरेत वेदांना ‘अ-पौरुषेय’ मानले आहे. वेद हे मनुष्यकृत रचना नाही ते अलौकिक आहे. कारण ब्रह्मज्ञानाच्या चार मुखानून चार वेद निघाले हे एक पौराणिक धारणेचे रूप आहे. परंतु वेदांच्या अ-पौरुषेयतेची सिद्धी मौर्यास शास्त्रज्ञ तर्क या आधारवर केली आहे – एक तथ्य असे कि महाभारत रामायण इ. ग्रंथांचा कर्ता याचे स्मरण किंवा उल्लेख केला जाऊ शकतो. त्याच परमाणे वेदांचा कर्ता कुणीच नाही.

कोणत्याही सत्य वाक्याला ‘वेदवाक्य’ म्हणतात. उदाहरणार्थ लौकिक ग्रंथ जसे रामायण, महाभारत इ. मध्ये पाठभेद मिळतात. परंतु वेदांमध्ये अतिप्राचीनता असूनही पाठभेद आढळत नाही हे एक महत्त्वपूर्ण तथ्य आहे की शिक्षा, व्याकरण सारखे षड् वेदांमधील वेदांना विकृत होण्यापासून वाचविले आहे व सम्यक ज्ञान लोकसंप्रेषित पोहोचविले. वास्तवधार्यांनी आवश्यकतेनुसार विचार व व्याख्यांच्या दृष्टीने वैदिक संप्रदाय साठवून केले ते अशा प्रकारचे आहेत.

१. नैकता:
२. याज्ञिक:
३. वैयाकरण:
४. ज्योतिष्य:
५. भाषाविद:
६. संप्रदायविद:
७. अध्यात्मविद:
८. ऐतिहासिक:

या संप्रदायांच्या माध्यमांनी वेदार्थ जाणव्याचा परंपरा वेदोतर काळत झाला.

वैदिक सृष्टीचे वेगवेगळे देव व ऋषी आहेत यातून स्पष्ट होते कि देवता स्तुत्य आहे, पृथ्वीनीय आहे. आपले कल्याण करणारे शक्ती स्वरूप आहे आणि स्तुती करता ऋषी त्या मंत्राचे दृष्टा होते, रचयिता मळे. काही ठिकाणी असे मंत्र उच्चरित आहे कि ऋषी म्हणतात हा आमचे मंत्र नाही, पूर्वी फाखून घेतले परंपरागत मंत्राचा आम्ही केवळ पाठ किंवा उच्चारण करित आहोत.

वैदिक परंपरा व 'श्रुति' हेच वेद आहेत जे परंपरेनी गुरुमुखातून श्रुत आहेत. सर्वात आश्चर्य हे कि विश्वातील विद्वानांना असे वाटते की वेद मंत्र उच्चारण व शास्त्र विधान वैज्ञानिक प्रतीशास्त्र नियमांनी त्यास अनुन्य बनवून ठेवणे यासाठी ऋषीमुनींना हाच शास्त्रीय नियम बनविण्याची आवशकता वासली. हे नियम पौरुषीय आहेत. ज्यांनी वेदांचे रक्षण केले आहेत. देवतांच्या चरित्रावर अधारित वैदिक आख्यान सुद्धा लोकत्वांनी वृत्त आहे. ईदरादी देवतांची स्तुती भौतिक जल्लुच्या पृथ्वी साठी वैदिक ऋषींनी त्यांची प्रार्थना केली. ऋग्वेदातील पृथ्वी लोकविज्ञान, परंपरा व नैतिक विचारांचे दर्शन होतल. भगवद्गीतेमध्ये भगवान श्रीकृष्ण म्हणतात -

"यश्चिराय साधूनां विनाशाय च दुष्कृताम् ।" ईदरांनी सुद्धा सज्जन लोकांच्या हितासाठी कार्य केले आणि लोकांना पीडा देणारे असुरांचा वृत्त रुपी शास्त्राने संहार केला. त्यामुळे स्पष्ट होते की लोक मंगलकारी देवतांचे चरित्र हे समाजातील लोकांसाठी अदरश भूत चरित्र आहेत ते मग वैदिक काळाचे असो किंवा पौराणिक काळचे.

'एकेन विज्ञाने सर्वम् विज्ञातं भवति' हे वेद वचन किती सार्थ आहे याचा अनुभव प्राचीन, अर्वाचीन शास्त्र निर्मात्यांना ध्यानधर्येत एकामागलेत येतो. भारतीय शास्त्रकारांनी ती अवस्था प्रत्यपूर्वक प्रत्य करून घेतली म्हणूनच अजोवर असलेले जगत्कारण हेच सर्व शास्त्रांचे मूळ आहे व हे वेद रुपी ज्ञान अपौरुषेय आहे हे ऋषी मुनी जाणतात. हे अनुभूतीने जाणलेले बंधार जरी अनेक असले तरी त्यातून प्रत्य विषय संकल्पाने पूर्ण होतो आणि

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एकेक विषयाचे एक शास्त्र निर्माण होते, नवे नवे शोध लागतल आणि मानवीय ज्ञान क्षेत्रे विस्तारु लागतात. जगतकारणांतून पुरण झालेले ज्ञान म्हणून सर्व ज्ञान शाखा परस्पर विरोधी वाटल तरी त्यांची मूळ समानता शोधाक्यास पाहिजे त्यातून विविध ज्ञान, विज्ञान शाखांचा विसर होतो. समानतेतून एकरूपता जाणवते व विरोध नाहीस होतो.

विदर्भ संत पूजा वधू शरी, गुलाबराव महाराजांनी शाश्वत वैदिक संस्कृती व परंपरा हि जगाच्या पाटीवरील सर्वप्रथम अस्तित्वात आलेली समृद्ध वाढलेली संस्कृती असून तीच जगभर पसरलेली होती असे साधार विषय मांडले व आर्याध्या इतिहासावर मवीन प्रकाश टाकला आहे. त्यांच्यानुसार वैदिक संस्कृति हि वैश्विक संस्कृती आहे. ह्या संस्कृतीचे पुनरुत्थान होऊन विशाला व्यापिल असे महाराजांचे ठाम मत आहे.

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String Source in Cosmological Model for Theory of Gravitation

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Abstract: We have investigated the cosmological model in presence of cosmic string source, for solving field equations in Brans-Dicke theory of gravitation for plane symmetric space time. It is observed that cosmological model exist. Some interesting physical and geometrical properties of the model are also discussed.

Keywords: Plane symmetric cosmological model, Cosmic string, Brans- Dicke theory, Tension density

1. Introduction

In the theory of gravitation, the plane symmetric model plays an important role. A space time that admits the three parameters groups of motion of the Euclidean plane is said to possess plane symmetry called plane symmetric space time. Einstein theory of gravitation serves as a basic for the construction of mathematical model of the universe. Cosmological model play an important role in understanding some essential feature of the universe such as the formation of galaxies during early stage of evolution. Brans-Dicke (1961) formulated a scalar tensor theory of gravitation in which gravity is mediated by a scalar field ϕ in addition to the usual metric tensor field g_{ij} present in the Einstein theory. In this theory the long range scalar field ϕ is generated by the whole of matter in the universe according to Mach's principle (Dicke 1964) and has the dimension of the universe of the gravitational constant G . The field equations in Brans- Dicke theory are

$$G_{ij} = -8\pi\phi^{-1}T_{ij} - \omega\phi^{-2}\left(\phi_{,i}\phi_{,j} - \frac{1}{2}g_{ij}\phi_{,k}\phi_{,k}\right) - \phi^{-1}(\phi_{,i}\phi_{,j} - g_{ij}\phi_{,k}\phi_{,k}) \quad (1)$$

$$\text{and } \square\phi = \phi_{,i}^{,i} = 8\pi\phi^{-1}T(3+2\omega)^{-1} \quad (2)$$

Where $G_{ij} = R_{ij} - \frac{1}{2}g_{ij}R$ is the Einstein tensor, T_{ij} is the stress energy of the matter, ω is the coupling constant, comma and semi comma denotes partial and covariant differentiation respectively.

The equation of motion

$$T^0_0 = 0 \quad (3)$$

are consequences of the field equation (1) and (2).

Some researcher there has been a lot of interest in cosmic strings and the cosmological models. Cosmic strings are one dimensional topological defects associated with spontaneous symmetry breaking, whose plausible production site is cosmological phase transitions in the early universe (Kibble, 1976 and Vilenkin 1985). Latelier (1983), Vilenkin (1981), Krori et al. (1990,1994) are some of the authors who have initiated the study of string cosmological models. Pimental (1996) (1997) studied inhomogeneous cosmological models in Brans Dicke scalar tensor theory and obtained exact solution which is conformally related to the non singular model recently found in general relativity. Rahman et al. (2001, 2002, 2003) have studied several aspects of cosmic string in Brans Dicke (1961), Saez-Ballester (1985) scalar tensor theories of gravitation and Lyra (1951) geometry.

2. Metric and Field Equations

We consider the plane symmetric space time

$$ds^2 = dt^2 - A^2(dx^2 + dy^2) - B^2 dz^2 \quad (4)$$

Where A and B are the functions of time t only.

The energy momentum tensor for the cosmic string is

$$T_{ij} = \rho u_i u_j - \lambda x_i x_j \quad (5)$$

Where ρ is the rest energy density of cloud of string with particles attached to them.

λ is the tension density of string

u^i is the cloud four velocity and

x^i is the direction of anisotropy

We have

$$u^i u_i = -x^i x_i = 1 \text{ and } u^i x_i = 0 \quad (6)$$

We consider

$$\rho = \rho_p + \lambda$$

Where ρ_p is the rest energy density of particle and x^i to be along z axis so that

$$x^i = (0, 0, A^{-1}, 0) \quad (7)$$

$$u^i = (0, 0, 0, 1) \quad (8)$$

The field equation (1), (2) and metric (4) for equation (5) with the help of equation (6), (7) and (8) can be written as

$$\begin{aligned} \frac{\ddot{A}}{A} + \frac{\ddot{B}}{B} + \frac{\dot{A}\dot{B}}{AB} &= -\frac{\omega}{2} \left(\frac{\dot{\phi}}{\phi} \right)^2 - \frac{\ddot{\phi}}{\phi} - \left(\frac{\dot{A}}{A} + \frac{\dot{B}}{B} \right) \frac{\dot{\phi}}{\phi} \\ 2 \left(\frac{\dot{A}}{A} \right) + 2 \left(\frac{\dot{A}}{A} \right)^2 &= 8\pi\phi^{-1}\lambda - \frac{\omega}{2} \left(\frac{\dot{\phi}}{\phi} \right)^2 - \frac{\ddot{\phi}}{\phi} - 2 \frac{\dot{A}}{A} \frac{\dot{\phi}}{\phi} \\ 2 \frac{\dot{A}\dot{B}}{AB} + \left(\frac{\dot{A}}{A} \right)^2 &= 8\pi\phi^{-1}\rho + \frac{\omega}{2} \left(\frac{\dot{\phi}}{\phi} \right)^2 - \left(2 \frac{\dot{A}}{A} + \frac{\dot{B}}{B} \right) \frac{\dot{\phi}}{\phi} \\ \left(2 \frac{\dot{A}}{A} + \frac{\dot{B}}{B} \right) \dot{\phi} + \ddot{\phi} &= \frac{8\pi\phi^{-1}}{(3+2\omega)} (\rho + \lambda) \\ \dot{\rho} + 2\rho \frac{\dot{A}}{A} + (\rho - \lambda) \frac{\dot{\phi}}{\phi} &= 0 \end{aligned} \quad (9)$$

Where the suffix (.) denotes ordinary differentiation with respect to time

Introducing the transformation,

$$A = e^{\alpha}, \quad B = e^{\beta}, \quad dt = A^2 B dT$$

$$\begin{aligned} \alpha'' - (\alpha')^2 + \beta'' - 2\alpha'\beta' + \frac{\omega}{2} \left(\frac{\phi'}{\phi} \right)^2 + \frac{\phi''}{\phi} - \frac{\alpha'\phi'}{\phi} &= 0 \\ 2\alpha'' - (\alpha')^2 - 2\alpha'\beta' + \frac{\omega}{2} \left(\frac{\phi'}{\phi} \right)^2 + \frac{\phi''}{\phi} - \frac{\beta'\phi'}{\phi} &= 8\pi\phi^{-1} e^{(4\alpha+2\beta)} \\ 2\alpha'\beta' + (\alpha')^2 - \frac{\omega}{2} \left(\frac{\phi'}{\phi} \right)^2 + 2 \frac{\alpha'\phi'}{\phi} + \frac{\beta'\phi'}{\phi} &= 8\pi\phi^{-1} e^{(4\alpha+2\beta)} \\ \phi'' = \frac{8\pi\phi^{-1}}{(3+2\omega)} (\rho + \lambda) e^{(4\alpha+2\beta)} \\ \rho' + 2\rho\alpha' + (\rho - \lambda)\beta' &= 0 \end{aligned} \quad (10)$$

Where the superscript primes indicates differentiation w. r. to T

3. String Cosmological Model

The set of field equation (10) being highly nonlinear, to get the determinate solution, we assume

$$\rho + \lambda = 0 \quad (11)$$

i.e. sum of rest energy density and tension density for a cloud of string vanishes.

Using equation (11), the set of equation (10) can be written as

$$2\alpha\alpha'' + 2\frac{\alpha'\phi'}{\phi} = 0$$

$$B'' + \frac{B'\phi'}{\phi} = 8\pi\phi^{-2}\rho x^{(2\alpha+2, \beta)}$$

$$\phi'' = 0 \quad (12)$$

$$\phi'' = 0$$

$$8\pi x^{(2\alpha+2, \beta)} = k_3$$

Which yield an exact solution given by

$$A = e^{\alpha} = (aT + b)^{1/2} \quad (13)$$

$$B = e^{\beta} = e^{\int \frac{1}{(aT+b)} \left(\frac{1}{(2\alpha+2)} \frac{d}{dT} \left(\frac{1}{(aT+b)} \right) \right) dT} e^{f(T)}$$

$$\phi = aT + b \quad (14)$$

$$\therefore 8\pi\rho = -8\pi\lambda = k_2 e^{\left[\frac{-2k_3}{(2\alpha+2)} \frac{d}{dT} \left(\frac{1}{(aT+b)} \right) \right]} \left(\frac{1}{(aT+b)} \right)^{2\alpha+2} \quad (15)$$

Where constants $a \neq 0$, $b \neq 0$ and k_i , $i = 1, 2, 3$ satisfy the relation

$$(k_1 k_2 + k_2 k_3 + k_1 k_3) + a(k_1 + k_2 + k_3) - \frac{\omega}{2} a^2 = 0 \quad (16)$$

Using equation (5) and (10), plane symmetric cosmological model for string source corresponding to above solution can be written as

$$ds^2 = e^{\left[\frac{-2k_3}{(2\alpha+2)} \frac{d}{dT} \left(\frac{1}{(aT+b)} \right) \right]} e^{f(T)} \left[(aT+b)^{\frac{1}{2}} dT^2 - dx^2 \right] - (aT+b)^{\frac{2\alpha}{2}} (dy^2 + dz^2) \quad (17)$$

Where the rest energy density of the cosmic string and scalar field in the model are given by the equation (14), (13) respectively

4. Some physical properties

The plane symmetric string cosmological model in Brans Dicke theory is represented by (16) when the sum of tension density λ and the rest energy density ρ of the cosmic

string vanishes. The model has no initial singularity. The scalar field ϕ given by the equations (14) and the energy density and tension density of the string given by equation (15) in the model (17) are free from initial singularities. The spatial volume of the model is given by

$$V = \sqrt{-g} = (aT + b)^{\frac{3k_1 + 2k_2}{\alpha}} e^{\left[\frac{2k_1}{12k_1 + 1} (aT + b)^{\frac{2k_1 + 1}{\alpha}} \right]} \quad (18)$$

This shows the anisotropic of the universe (17) with time $k_1 > 0$

In order to obtain more clear and simple behavior of the physical kinematical variables with their simple explicit expressions, we choose the constants as

$$k_1 = k_2 = k_3 = 1 = \omega = b; \quad \alpha \neq 0, \quad b \neq 0 \quad (19)$$

Therefore the following expressions for the parameters are

$$\text{Spatial volume } V = e^{\left[\frac{2}{3}(T+1)^2 \right]} T + T^2 \quad (20)$$

$$\text{Expansion scalar } \theta = \frac{1}{3} u^\mu_{;\mu} = \left[\frac{1}{T+1} + \frac{(T+1)^2}{9} \right] \quad (21)$$

Shear scalar

$$\sigma = \frac{1}{2} \sigma_s \sigma^s = \frac{1}{2} \left[\frac{1}{6} \left(1 - \frac{2}{e^{\frac{2}{3}(T+1)^2 T + T^2}} + \frac{1}{e^{\frac{2}{3}(T+1)^2 T + T^2}} \right) \right] \left[\frac{1}{3(T+1)} + \frac{(T+1)^2}{27} \right]^2 \quad (22)$$

$$8\pi p = -8\pi \lambda = e^{-\left[\frac{2}{3}(T+1)^2 T + T^2 \right]} \quad (23)$$

$$8\pi p_s = 2 e^{\left[\frac{2}{3}(T+1)^2 T + T^2 \right]} \quad (24)$$

The scalar field ϕ in the model takes the form

$$\phi = T + 1 \quad (25)$$

The energy condition shows that $\rho > 0$ and $\rho_s > 0$. The rest energy density ρ , string tension density λ and the particle density ρ_s tends to zero as time T increases indefinitely. The spatial volume tends to infinity and expansion scalar θ and shear scalar σ tends to zero as $T \rightarrow \infty$. The model (17) can be considered as an inflationary model of string cosmology in Brans Dicke theory of gravitation. From equation (15), it can be observed that for $k_3 = 0$, $\rho = \lambda = 0$ and the model (17) degenerates into the singularity free plane symmetric vacuum model in Brans Dicke theory.

Conclusion:

We considered the plane symmetric metric and Brans Dicke (1961) field equation of string. While solving the field equation, we have assumed that sum of the energy density and tension density of the massive string is zero. The cosmological model obtained, which represent an inflationary plane symmetric string cosmological model in Brans Dicke theory of gravitation.

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Enhanced thermoluminescence properties of $\text{CaSrAl}_2\text{SiO}_7: \text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor :
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Enhanced thermoluminescence properties of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor

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ABSTRACT

The application of thermoluminescence technique in radiation dosimetry spans field of health physics, biological and geological sciences and personnel monitoring; this led to the search for new compositions with desirable dosimetric properties. In the present work, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphores were prepared by solid-state reaction method and their TL properties were studied in detail. The comparison of their TL results showed that co-doping of Ce^{3+} ions enhanced TL response of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphors; this was also verified from measurement of TL emission spectra of the samples. Optimized glow curves were analysed and TL parameters were extracted from Chen's method. Co-doped $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor is found to be useful in dosimetric application.

1 Introduction

When an insulating or a superconducting material is exposed to any kind of ionizing radiation, deposited energy is stored in the defect sites and colour centres of the crystal lattice. Due to action of heat energy, a fraction of this stored energy released and emitted as visible light which is called thermoluminescence [1, 2]. Nowadays application of various radiations such as ultraviolet, X-rays, β -rays, γ -rays in the different fields like medical, industrial, agriculture, etc., is increasing [3]. Thermoluminescence (TL) is one of

the techniques used in radiation dosimetry [4]. Thermoluminescent materials are more investigated in on-going researches, because they found to have increasing application in thermoluminescence dosimeters. Thermoluminescent dosimeters necessarily have linearity of TL response with exposed radiation dose.

$\text{CaSrAl}_2\text{SiO}_7$ is one of the members of melilite group; these melilites that are basically silicate-based materials showed their use in TL dosimetry. In this work, thermoluminescence properties of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphor are investigated and also

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observed that co-doping of Ce^{3+} ions enhanced TL of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphor. The effect of different Tb^{3+} concentration in both single and co-doped samples on the TL glow curves has been recorded and glow curve is analysed by computerized glow curve deconvolution method. To the best of our knowledge, there is no earlier report on thermoluminescence investigation on $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors.

2 Synthesis and experimental

Phosphors for the study were prepared by solid-state reaction method at high temperature using analytical reagent (AR) grade raw chemicals with more than 99% accuracy. Calcium carbonate, strontium carbonate, aluminium oxide, silicon di-oxide, ceric oxide and terbium oxide were the starting materials. The amounts of the raw chemicals were calculated according to stoichiometric ratio in $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%), Tb^{3+} (y mol%), where $y = 1.0, 3.0, 5.0, 7.0, 10.0$ mol%. Required quantities of raw materials were mixed homogeneously using agate mortar and pestle for 3 h. The resultant mixtures were fired at high temperature 1300°C for 5 h. It was then slowly cooled to room temperature inside the closed furnace. The powders so formed were collected by crushing the prepared samples. Now the samples were ready for all other characterization studies.

Phase of synthesized phosphor was confirmed by X-ray diffraction (XRD) technique using $\text{Cu-K}\alpha$ radiation with the help of D2 phaser Bruker diffractometer. Thermoluminescence investigation of phosphor was performed by TLD reader, Nucleonix TL 1009f with constant heating rate of 5°C/s . As sample must be exposed to any kind of radiation before measurement of TL, UV cabinet is used, where the samples were exposed to UV radiation of 254 nm. Band pass filters of different wavelengths have been used to record TL emission spectrum. To record TL emission spectrum, the processes used in recording TL glow curves were repeated; here the sample that is placed in canthal strip was covered with band pass filter. The band pass filter allows the signals of selected range of wavelengths; hence in TL emission spectrum, characteristic emission peaks were observed.

3 Results and discussion

3.1 XRD investigation

To verify the preparation of phosphors, they were analysed by XRD tool that reveals the phase composition. Figure 1 shows the XRD patterns of Tb^{3+} singly doped and $\text{Ce}^{3+},\text{Tb}^{3+}$ co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphors along with JCPDS file. It is clear that phosphors have good crystallization with no any impurity phase, as experimental diffraction patterns well matched with JCPDS file 26-0327 of $\text{CaSrAl}_2\text{SiO}_7$. Hence, the as-prepared phosphors have tetragonal crystallographic structure whose space group is $I4_2m$ [5].

3.2 Thermoluminescence investigation

Thermoluminescence (TL) is light emission due to moderate heating of a solid, previously exposed to ionizing radiation. The absorption of the ionizing radiation (UV, X-, β - or γ -rays, high-energy particles) creates traps, and fills them (or those already exist) with electrons and/or holes even at low temperatures. The subsequent heating migrates the electrons

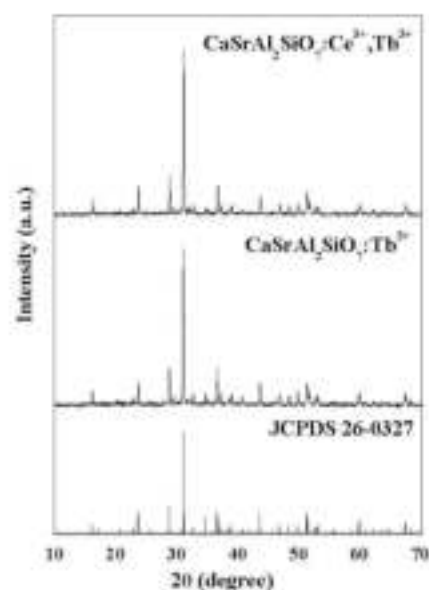


Fig. 1 Diffraction patterns of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors

and/or holes in the lattice, until they fall into other traps (or recombine) with a consequent photon emission [6]. Thermoluminescent emission from TL materials is very sensitive to the amount and nature of dopant element and radiation effect. So in the present research work, we used Ce^{3+} and Tb^{3+} as single and co-dopant ions with their various concentrations; after that TL measurement was performed for each sample with optimized dopant and co-dopant concentration with various ultraviolet (UV) exposure time and then TL kinetic parameters were extracted from the Chen's peak shape method.

3.2.1 Thermoluminescence of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$

Let us first focus on the investigated thermoluminescence properties of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ phosphor; this was already discussed in our previous work in which Ce^{3+} concentration was varied as 0.1, 0.3, 0.5, 1.0, 2.0, 3.0 and 4.0 mol% [7]. The optimized glow curve was obtained for 0.5 mol% Ce^{3+} -doped $\text{CaSrAl}_2\text{SiO}_7$ phosphor. This optimized phosphor showed an increment in TL intensity up to 35 min of UV irradiation time. As we further increased UV exposure time, saturation in TL intensity was found.

3.2.2 Thermoluminescence of $\text{CaSrAl}_2\text{SiO}_7\text{:Tb}^{3+}$

At the second attempt, TL properties of $\text{CaSrAl}_2\text{SiO}_7\text{:Tb}^{3+}$ phosphor was seen and this report is dealing here for the first time. Figure 2 represents a series of Tb^{3+} concentration (1.0, 3.0, 5.0, 7.0, 10.0 mol%)-dependent TL glow curves of $\text{CaSrAl}_2\text{SiO}_7\text{:Tb}^{3+}$ samples. TL peak position is independent of the concentration of Tb^{3+} element and exhibits no shift with change in doping concentration. The only variation obtained in the TL peak intensity while increasing Tb^{3+} concentration. It can be understood from the inset of Fig. 2 that TL intensity of sample first increased with rise in Tb^{3+} concentration, attained a highest value at 5.0 mol% of Tb^{3+} ions and then decreased with further increase in Tb^{3+} concentration; this behaviour arises from concentration quenching process after a particular Tb^{3+} concentration. As we increase impurity concentration, there is an increase in the number of defects/traps which in turn implies a growth in the density of charge carriers being trapped upon irradiation. Therefore the initial rise in the TL peak intensity or area of the glow curves. Furthermore, on being thermally stimulated,

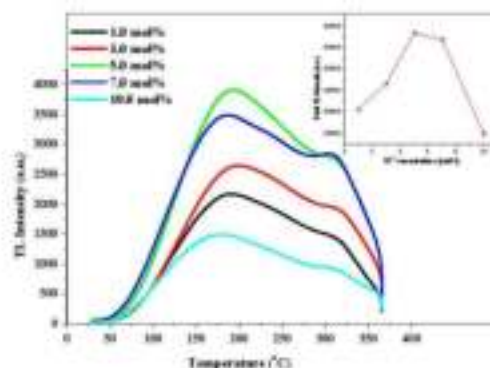


Fig. 2 Concentration-dependent TL glow curves of $\text{CaSrAl}_2\text{SiO}_7\text{:Tb}^{3+}$ phosphors with 10 min UV exposure time

these charge carriers release from traps which in turn recombine with their counterparts at the recombination centre and yield diverse TL glow peaks with elevated height [8].

As thermoluminescence properties are greatly affected by exposure time, TL property of $\text{CaSrAl}_2\text{SiO}_7\text{:Tb}^{3+}$ (5.0 mol%) phosphor was measured with different UV exposure time. For variable UV exposure time, some of the selected TL glow curves of $\text{CaSrAl}_2\text{SiO}_7\text{:Tb}^{3+}$ (5.0 mol%) are represented in Fig. 3. Spectral behaviour was same with increase in length of UV exposure time but peak TL intensity or glow curve area altered with increase in exposure time. The growth in TL response was observed as exposure time was boosted and after 60 min of UV exposure TL response diminished with more added exposure time. With increasing irradiation dose more and more trapping centres or luminescent centres responsible for the TL glow peaks are getting filled. Upon thermal stimulation, these traps liberate their charge carriers and they get recombine with their counterparts, giving rise to different glow peaks. When all the trapping centres that are subjected to desired TL emission get filled, the saturation or decrease in the TL intensity starts appearing [8–10].

3.2.3 Thermoluminescence of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}\text{:Tb}^{3+}$

Thermoluminescence experiments were performed on the series of $\text{CaSrAl}_2\text{SiO}_7\text{:Ce}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7\text{:Tb}^{3+}$ phosphors that the results point out optimal concentration of Ce^{3+} as $x = 0.5$ mol% and of Tb^{3+} as $y = 5.0$ mol%. In the present work, co-doping

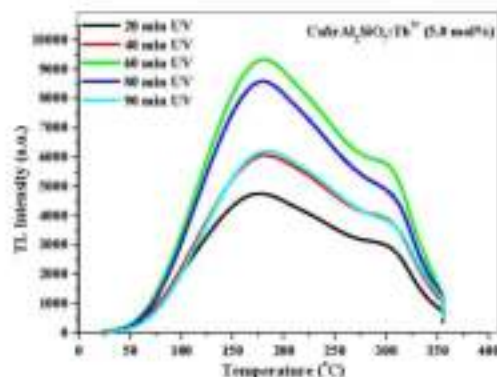


Fig. 3 Effect of UV exposure time on the TL glow curve of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) phosphor

of Ce^{3+} and Tb^{3+} was also performed in $\text{CaSrAl}_2\text{SiO}_7$ host and TL properties of this co-doped sample were also investigated and compared with TL of singly doped samples. Co-doping was performed with fixed concentration of Ce^{3+} ($x = 0.5$ mol%) and variable concentration of Tb^{3+} ($y = 1.0, 3.0, 5.0, 7.0, 10.0$ mol%).

Figure 4 shows TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ ($x = 0.5$ mol%), Tb^{3+} ($y = 1.0, 3.0, 5.0, 7.0, 10.0$ mol%) phosphors with fixed UV irradiation time. Glow curves for all samples had similar shape but with change in glow curve area. Concentration quenching occurred at 5.0 mol% of Tb^{3+} ions (see inset of Fig. 4). Like Ce^{3+} and Tb^{3+} singly doped samples, impact of various UV irradiation time on the TL glow curve of optimized co-doped $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%), Tb^{3+} (5.0 mol%) sample was seen and this result is depicted in Fig. 5. It can be clearly seen from Fig. 5 that TL intensity of optimized co-doped phosphor increased up to 100 min of UV exposure and then decreased.

3.2.4 Evaluation of TL parameters

To determine TL parameters, broad glow curves of 60-min UV exposed $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%) and 100-min UV exposed $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%), Tb^{3+} (5.0 mol%) samples were first applied to computerized glow curve deconvolution (CGCD) method. Both samples were found to have four overlapping peaks in their parent glow curves. The position of constituent peaks is shown in Fig. 6. TL parameters like geometrical shape factor (s_g),

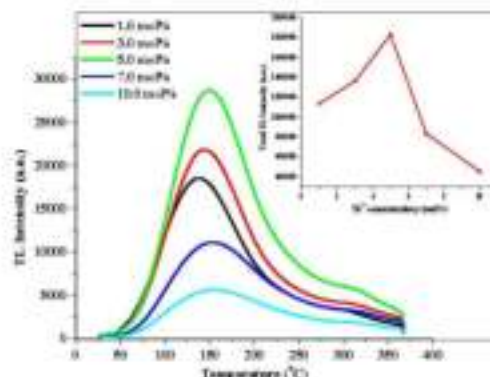


Fig. 4 Tb^{3+} concentration-dependent TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor with 30-min UV exposure time

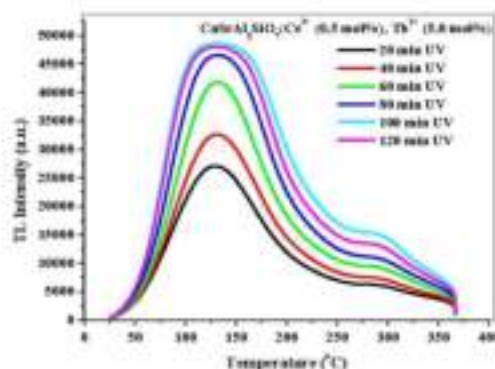


Fig. 5 Effect of UV exposure time on the TL glow curve of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphor

order of kinetics (b), frequency factor (S) and activation energy (E) were extracted using Chen's peak shape method. The calculated TL parameters of Tb^{3+} -doped and $\text{Ce}^{3+}, \text{Tb}^{3+}$ co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphors are listed in Table 1.

3.2.5 TL emission spectra

Figure 7 represents comparison of TL emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphors. Emission spectra were recorded using interference band pass filters of various wavelengths from 400 to 700 nm. Emission spectra of Ce^{3+} -doped sample was already discussed in our previous article [7], here it is shown for comparison only. The three samples have characteristic

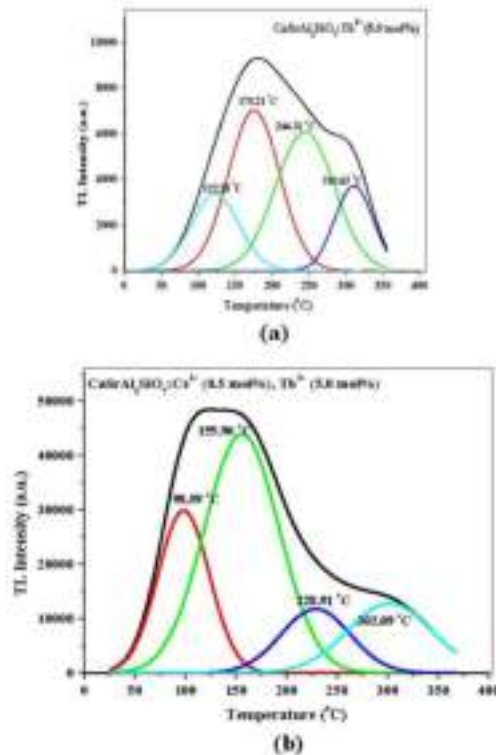


Fig. 6 Representation of peak deconvolution on parent glow curve of a $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and b $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphors

emission peaks of respective dopant and co-dopant ions. $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ sample has emission peaks at around 480, 545 and 590 nm these are due to characteristic transition $^5\text{D}_4 \rightarrow ^7\text{F}_6$, $^5\text{D}_4 \rightarrow ^7\text{F}_5$ and $^5\text{D}_4 \rightarrow ^7\text{F}_4$ of Tb^{3+} ions [11]. $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ sample has characteristic peak of Ce^{3+} ions at around 420 nm, due to $5d (^5\text{D}_{3/2}) \rightarrow 4f (^5\text{F}_{5/2})$ [12, 13] and

Table 1 Calculated TL parameters

Phosphor	T_m (°C)	μ_g	b	E (eV)	S (s^{-1})
$\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ (5.0 mol%)	122.28	0.48	1	0.46	8.07×10^6
	175.21	0.48	1	0.57	2.45×10^7
	244.31	0.49	1	0.75	1.82×10^8
	310.63	0.70	2	1.41	2.12×10^{13}
$\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$ (0.5 mol%), Tb^{3+} (5.0 mol%)	98.59	0.49	1	0.54	2.35×10^6
	155.96	0.5	2	0.53	1.4×10^7
	228.91	0.49	1	0.78	7.45×10^8
	302.09	0.49	1	0.82	1.37×10^8

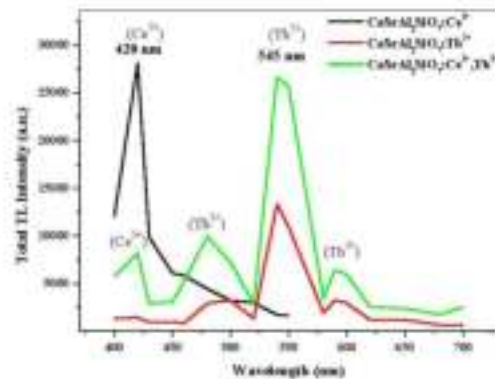


Fig. 7 TL Emission spectra of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ phosphors

characteristic peaks of Tb^{3+} ions situated at around 480, 545 and 600 nm; these are due to above-mentioned transitions of Tb^{3+} ions.

Figure 8 shows the comparative TL glow curves of the three optimized phosphors. $\text{Ce}^{3+}, \text{Tb}^{3+}$ co-doped phosphor has most intense TL properties among all three. Also from Fig. 7 it is clearly visible that TL emission of cerium ions is decreased, while TL emission of terbium ions is increased in co-doped sample; hence co-doping of small amount of Ce^{3+} ions, i.e., 0.5 mol%, may enhance TL emission of Tb^{3+} ions in $\text{CaSrAl}_2\text{SiO}_7$ host.

Present study concludes that among the three, $\text{Ce}^{3+}, \text{Tb}^{3+}$ co-doped $\text{CaSrAl}_2\text{SiO}_7$ phosphor has almost linear increment in total TL intensity with respect to UV exposure time up to 180 min, which is very large range as compared to other UV irradiated silicate, aluminate and aluminosilicate-based phosphors such as $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Ce}^{3+}, \text{Tb}^{3+}$ [14]; $\text{Ca}_2\text{Al}_2\text{SiO}_7:\text{Ce}^{3+}$ [15]; $\text{BaMgAl}_{10}\text{O}_{17}:\text{Ce}^{3+}$ [16]. Hence

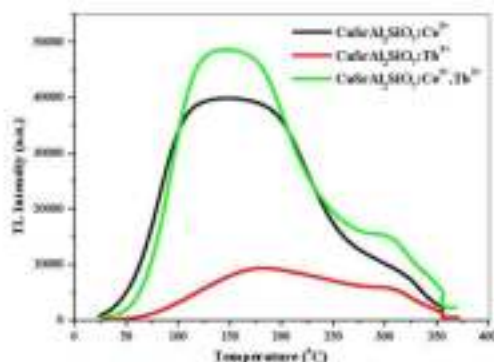


Fig. 8 Comparative TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}$, $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors

$\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphor may be most appropriately useful for UV dosimeter application.

4 Conclusions

$\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors were prepared by solid-state reaction method. Detail comparative TL study of singly doped and co-doped samples was investigated. Concentration-dependent TL glow curves of $\text{CaSrAl}_2\text{SiO}_7:\text{Tb}^{3+}$ phosphor and $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+},\text{Tb}^{3+}$ phosphors were measured and found that 5.0 mol % is optimized concentration of Tb^{3+} ions in both samples. Tb^{3+} singly doped sample showed linear increment in TL intensity up to 60 min, while $\text{Ce}^{3+},\text{Tb}^{3+}$ co-doped sample showed this linearity up to 100 min, also TL properties of co-doped sample got enhanced with inclusion of 0.5 mol% of Ce^{3+} ions, this was also verified from TL emission spectra. Hence the optimized $\text{CaSrAl}_2\text{SiO}_7:\text{Ce}^{3+}(0.5 \text{ mol}\%),\text{Tb}^{3+}(5.0 \text{ mol}\%)$ phosphor may be a potential candidate for UV dosimeter application.

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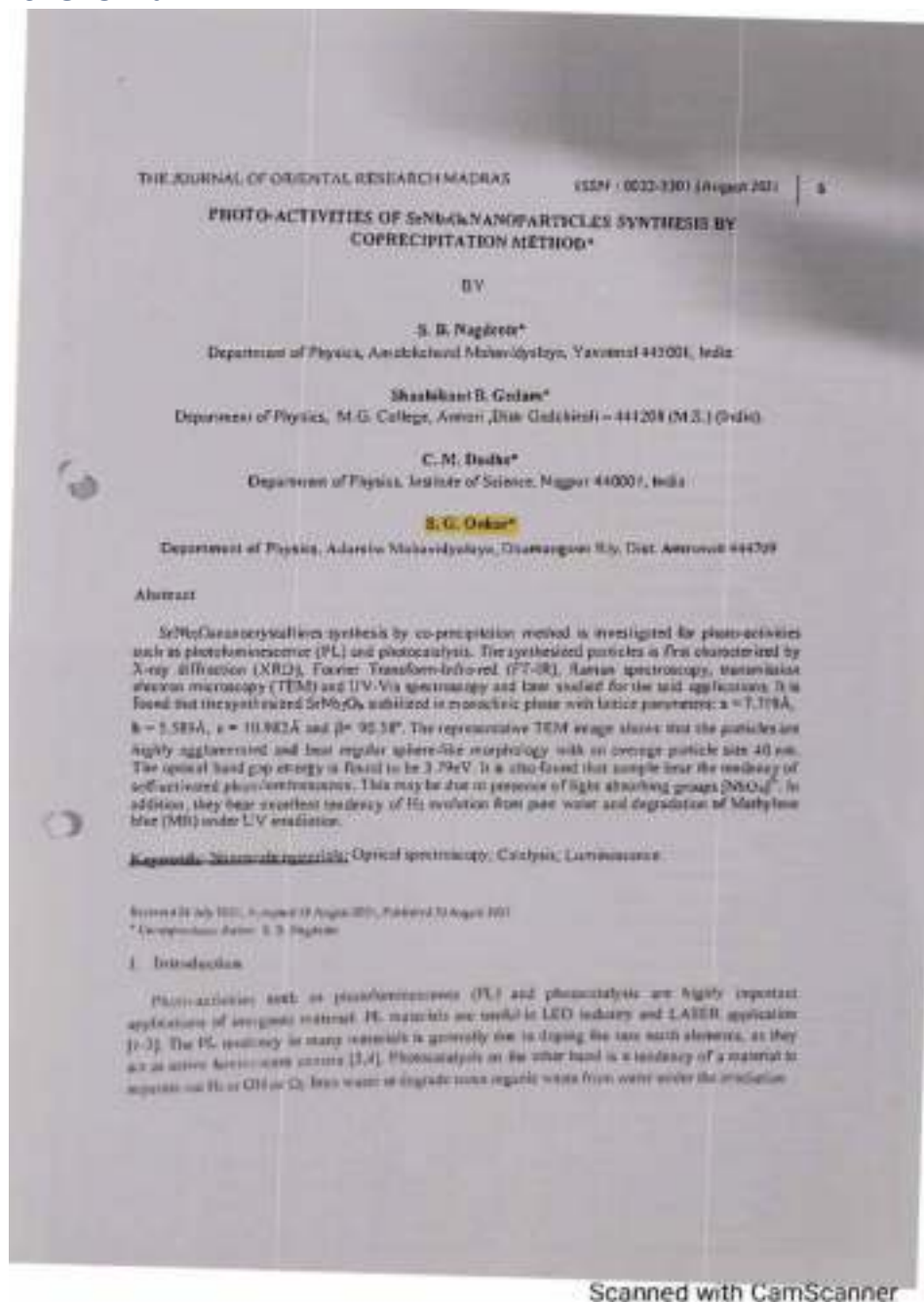
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PHOTO-ACTIVITIES OF SrNb₁₀O₆ NANOPARTICLES SYNTHESIS BY COPRECIPITATION METHOD: S. G. Onkar



of UV or Visible light. This tendency will act as a powerful tool for the generation of hydrogen energy and for controlling the environmental issues [5-8].

The literature survey shows that alkaline earth niobates $A^{2+}Nb_2O_6$ ($A^{2+} = Ca, Sr, Ba$) bear tendency of PL and photocatalysis without doping rare earth elements [5-7]. The PL in such cases may be termed as self-activated luminescence. Fundamentally, self-activated PL and photocatalysis are the photogenerated charge-dependent phenomena. The photogenerated electron which is excited from VB to CB will return back to VB where it recombines with the hole [8] and during its return journey emits the energy as a PL. On the other hand, photocatalysis reaction occurs on the surface of the particle. In photocatalysis, the electrons in CB trap by molecules of the solution before the recombination. This means, if the recombination rate is high the PL intensity will be more and the photocatalytic activity will be low [8].

$SrNb_2O_6$ is an important member of alkaline earth niobates, stabilizes mostly in Columbite phase. In comparison with the other columbite phase compounds it possesses interesting dielectric relaxation properties [9], and has relatively higher dielectric constant (ϵ_r) with low dielectric loss [10]. It was also studied for photoluminescence and photocatalysis [5]. However, these properties might be influenced by the method of preparation of the particles, crystal structure and size of the particles [11].

As far as the synthesis of $SrNb_2O_6$ nanoparticles is concerned, various techniques such as electrochemical [12], sol-gel combustion [13], hydrothermal [14], solid state reaction [3] coprecipitation [15] methods are reported. According to Ravi [15] a good quality of $SrNb_2O_6$ nanoparticles can be prepared by coprecipitation method because of better composition control, better particle morphology control and good powder reactivity. Therefore, in the present paper we preferably picked up coprecipitation technique. However, a modification in terms of reheating the sample at an elevated temperature has been made in order to introduce oxygen vacancies [16]. The oxygen vacancy seems to be necessary to distort the NbO_6 octahedra which after distortion may act as a center for self-activated luminescence. At the same time care was taken to maintain the nanocrystalline nature of the compound. So, the reheating temperature was maintained around 800°C. The higher temperature can increase the size of the particles and will hinder the photocatalytic properties of the compound.

2. Material and Methods

2.1 Material synthesis

AR grade chemicals such as $SrCl_2 \cdot 6H_2O$, Nb_2O_5 , ammonium oxalate and ammonium hydroxide were used for the synthesis of monoclinic $SrNb_2O_6$ nanoparticles. As mentioned in the introduction section, a coprecipitation method was used for the preparation of orthorhombic $SrNb_2O_6$ nanoparticles. The details of the procedure are already given elsewhere [13]. The obtained precursor was calcined at first at 650°C for 4 hrs to get a fine powder. It was then reheated at 800°C for another 6h.

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2.2 Characterization methods

X-ray diffractions (XRD) data of above mentioned synthesized samples of SrNb_2O_6 were collected by using an X-ray diffractometer (D8 ADVANCE, BRUKER, GERMANY) employing $\text{Cu-K}\alpha$ radiation ($1.54\text{\AA}$) with the step size of 0.02° . Indexing of the peaks pattern in monoclinic phase was done by the least squares fit method using the structural formula [17]

$$\frac{1}{d_{hkl}^2} = \frac{h^2}{a^2 \sin^2 \beta} + \frac{k^2}{b^2} + \frac{l^2}{c^2 \sin^2 \beta} - \frac{2hl \cos \beta}{ac \sin^2 \beta} \quad (1)$$

Where h, k, l are the miller indices, a, b, c and β are the lattice parameters, and d_{hkl} is the inter planer spacing.

Transmission electron microscopy (TEM) (TECNAI G2 20 ULTRA-TWIN, FEI, NETHERLANDS) was employed to obtain images of as prepared particles. These images were used for the estimation of particle size. The average size of the particles was also estimated from the XRD peaks by using Scherrer relation

$$D = \frac{kl}{d \sin \theta} \quad (2)$$

where, D is the average grain size, λ is the X-ray wavelength, θ is the peak angle, k is equal to 0.89 for spherical particles, and d is the full width at half the maxima in radian.

FT-IR spectrum of the synthesized SrNb_2O_6 was obtained by using a FT-IR spectrometer (THERMO NICOLET, AVATAR 370, USA).

Raman spectra of the synthesized compound was recorded in the shift range of $0-1000\text{cm}^{-1}$ by using laser Raman spectrometer (LIRA-300, LAMBDA, AUSTRALIA) having wavelength 532nm . For this measurement, the pellets were prepared by mixing the synthesized powder with a few drops of 1 wt% solution of polyvinyl alcohol (PVA) and pressed isostatically under the pressure of 5-6 t for 5 min. finally they were heat-treated at 800°C for 5 h to make them PVA-free.

UV-Vis spectrum of as prepared SrNb_2O_6 pellet was recorded with reference to the mirror by using a spectrometer (UV-VIS 1700, SHIMADZU, JAPAN). For analysis, it was then converted into Kubelka-Munk diffuse spectrum by using a relation [18,19],

$$[F(R_\infty)h\nu]^{1/2} = C(h\nu - E_g) \quad (3)$$

where, C is a constant, and $n = 1/2$ for the estimation of optical direct band-gap energy.

Function $F(R_\infty)$ is called Kubelka-Munk function or remission and expressed as,

$$F(R_\infty) = \frac{K}{S} = \frac{(1-R_\infty)^2}{2R_\infty} \quad (4)$$

where, S and K are the Kubelka-Munk scattering and absorption coefficients respectively.

The limiting reflectance (R_∞) is defined as,

$$R_\infty = \frac{P_{\text{sample}}}{P_{\text{reference}}} \quad (5)$$

From the extrapolation of the linear region of K-M diffuse spectrum the value of optical band-gap energy was determined. A spectro fluorimeter (F-7000, HITACHI, JAPAN) was used to study the self-activated photoluminescence spectrum of the sample.

The photocatalytic activities of the synthesized SrNb_2O_6 nanocrystalline powder were examined by the evolution of H_2 from pure water at room temperature and by the degradation of methylene blue. A 200W high-pressure ultraviolet lamp was used as the source of radiation. For the first part, the suspension of catalyst in water was prepared by adding 50mg SrNb_2O_6 nanoparticles in 200ml pure water with constant stirring for 30min. This suspension was then poured into a photocatalytic reactor which is made up of a thick glass and exposed under UV-radiations with continuous stirring. The source was maintained at a distance of 12cm from the reactor. The temperature of the reactor was maintained at constant room temperature by using water-circulation arrangement. The amount of H_2 gas evolved out from the suspension was measured by using a measuring cylinder fully filled with water and placed upside down in a jar filled with water.

For the next part, 50 mg of photocatalyst was added into 100 ml of (1ppm) methylene blue (MB) solution, it was stirred well and exposed under the same UV-Vis light from 12cm. After every 10 minutes about 1 ml of MB solution was taken out from the reactor. By removing catalyst using centrifuge action, the UV-Vis spectra was recorded by using the spectrometer (Labindia UV 7000*, UV-Vis).

3. Results and Discussion

3.1 Structural, morphological and optical

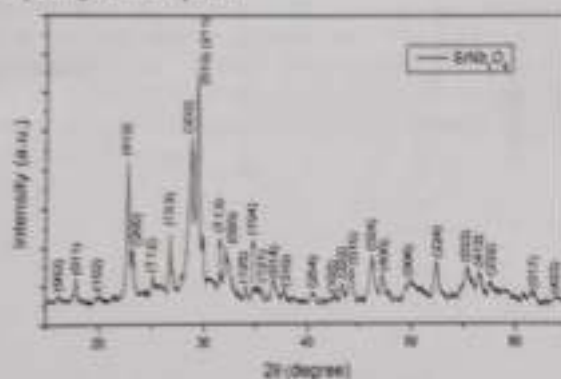


Fig. 1 XRD patterns of the synthesized SrNb_2O_6 nanoparticles.

Fig. 1 is the XRD pattern of as prepared SrNb_2O_6 compound. The peak pattern shows good agreement with the JCPDS file no. 772431, clearly indicates that the compound stabilized in monoclinic phase. The indexing of peaks and cell parameters were computed by Eq (1). The cell parameters were found to be: $a = 7.719\text{\AA}$, $b = 5.589\text{\AA}$, $c = 10.982\text{\AA}$ and $\beta = 90.38^\circ$.

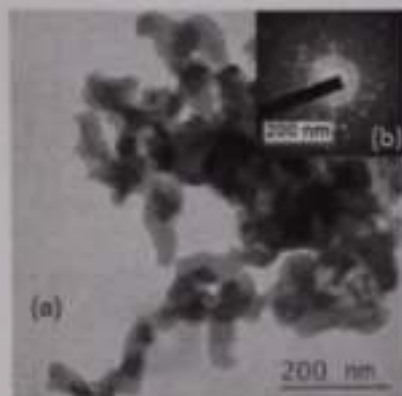


Fig.2(a) TEM image and (b) SAED image of SrNb_2O_6 nanoparticles.

Fig. 2a is the representative TEM image of as prepared SrNb_2O_6 compound. It seems that the particles are agglomerated and bear irregular sphere-like morphology. The average particle size was found to be $\sim 40\text{nm}$. Its crystalline nature was confirmed by SAED image and XRD. The SAED image of as prepared SrNb_2O_6 is shown in Fig. 2b. It consists of circular rings with bright spots. Such type of patterns, in general, can be assigned to nano-crystalline nature of the material [17]. Similarly, a fine XRD peaks pattern as appeared in Fig. 1 confirms the crystallinity of the particles. The size of the particles estimated from (013), (202), (012), (024), (224) and (033) XRD peaks using Scherrer formula (Eq. 2) was found to be $\sim 25\text{nm}$. This value is smaller than the size estimated from TEM, may be due to agglomeration of the particles [15, 20].

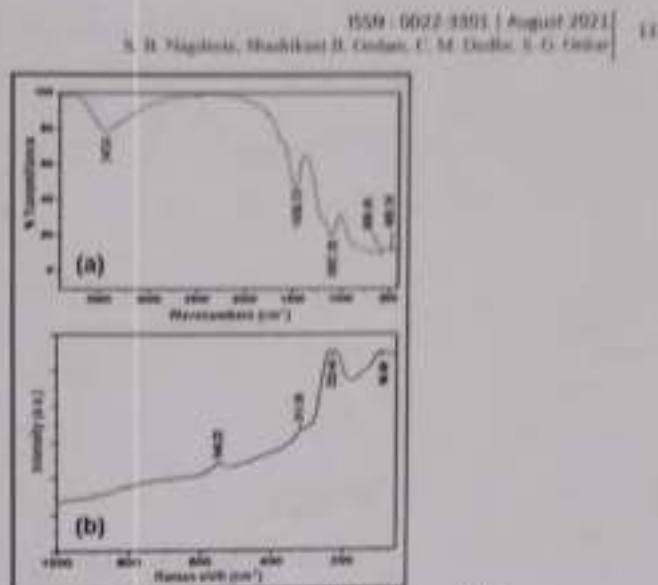
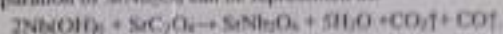


Fig.3 (a) FT-IR and (b) Raman spectra of SrNb_2O_6 nanoparticles.

FT-IR spectrum of material is shown in Fig.3a. This figure shows many vibration bands centered at 3450 , 1458.72 , 1097.28 , 600.45 and 469.16cm^{-1} . The vibration band centered at 3450cm^{-1} can be assigned to $-\text{OH}$ stretching. Similarly, bands centered at 1458.72 and 1097.28cm^{-1} can be assigned to the stretching vibrations of $\text{C}-\text{O}$ and $\text{C}=\text{O}$ [21,22]. The origin of these bands is in synthesis process. The reaction for the preparation of SrNb_2O_6 can be represented as:



The gases which are liberated during the process may get absorbed on the surfaces of the particles and reflected their existence in FTIR.

The vibration bands in the finger-print region ($1000-400\text{ cm}^{-1}$) are corresponding to inorganic structure. Two vibration bands (Fig.3a); one centered at 469.16cm^{-1} and other at 600.45 cm^{-1} are observed in this region. The wide band at 600.45 cm^{-1} can be assigned to the $\text{Nb}-\text{O}$ stretching vibrations and the band centered at 469.16 cm^{-1} to the bending vibrations of $\text{O}-\text{Nb}-\text{O}$ bond angles [23,24].

The stretching and bending vibrations in SrNb_2O_6 compound were also confirmed by Raman study.

Fig. 3b shows the Raman spectra of synthesized monoclinic SrNb_2O_6 . It shows bands of Raman shifts at 546.12 , 311.55 , 232.44 and 96 cm^{-1} . The medium and broad band centered at 546.22cm^{-1} is corresponding to the $\text{Nb}-\text{O}$ stretching vibration [24,25]. Similarly, broad band centered at 311.55 cm^{-1} and strong band centered at 232.44 cm^{-1} are corresponding to the $\text{O}-\text{Nb}-\text{O}$ bending vibrations. The strong band centered at 96 cm^{-1} may be due to the lattice vibration [24].

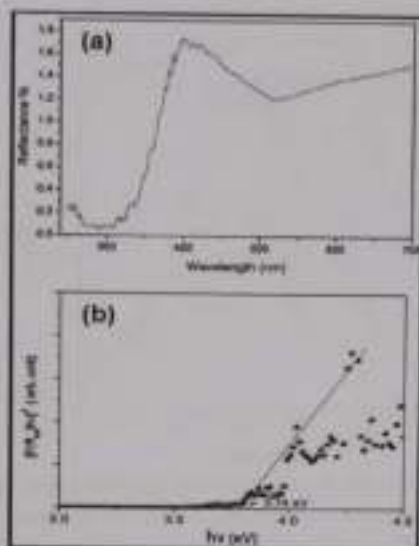


Fig. 4(a) UV-Vis diffuse reflectance spectrum & (b) corresponding K-M transform spectrum

Fig. 4a shows the UV-Vis diffuse reflectance spectrum of the SeNb_2O_6 nanoparticles, and Fig. 4b is its corresponding K-M transform diffuse spectrum which was obtained by using Eqs (3), (4) and (5). It was found that the optical band width is about 3.79 eV which is comparable to the reported one [5]. The corresponding absorption edge is about 327 nm. This means UV photons having wavelengths less than 327 nm will be absorbed by the compound.

3.2. Photoluminescence and photocatalysis:

Fig. 5 shows the PL emission spectrum ($\lambda_{\text{ex}} = 254 \text{ nm}$) of the synthesized monoclinic SeNb_2O_6 nanoparticles. It is observed that the emission peak bears higher intensity as compared to its bulk counterpart, and centering at 461 nm [5]. This self-activated PL may be a consequence of oxygen deficiency.

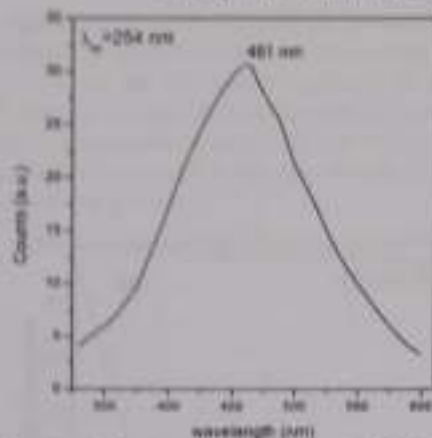


Fig.5 PL emission (λ_{ex} =254nm) spectrum of monoclinic SrNb_2O_6 nanoparticles.

In alkaline earth niobates, self-activated luminescence are originated from the absorbing group $[\text{NbO}_6]^{5-}$ which are greatly influenced by the oxygen vacancies [20]. The formation of oxygen vacancies is a consequence of consumption of large amount of oxygen to burn out organic matter present in the precursor during the preparation of material [26].

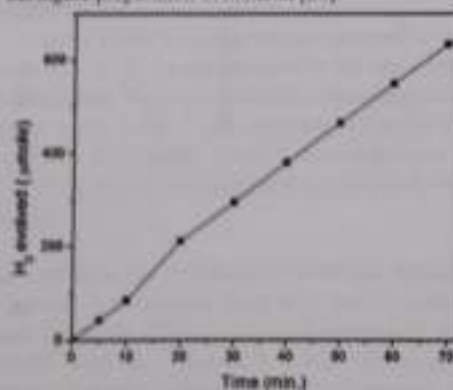


Fig.6: Photocatalytic H_2 evolution activity of SrNb_2O_6 nanoparticles.

The photocatalytic activities of the nanoparticles were studied under UV-visible source. Since the light absorbing group $[\text{NbO}_6]^{5-}$ is present in the nanoparticles, they absorb UV light and generate holes and electrons on the surface of the particles. These charges then may react with water molecules giving rise to the separation of O_2 and H_2 . The literature survey shows that the alkaline

earth niobates favor the liberation of H_2 only [3,12,23] and this tendency in the present case is more efficient. Fig. 6 shows a graph of amount of H_2 evolved by the synthesized $SrNb_2O_6$ nanoparticles against time. It is observed that the H_2 evolution tendency of the synthesized $SrNb_2O_6$ nanoparticles is about 30-40 times more than the reported value for monoclinic $SrNb_2O_6$ ceramics with particle size $1\mu m$ [5]. This may be simply because of about 40 times more surface area of the present nanoparticles as compared to ceramics of size $1\mu m$, estimated by formulae of volume and surface area $4/3.\pi r^3$ and $4.\pi r^2$ respectively.

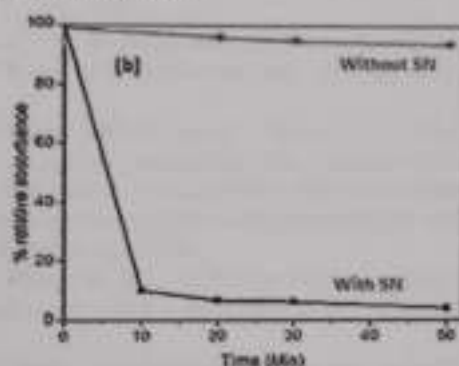


Fig. 7 Relative absorbance of MB with and without catalyst ($SrNb_2O_6$ nanoparticles)

The photocatalytic activity of $SrNb_2O_6$ nanoparticles for dye degradation was also studied for controlling the environmental issues. The relative maximum absorbance with and without $SrNb_2O_6$ nanoparticles against time is shown in Fig. 7. This figure shows that the % absorbance of light by MB with $SrNb_2O_6$ nanoparticles falls rapidly within a short interval of time, whereas without catalyst it falls only by ~7.5% in 30 min, indicating that the present $SrNb_2O_6$ act as an excellent catalytic agent under UV light.

4. Conclusion

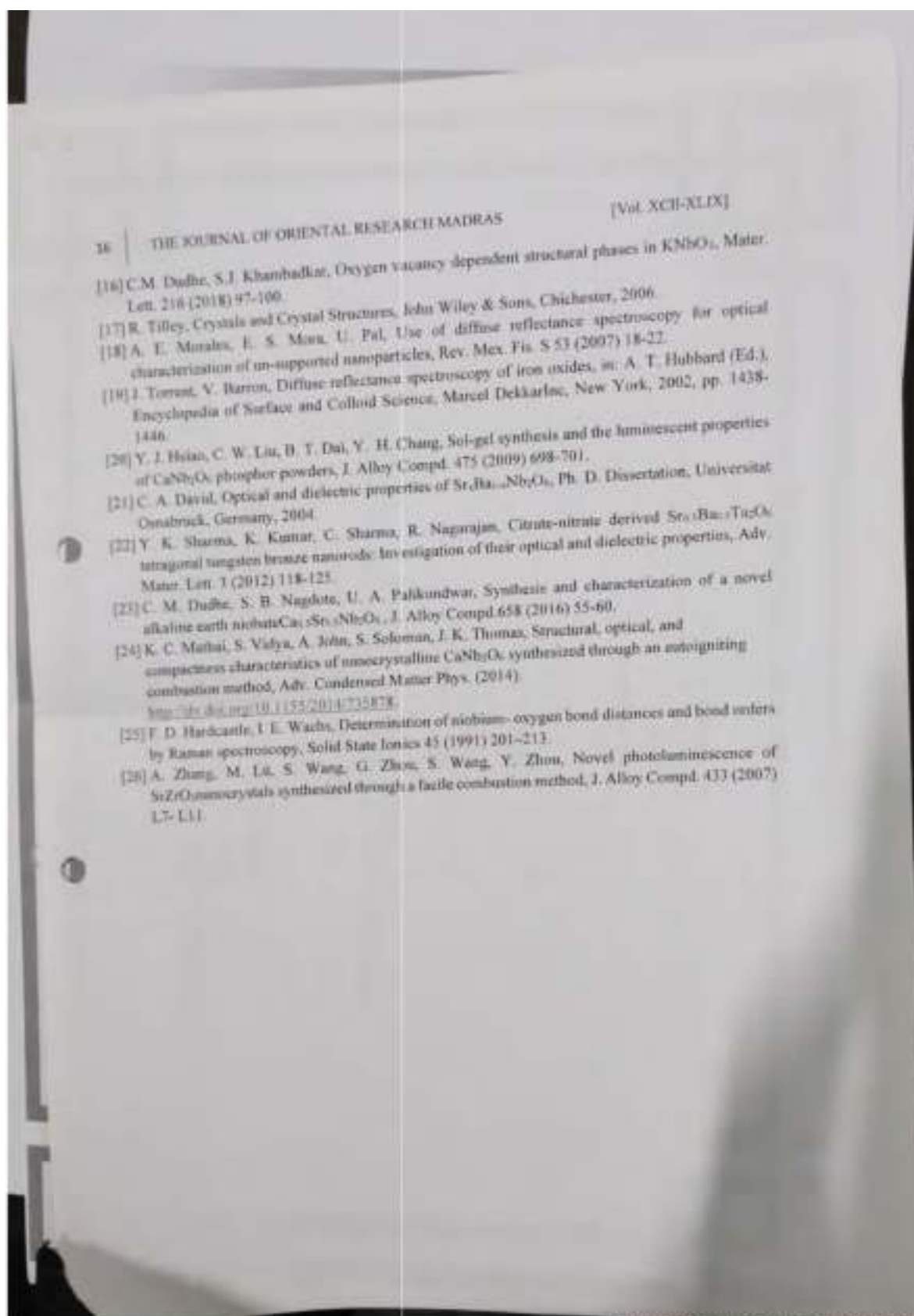
$SrNb_2O_6$ nanoparticles synthesized by a coprecipitation method stabilize in monoclinic phase. The $SrNb_2O_6$ powder stabilized in monoclinic phase with lattice parameters: $a = 7.719\text{\AA}$, $b = 5.589\text{\AA}$, $c = 10.982\text{\AA}$ and $\beta = 90.38^\circ$. The optical band gap energy as estimated by UV-Vis spectroscopy and using K-M theory was found to be 3.79 eV. The nanoparticles bear absorbing groups $[NbO_6]^{2-}$ from which self-activated photoluminescence originates. This activity was found to be considerable in the present nanoparticles. Similarly, the present nanoparticles of $SrNb_2O_6$ exhibit excellent H_2 evolution tendency from pure water under UV light as well as degradation of MB aqueous solution. Thus, the monoclinic $SrNb_2O_6$ seem to be an excellent candidate for solving the problems of green energy generation and water pollution, and also for PL applications.

Acknowledgment

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[Vol. XCII-XLIX]

16 | THE JOURNAL OF ORIENTAL RESEARCH MADRAS

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Introductory study on machine learning tool WEKA : Dr. S. A. Rodge, S. K. Shelke, Dr. S. A. Ghogare





Introductory study on machine learning tool WEKA

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Abstract:

Data mining is the process of identifying or extracting new patterns from massive datasets or databases, and it is a powerful new branch of computer science with a lot of potential. It's widely utilized in marketing, surveillance, fraud detection, artificial intelligence, scientific research, and a variety of other industries. Today the size of data is increasing day-by-day. To analyze the dataset from different angles and Data mining techniques the WEKA software is used. This paper describes to understand the basic of WEKA. It also explains the basic techniques to use WEKA and how datasets are load into it. WEKA also Supports Variety of Classifier that also explain in this paper.

Keywords: Data mining, data preprocessing, classification, cluster analysis, JRip, Weka tool et.

1. Introduction:

Today the rapid growth of data over the internet, product related word-of-mouth conversation have migrated to online markets, creating active electronic communication that provide a wealth of information. In data mining, the data is stored electronically and the search is computerized or at least augmented by machine. The use data mining is to gain knowledge, not just predictions [1]. Data Mining is the process of extract hidden information from the data set and it is also called as knowledge discovery. This Techniques is used in various applications such as Education, Banking, Market analysis, Telecom industry, Retail Industry, DNA Analysis and Many more. WEKA is open source software an open source software provides tools for data preprocessing, implementation of several Machine Learning algorithms, and visualization tools which help researchers to develop machine learning techniques and apply them to real-world data mining problems. There are some authors who uses WEKA in their dataset: Y. K. Salal, S. M. Abdullaev, Mukesh Kumar (2019), discovers execution forecast in Academic with assistance of Data Mining procedures to improve instruction quality. Gagangot Kaur, Amit Chhabra (2014). The paper portrays successful data borrowing frameworks for foreseeing diabetes from therapeutic records of patients. Pima Indians Diabetes dataset of 768 events is used by maker V. Veeralakshmi, Dr. D. Ramyachitra (2015) investigate execution of three principles classifier algorithms. The calculation is RIDOR, JRip, and Decision Table. IRIS Dataset is picked for correlation. Dr S.R.Kalimegh(2017) uses the WEKA with the data set of Indian news and come up with results.

2. Methodology

2.1 WEKA: WEKA was developed at the University of Waikato in New Zealand. The name stands for Waikato Environment for Knowledge Analysis. The system is written in Java and distributed under the terms of the GNU General Public License [2][3]. It runs on almost any platform and has been tested under Linux, Windows, and Macintosh operating systems. It provides a uniform interface to many different learning algorithms, along with methods for pre and post processing and for evaluating the result of learning schemes on any given dataset. This tool also supports the variety file formats for mining include ARFF, CSV, LibSVM, and C4.5[4][5]. The easiest way to use WEKA is through a graphical user interface called Explorer as shown in Fig. I. Fig. II Shows the ARFF File Opening by WEKA and Fig. III ARFF file processing by JRip. The Step by steps use of WEKA for result purpose is as explain below.



Fig. I: WEKA GUI Explorer



Fig. II: ARFF File Opening by WEKA



Fig. III: ARFF file processing by J48

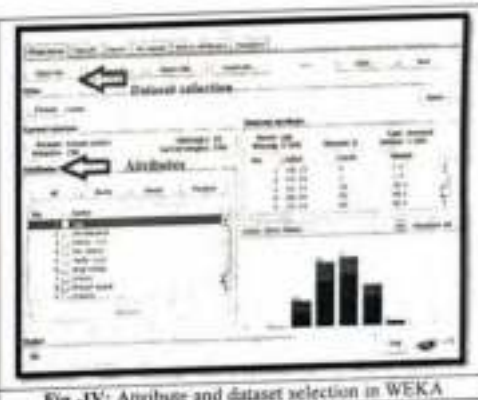


Fig.-IV: Attribute and dataset selection in WEKA

2.1.1 WEKA Application Interfaces

As shown in above Fig-I there are multiple option are available in WEKA application Interface.

- **Explorer:** explorer is environment for exploring data. Explorer contain pre-processing of data, attribute Selection, learning, Visualization.
- **Experimenter:** Experimenter is environment for performing experiments and conducting statistical tests between learning schemes.
- **Knowledge Flow:** Knowledge Flow is Java-Beans-based interface for setting going Machine Learning tests.
- **Simple Command-line:** Provides simple command-line interface that allows direct execution of WEKA commands for operating systems.

2.1.2 Load data into WEKA explorer:

- **Opening Data Set:** In WEKA, the data set taken from explorer window, as shown in fig.I the then click on "open file" and the browse to navigate to the data folder within the WEKA-3.8.5. folder or select dataset supported by WEKA. The data Selection as shown in Fig IV. After selection of data set the attributes are available on the WEKA screen. By using Attributes, we can visualize the data in graphical form. There are many more options are available to understand the dataset in various ways.
- **Select Classifier:** After Successful Selection of Data set, Next step to select Classifier which is available on next Tab i.e. 'Classify'. Depending upon the use we can select the Classifier and then apply to dataset.
- **Test Option:** The result of applying the chosen classifier will be tested according to the options that are set by clicking in the "Test options" box. There are four test modes: Use training set, supplied test, Cross Validation folds, Percentage split.

- **Use Training Set:** The accuracy of the classifier is based on the class of instances against their actual classes
- **Supplied Test Set:** In the Supplied test a separate file is loaded. Based on this evaluation of the classifier take place on the basis of the authenticity of the prediction of the class of a set of instances loaded from that file
- **Cross-validation:** The cross validation is also called as K-Folds cross Validation. In K-fold Cross-validation, the dataset is divided into k sets. In the first run, k-1 sets are used for training the classifier and the remaining one set is used for testing the classifier. In the next run, another set is held out as the test data and the remaining k-1 sets are used as training sets. Thus, each set is held out in turn as the testing set and the process is repeated kth times. So, each data set used once for testing and k-1 times for training.
- **Percentage Split:** The data set is divided into two sections in Percentage Split based on the value supplied in the percent field. typical split worth is 66% to 34% for train and test sets separately. The classifier is evaluated on how well it predicts the class of the test data.

3. Advantages

1. WEKA is user friendly which helps to develop new machine learning plans.
2. WEKA loads information record in format of ARFF, CSV, C4.5, and binary.
3. It is open source, Free, Extensible can be incorporated into other java bundles.

4. Limitation

1. Worse connectivity to excel spreadsheet and non-java based databases.
2. CSV reader not as robust as in Rapid Miner.
3. Weka is much weaker in classical statistics.
4. Does not have arrangement to store parameters for scaling to apply to future datasets.
5. Does not have programmed arrangement for Parameter enhancement of learning/statistical techniques

5. Conclusion

WEKA help us researchers to predict the Knowledge from their data set, which help to predict future knowledge from the existing dataset. The basic information provided in this paper may be help new research scholar to gain valuable output from this Software. This paper help to understand the basic working of WEKA, it also shows how data set load into it. Along with this advantages and disadvantages are also discuss.

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Development of linear array transducer using field _II

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ABSTRACT

Science past decades, ultrasound imaging technology has satisfied outstanding improvement in obtaining significant diagnostic information from patients in a fast, noninvasive approach. Although the technology has benefited from sophisticated signal processing technology and imaging system integration, much of this progress has been derived from the improvement of ultrasonic transducers. Ultrasonic arrays are used in various applications together with medical imaging. In this particular case is important to accomplish precise information regarding the magnitude and position of the peak pressure and intensity produced by the imaging probe. This paper presents the design of linear array transducers for ultrasonic measurements.

Keywords--Ultrasonic, Linear array Transducer, medical imaging, Field-II GUI, TX/RX Fields.

1 INTRODUCTION

For the period of the second half of 20th current century the medical imaging is grown through Ultrasound tool speedily. The part of novel technology is the use of computers to decide problems by simulating theoretical models (Numerical simulations) that has taken place alongside pure theory and experiment during the last few decades. These numerical simulations permit one to resolve problems that may not be accessible to direct experimental study or too complex for theoretical analysis. Computer simulations can link the gap between analysis and experiment [1].

These numerical simulations have emerged as a new branch in science and technology complementing both experiments and theory. A simulation can sometimes replace physical experiments, even though most often a simulation and an experiment are complementary. The results of scientific experiments are often explained by simulations and simulations are often calibrated by experiments. The experiments provide input for the simulations, which are viewed as experimenting with theoretical models. The feedback of numerical results into theoretical

modeling and continues interaction with laboratory experiments and analytical theory makes computing a vital tool for science. Consequently the increased in computing power in both speed and storage has given computational electronics its significance. Improved computer capacity and the solution algorithms themselves, have a big outcome on the excellence of solution obtained. A numerical model can be used to understand measurements and observations enlarge existing analytical models into new parameter regimes and quantitatively test existing theories that can be done by comparing model predictions to experimental information.

The mutual weak point of both experiment and theory is cover up by the numerical simulations examination and experiment. A third dimension in ultrasonic measurements, of equivalent status and significance to experiment and analysis is nothing but the simulation determination [1]. It has taken an everlasting place in every one aspect of ultrasonic measurements from basic research to engineering design.

A novel and potentially powerful tool is the computer experiment. One can resolve novel and uncertain aspects of usual process, by combining predictable theory, experiment and computer simulation [1]. Such aspects could frequently neither have been understood nor revealed by analysis or experiments alone.

More than the last half century much development has been made in medical device technology. One particular medical technology that has enhanced speedily over the last 30 years is ultrasound. This advancement in technology however has brought with it the rapid obsolescence of system design. The accomplishment of modern electronics is built on the possibility to precisely predict system performance by the use of simulation tools. This model can be extended to components such as piezoelectric transducers attached to the electronics [2]. The ability to simulate both piezoelectric transducer and electronics together renders possible efficient optimizations at system level, i.e. minimizing size, price and power consumption [3].

The systems for images processing in the medical field are very important calling for new techniques, much more superior and performing than they used to be, in order to provide a acceptable analysis and diagnosis. Amongst the medical techniques using computer sciences, it can be mentioned: scintigraphy, echography, tomography, radiography, quantitative microscopy, nuclear magnetic resonance. Ultrasound, widely used in many areas of medicine, provides a secure

and efficient means for diagnosis and therapy. When the medium becomes complex solving the wave propagation formula becomes virtually not possible. Modeling becomes much more complex inside the body because the ultrasound propagation speed is different for each tissue and it is known that tissues are not a homogeneous medium for ultrasound wave propagation [4]. Therefore, it is important to know how the ultrasound wave is generated and the ultrasound wave beam shaped.

II SPATIAL IMPULSE THEORY

The pressure field generated by the aperture is found by the Rayleigh integral [5]

$$p(\vec{r}_1, t) = \frac{\rho_0}{2\pi} \int_S \frac{\partial v_n(\vec{r}_2, t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{|\vec{r}_1 - \vec{r}_2|} dS \quad (1)$$

Where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then; where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

$$\Psi(\vec{r}_1, t) = v_n(t) * \int_S \frac{\partial(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{2\pi |\vec{r}_1 - \vec{r}_2|} dS \quad (2)$$

Where * denotes convolution in time. The integral in this equation

$$h(\vec{r}_1, t) = \int_S \frac{\partial(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{2\pi |\vec{r}_1 - \vec{r}_2|} dS \quad (3)$$

Represent the spatial impulse response. The continuous wave field can be found from the Fourier transform of

$$p(\vec{r}_1, t) = p_s \frac{\partial v(t)}{\partial t} * h(\vec{r}_1, t) \quad (4)$$

The impulse response includes the excitation convolved with both the transducers electro-mechanical impulse response in transmit and receive. The final signal for a collection of scatters is calculated as a linear sum over all signals from the different scatters [6-7].

III LINEAR ARRAY TRANSDUCER

The linear array is the fundamental type of multi-element transducer and it scans the region of interest by exciting the elements situated over the region. The field is focused on the region by introducing time delay in the excitation of the concerned individual elements, so initially concave beam is emitted. Here a Fig.1 shows general design format of 16 element linear array transducer having height, width and kerf of individual element are taken as 5 mm, 0.2 mm and 0.02 mm respectively. The transducers are situated at the center of the coordinate system. To achieve focal length of 30 mm from the center of transducer the electronic focusing is included.



Fig. (1) Design format of linear array transducer (Height=5mm, Width=0.25mm, Kerf=0.02mm)

In this paper a linear array transducer of 16 elements is simulated using FIELD-II program with center frequencies 5MHz. For this specified linear array transducer, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30)

IV RESULT AND DISCUSSION

The calculation of the impulse response is facilitated by projecting the field point onto the plane of the aperture. In this way, the problem became two-dimensional and the field point is given

Table: 1 shows the parameters for 16 element array transducer, excitation pulse and medium used for this centre frequency (f_0) used is 5MHz. Figs. (a-m) shows; Element impulse response for 16 element array, TX Field image for 16 element array, TX Axial waveform for 16 element array, TX Lateral beam plot for 16 element array, TX/RX Field image for 16 element array, TX/RX Axial waveform for 16 element array, TX/RX Lateral beam plot for 16 element array, K- space TX/RX field image for 16 element array, K-space axial slice for 16 element array, K-space lateral slice for 16 element array, Detected image for 16 element array, Detected image axial slice for 16 element array and Detected image lateral slice for 16 element array.



Table 1

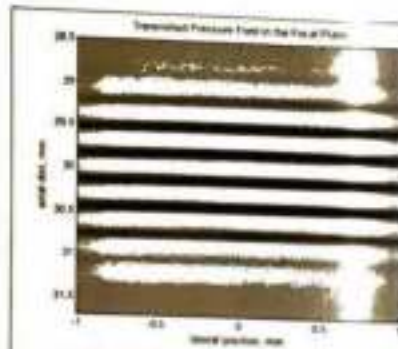


Fig. (b) TX Field image

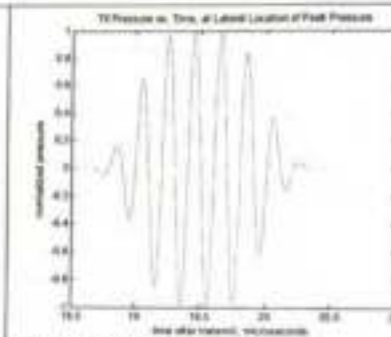


Fig. (c) TX Axial waveform

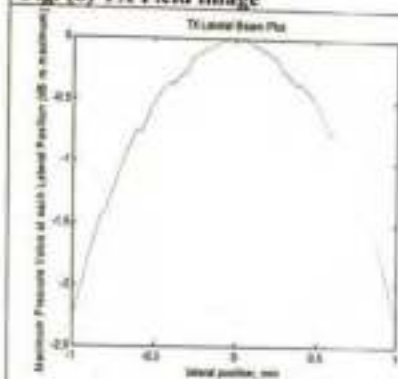


Fig. (d) TX Lateral beamplot

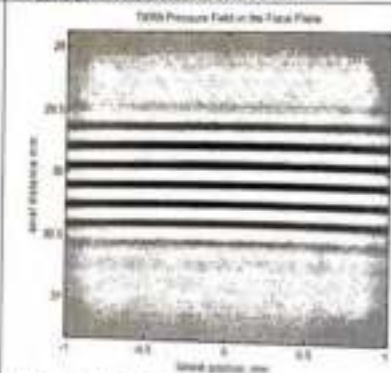


Fig. (e) TX/RX Field image

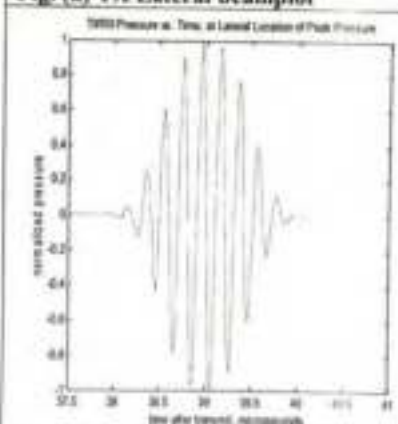


Fig. (f) TX/RX Axial waveform

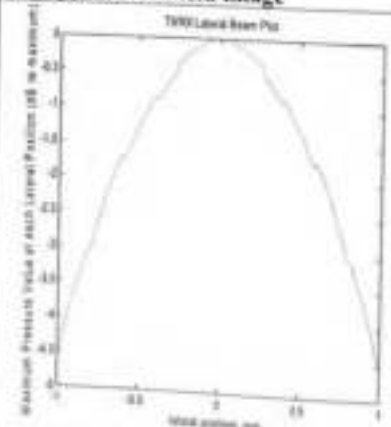
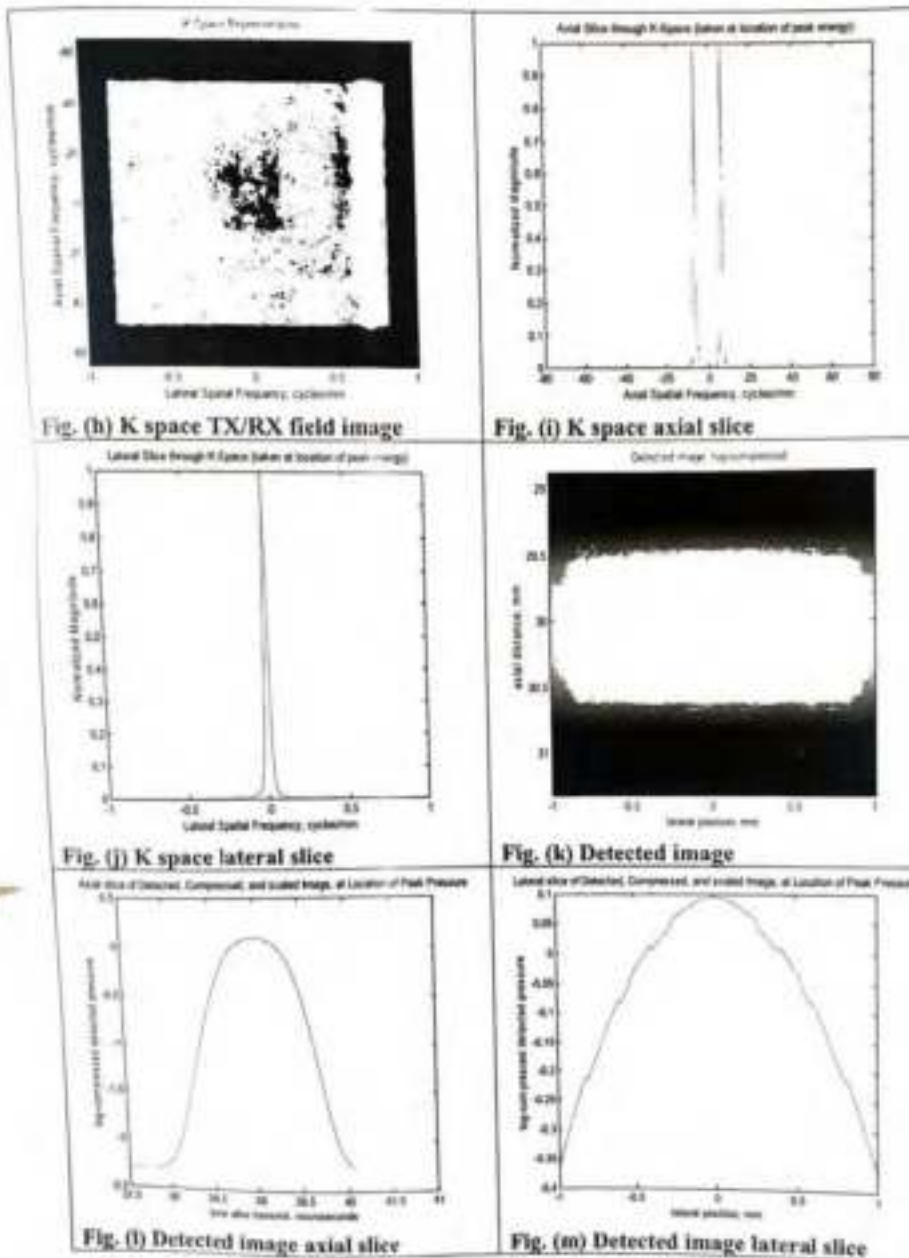


Fig. (g) TX/RX Lateral beam-plot



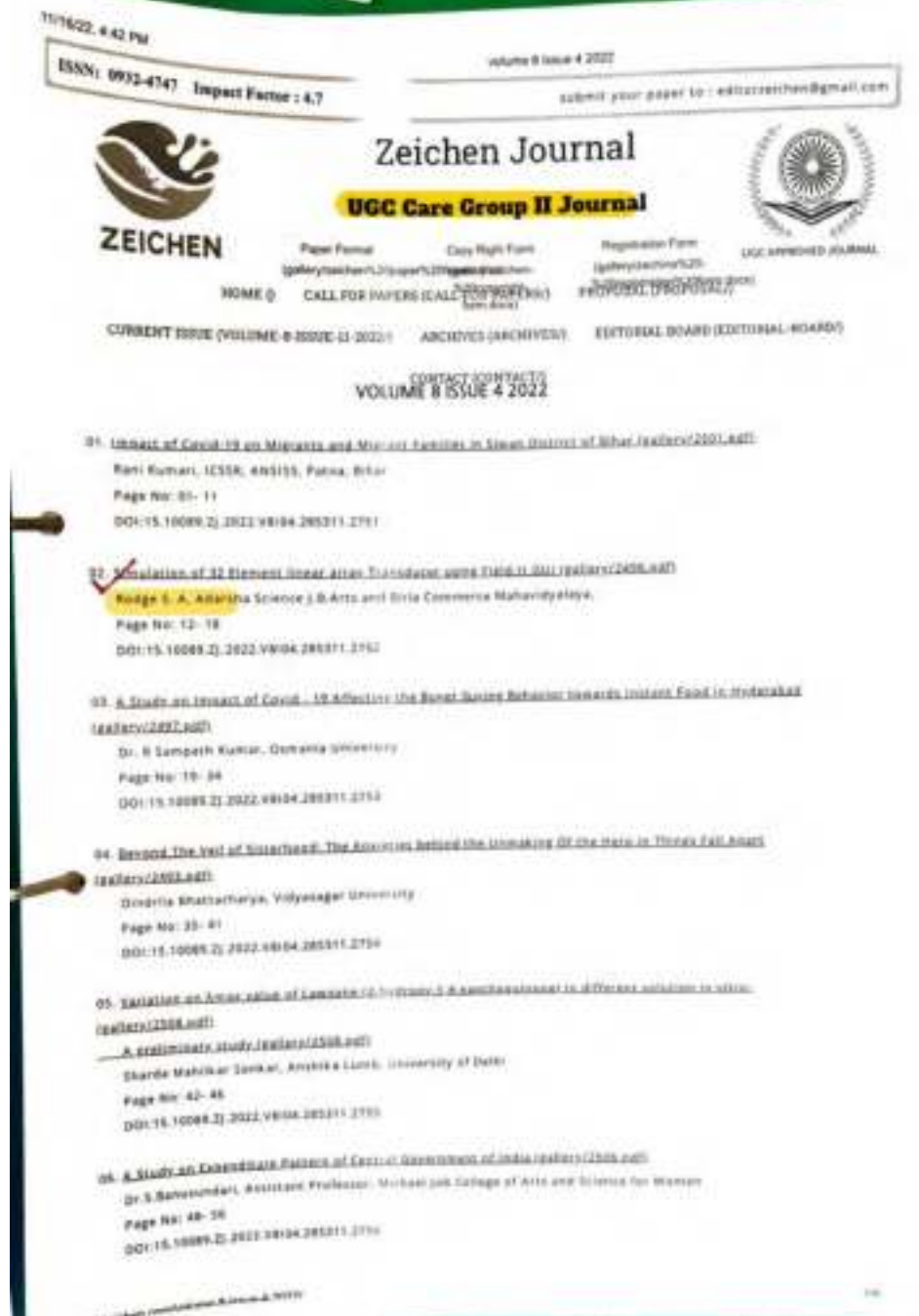
V CONCLUSION

The paper attempts to present a coherent analysis of the focusing strategies for 2-D array transducer design and properties, based on linear acoustics. The delays on the individual transducer elements and their relative weight or apodization are changed continuously as a function of depth. This yields near perfect focused images for all depths and has increased the contrast in the displayed image, thus, benefitting the diagnostic importance of ultrasonic imaging.

References:

- [1] Gandole, Y. B. (2012). Computer Modeling and Simulation of Ultrasonic System for Material Characterization. [http://www.oalib.com/search? kw=Y.%20B.%20Gandole &searchField=authors](http://www.oalib.com/search?kw=Y.%20B.%20Gandole&searchField=authors).
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Simulation of 32 Element linear array Transducer using Field-II GUI: Dr. S.A.Rodge



Linear array transducer of 32 Elements with center frequency 10 MHz :Dr. S. A. Rodge, Mr. S. K. Shelke, Mr. V. G. Gabhane, Mr. H. E. Salve



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Linear Array Transducer of 32 Elements with Center Frequency 10 MHz

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ABSTRACT

Ultrasonic arrays are used in many applications including medical imaging. In this specific case is important to achieve precise information about the magnitude and position of the peak pressure, intensity, detected image and various pressure fields produced by the probe. This paper presents the simulation of linear array transducers of 32 elements with center frequency 10 MHz for ultrasonic measurements.

Keywords—Ultrasonic, Linear array, Transducer, medical imaging, Field-II GUI, Tx Rx Field images.

1 INTRODUCTION

For the period of the second half of 20th current century the medical imaging is grown through Ultrasound tool speedily. The part of novel technology is the use of computers to decide problems by simulating theoretical models (Numerical simulations) that has taken place alongside pure theory and experiment during the last few decades. These numerical simulations permit one to resolve problems that may not be accessible to direct experimental study or too complex for theoretical analysis. Computer simulations can link the gap between analysis and experiment [2].

More than the last half century much development has been made in medical device technology. One particular medical technology that has enhanced speedily over the last 30 years is ultrasound. This advancement in technology however has brought with it the rapid obsolescence of system design. The accomplishment of modern electronics is built on the possibility to precisely predict system performance by the use of simulation tools. This model can be extended to components such as piezoelectric transducers attached to the electronics [3]. The ability to simulate both piezoelectric transducer and electronics together renders possible efficient optimizations at system level, i.e., minimizing size, price and power consumption [4].

2. SPATIAL IMPULSE THEORY

The pressure field generated by the aperture is found by the Rayleigh integral [5]

$$p(\vec{r}_1, t) = \frac{\rho_0}{2\pi} \int_S \frac{\partial v_n(\vec{r}_2, t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{|\vec{r}_1 - \vec{r}_2|} dS \quad (1)$$

Where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then: where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

$$\Psi(\vec{r}_1, t) = v_n(t) * \int_S \frac{\partial(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{2\pi |\vec{r}_1 - \vec{r}_2|} dS \quad (2)$$

Where * denotes convolution in time. The integral in this equation

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Represent the spatial impulse response. The continuous wave field can be found from the Fourier transform of

$$p(\vec{r}_1, t) = \rho_0 \frac{\partial v(t)}{\partial t} * h(\vec{r}_1, t) \quad (4)$$

The impulse response includes the excitation convolved with both the transducers electro-mechanical impulse response in transmit and receive. The final signal for a collection of scatters is calculated as a linear sum over all signals from the different scatters [6-7].

3 SIMULATION OF LINEAR ARRAY TRANSDUCER

The linear array is the fundamental type of multi-element transducer and it scans the region of interest by exciting the elements situated over the region. The field is focused on the region by introducing time delay in the excitation of the concerned individual elements, so initially concave beam is emitted.

Here a Fig.1 shows general design format of 16 element linear array transducer having height, width and kerf of individual element are taken as 5 mm, 0.2 mm and 0.02 mm respectively. The transducers are situated at the center of the coordinate system. To achieve focal length of 30 mm from the center of transducer the electronic focusing is included.



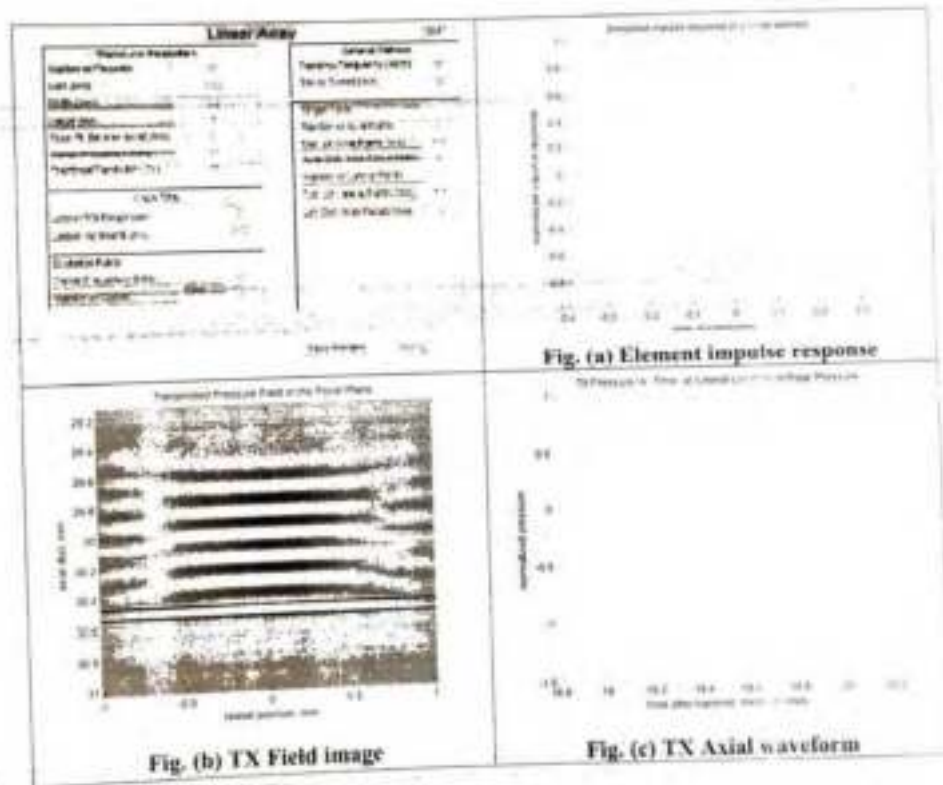
In this paper a linear array transducer of 16 elements is simulated using FIELD-II program with center frequencies 5MHz. For this specified linear array transducer, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30).

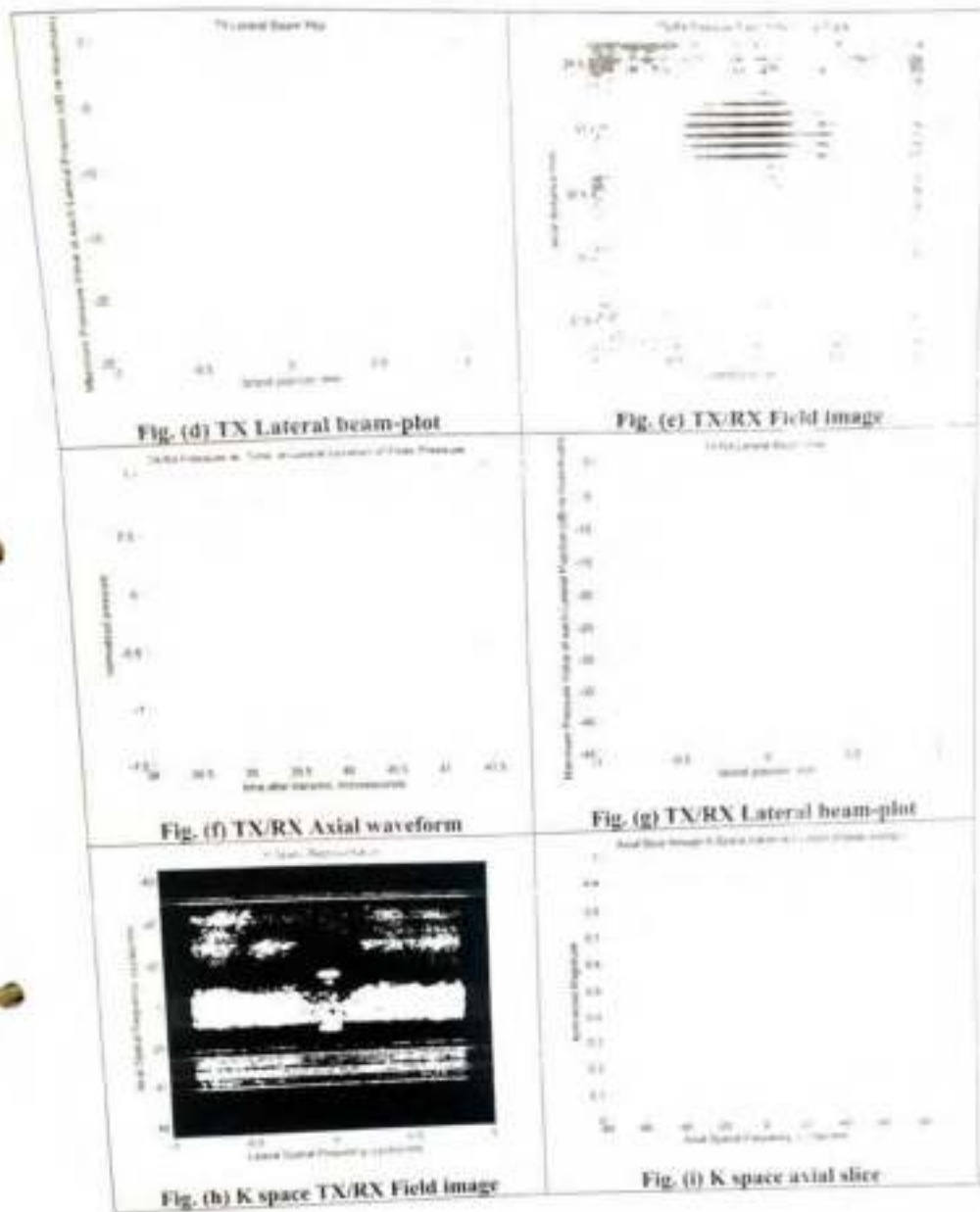
4 RESULT AND DISCUSSION

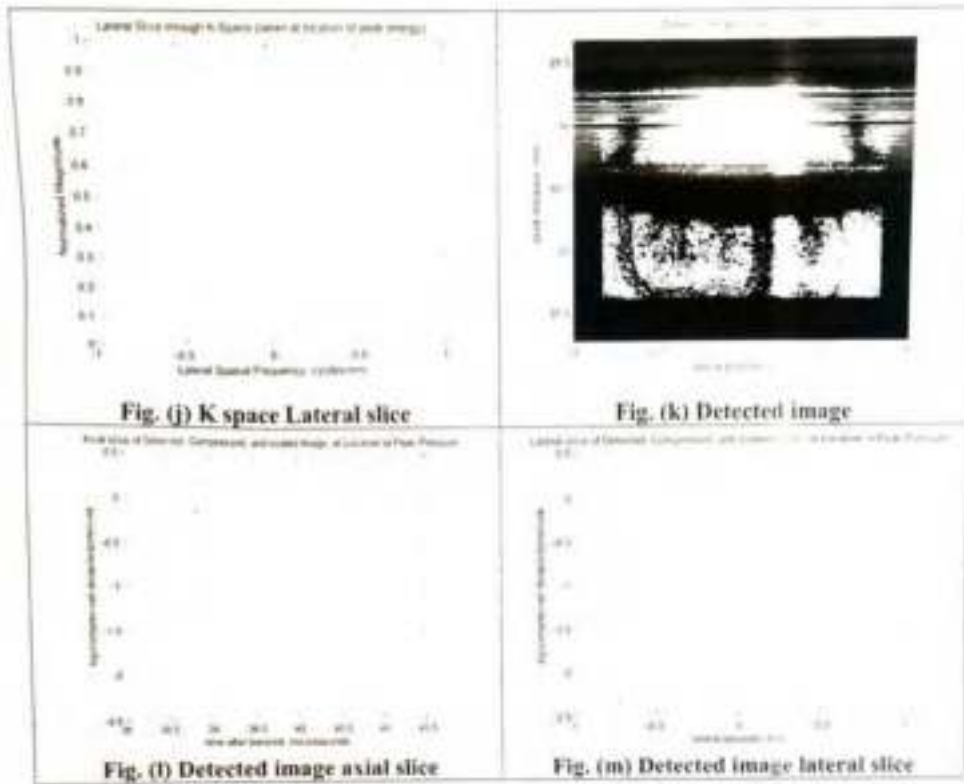
The calculation of the impulse response is facilitated by projecting the field point onto the plane of the aperture. In this way, the problem became two-dimensional and the field point is given as a (x, y) coordinate set and a height z above the plane. The spatial impulse response is, thus, determined by the relative length of the part of the arc that intersects the aperture [8]. Thereby it is the crossing of the projected ultrasonic waves with the edges of the aperture that determines the spatial impulse responses as a function of time. In this paper by using FIELD-II program created a 32-element linear array transducer with center frequency $f_0 = 10\text{MHz}$. The speed of sound in

tissue is $c = f\lambda = 1540\text{m/s}$. The sampling frequency used was $f_s = 100\text{MHz}$. The elements had a width and height of 0.25mm and 5mm respectively. The focal-point was set to 30mm .

Table: 1 shows the parameters for 32element array transducer, excitation pulse and medium used for this center frequency (f_0) used is 10MHz Figs. (a-m) shows; Element impulse response for 32 element array, TX Field image for 32 element array, TX Axial waveform for 32 element array, TX Lateral beam plot for 32 element array, TX/RX Field image for 32 element array, TX/RX Axial waveform for 32 element array, TX/RX Lateral beam plot for 32 element array, K- space TX/RX field image for 32 element array, K-space axial slice for 32 element array, K-space lateral slice for 32 element array, Detected image for 32 element array, Detected image axial slice for 32 element array and Detected image lateral slice for 32 element array.







5 CONCLUSION

The paper attempts to present a coherent analysis of the focusing strategies for 2-D array transducer design and properties, based on linear acoustics. The delays on the individual transducer elements and their relative weight or apodization are changed continuously as a function of depth. This yields near perfect focused images for all depths and has increased the contrast in the displayed image, thus, benefitting the diagnostic importance of ultrasonic imaging. If the center frequency and number of elements in transducer is increased then contrast in the detected image is increased, this also increases the diagnostic status of ultrasonic imaging.

References:

- [1] T. R. Moeker, "Thickness mode piezoelectric transducers," *Ultrasonics* **10**, 26-36, 1972.

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Study the response of Ultrasonic linear array transducer with different number of elements: Dr. S.A.Rodge



Study The Response of Ultrasonic Linear Array Transducer with Different number of Elements

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ABSTRACT

Ultrasonic arrays are used in many applications including medical imaging. In this specific case is important to achieve precise information about the magnitude and position of the peak pressure and intensity produced by the probe. This paper presents the simulation of linear array transducers of 16, 32 and 64 elements for ultrasonic measurements.

Keywords—Ultrasonic, Linear array, Transducer, medical imaging, Field-II GUI, Tx-Rx Field images.

INTRODUCTION

In the biomedical field, the systems for images processing are very important calling for new techniques, much more advanced and performing than they used to be, in order to provide a correct analysis and diagnosis. Ultrasound, widely used in many areas of medicine, provides a safe and efficient means for diagnosis and therapy. There are considerable efforts in designing transducers and determining the characteristics of the emitted field. To simulate the pressure response from transducers, a model which can predict the volt-to-surface acceleration conversion of multilayered transducers is needed. Methods for modeling piezoelectric transducers are well known in literature [1-6]. Most of these models are based on electrical equivalent circuits benefiting from transmission line theory to represent the electromechanical coupling and acoustic wave propagation, and others rely on deriving impedance matrices for describing the transducer behavior. All of these methods have their advantage and disadvantages depending on the application of use.

There are considerable efforts in designing transducers and determining the characteristics of the emitted field. Field II program [7], developed by J.A. Jensen, can simulate all kinds of ultrasound transducers using linear acoustics and it utilizes the Topholme-Stepanishen method for calculating spatial impulse responses. The program is capable of calculating the fields for both the pulsed and continuous wave case for a large number of different transducers and allows visualization of simulating transducers. The calculation of the spatial impulse response assumes linearity [8] and any complex-shaped transducer can therefore be divided into smaller apertures and the response can be found by adding the responses from the sub-apertures.

SPATIAL IMPULSE THEORY

The pressure field generated by the aperture is found by the Rayleigh integral [9]

$$p(\vec{r}_1, t) = \frac{\rho_0}{2\pi} \int_S \frac{\vec{r}_1 \cdot \vec{r}_2}{|\vec{r}_1 - \vec{r}_2|^3} \frac{\partial v_n(\vec{r}_2, t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{\partial t} dS \quad (1)$$

where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, $\vec{r}_1 \cdot \vec{r}_2$ is the velocity normal to the transducer surface. Using the velocity potential Ψ , and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, $\vec{r}_1 \cdot \vec{r}_2$ is the velocity normal to the transducer surface. Using the velocity potential Ψ , and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

$$\Psi(\vec{r}_1, t) = v_n(t) * \int_S \frac{\partial(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{2\pi |\vec{r}_1 - \vec{r}_2|} dS \quad (2)$$

where * denotes convolution in time. The integral in this equation

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represent the spatial impulse response. The continuous wave field can be found from the Fourier transform of:

$$p(\vec{r}_1, t) = \rho_0 \frac{\partial v(t)}{\partial t} + h(\vec{r}_1, t) \quad (4)$$

The impulse response includes the excitation convolved with both the transducers

electro-mechanical impulse response in transmit and receive. The final signal for a collection of scatterers is calculated as a linear sum over all signals from the different scatterers [10].

LINEAR ARRAY TRANSDUCER

The linear array is the fundamental type of multi-element transducer and it scans the region of interest by exciting the elements situated over the region. The field is focused on the region by introducing time delay in the excitation of the concerned individual elements, so initially concave beam is emitted. Here a 16 element linear array transducer is designed using FIELD-II program as shown in the Fig.1, height, width and kerf of individual element are taken as 5 mm, 0.2 mm and 0.02 mm respectively. The transducer is situated at the center of the coordinate system. The electronic focusing is incorporated to achieve focal length of 30 mm from the center of transducer. For the above linear array transducer, an excitation signal of two cycles of sinusoidal pulses is given in Fig. 2(a). The impulse response pattern obtained for each element is shown in Fig. 2(b).

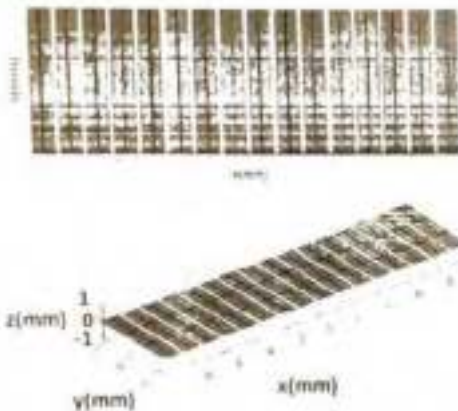


Fig.1 Design of linear array transducer (Height=5mm, Width=0.25mm, Kerf=0.02mm)

For this specified linear array, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30) whose plot is shown in Fig. 4. Fig. 4(a) shows the field generated while Fig. 4(b) shows the pressure profile at focal distance. Similarly Fig.5 illustrates the lateral beam pattern generated by the assumed linear array transducer. The other simulation parameters chosen are central transducer frequency (f0) =5 MHz, acoustic speed (c0) =1540m/s, fractional bandwidth 50%, center frequency of excitation pulse = 7.5 MHz, Number of cycle=5.

IV RESULT AND DISCUSSION

The calculation of the impulse response is facilitated by projecting the field point onto the plane of the aperture. In this way, the problem became two-dimensional and the field point

is given as a (x, y) coordinate set and a height z above the plane. The spatial impulse response is, thus, determined by the relative length of the part of the arc that intersects the aperture. Thereby it is the crossing of the projected ultrasonic waves with the edges of the aperture that determines the spatial impulse responses as a function of time.

By using FIELD II program were created three linear arrays with the same characteristics, but with different number of elements: 16 elements, 32 elements and 64 elements. Respectively. For the simulations the transducer center frequency was set to $f_0 = 5\text{MHz}$. The speed of sound in tissue is $c = 1540\text{m/s}$, which gives a wavelength of mm. The sampling frequency used was $f_s = 100\text{MHz}$. The elements had a width and height of 0.25mm and 5mm respectively. The focal-point was set to 30mm.

Then the normalized spatial impulse response for this aperture was calculated and plotted by time. Figures 2, 3, 4 and 5 shows the normalized spatial impulse response. Transducer Pressure field, Lateral beam plot and Transmitted Pressure Field in the focal plan for 16 elements. Figures 6, 7, 8 and 9 shows the normalized spatial impulse response, Transducer Pressure field, Lateral beam plot and Transmitted Pressure Field in the focal plan for 32 elements. Figures 10, 11, 12 and 13 shows the normalized spatial impulse response, Transducer Pressure field, Lateral beam plot and Transmitted Pressure Field in the focal plan for 64 elements.

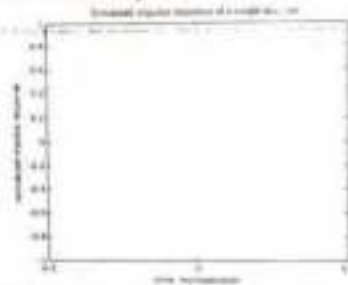


Fig. 2: Normalized impulse response of a single element.

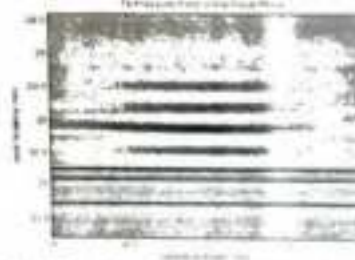


Fig. 3: Transducer Pressure field for 16 elements.

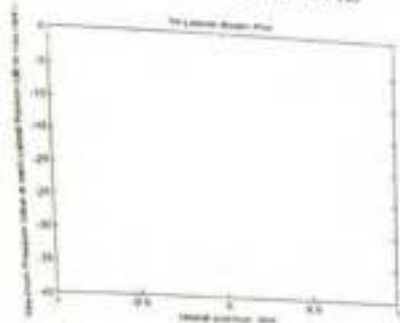


Fig. 4: Lateral beam plot of 16 elements

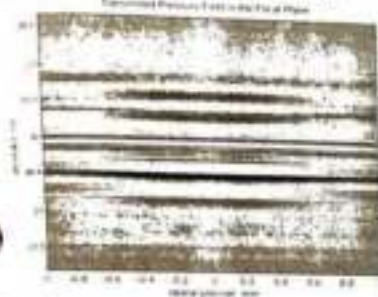


Fig. 5: Transmitted Pressure Field in the focal plane for 16 elements.

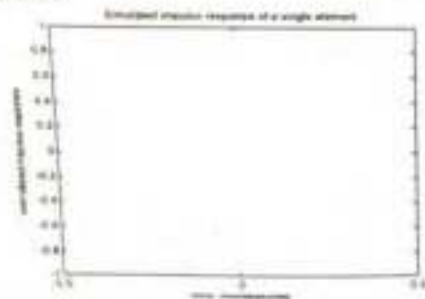


Fig. 6: Normalized impulse response of 16 elements.

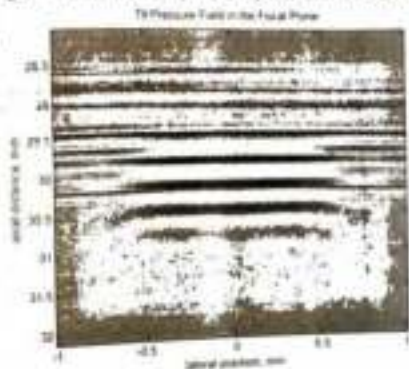


Fig. 7: Transducer Pressure field for 32 elements.

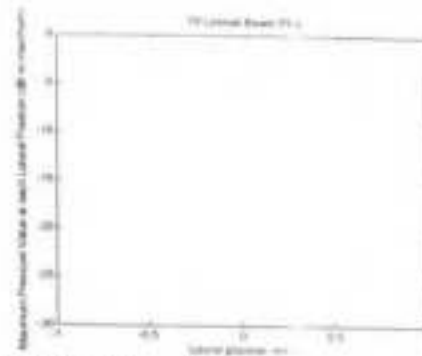


Fig. 8: Lateral beam plot of 32 elements.

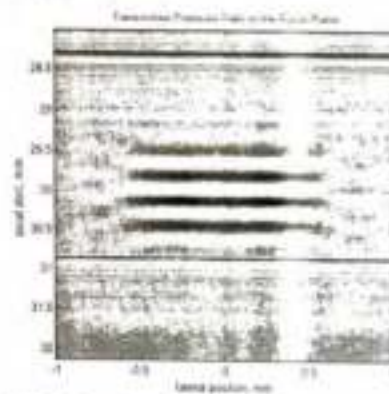


Fig. 9: Transmitted Pressure Field in the focal plane for 32 elements.

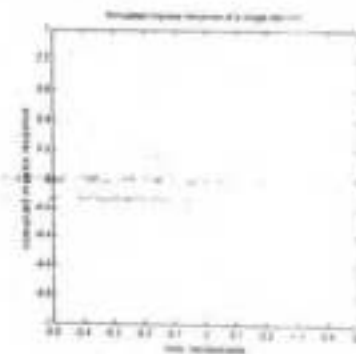


Fig. 10: Normalized impulse response of 32 elements.

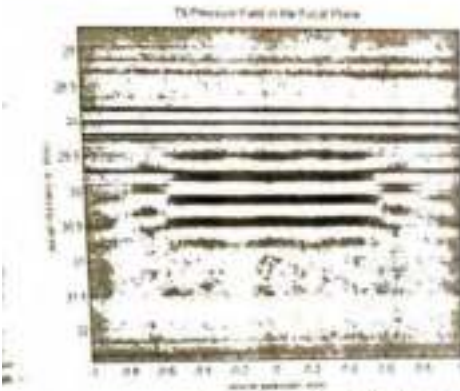


Fig. 11: Transducer Pressure field for 64 elements.

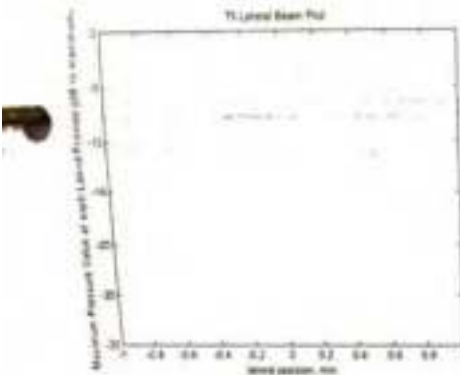


Fig. 12: Lateral beam plot of 64 elements.

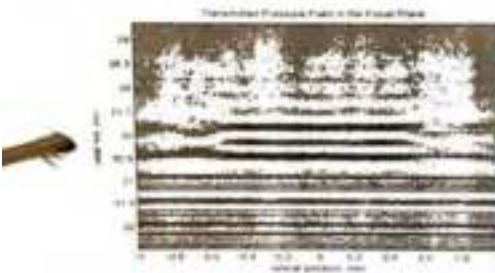


Fig. 13: Transmitted Pressure Field in the focal plane for 64 elements.

V CONCLUSION

The paper attempts to present a coherent analysis of the focusing strategies for 2-D array transducer design and properties, based on linear acoustics. The delays on the individual transducer elements and their relative weight or apodization are changed continuously as a function of depth. This yields near perfect focused images for all depths and has increased the contrast in the displayed image, thus, benefiting the diagnostic value of ultrasonic imaging. If the number of elements in the array transducer is increased then the contrast of detected image and pressure field generated across the transducer is increased, this will increase the diagnostic value of ultrasonic imaging.

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Simulation of linear array Transducer with center frequency 5 MHz: Dr. S. A. Rodge

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Simulation of Linear Array Transducer with Center Frequency 5 MHz

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ABSTRACT

Over the past decades, ultrasound imaging technology has satisfied outstanding improvement in obtaining significant diagnostic information from patients in a fast, noninvasive approach. Piezoelectric transducers are important elements of various broadband ultrasonic systems, either pulse-echo or through-transmission, used for imaging and detection purposes [1]. In ultrasonic broadband applications such as medical imaging, or non-destructive testing, piezoelectric transducers should generate/receive ultrasonic signals with good efficiency over a large frequency range. This implies the use of piezoelectric transducers with high sensitivity, broad bandwidth and short-duration impulse responses. High sensitivity provides large signal amplitudes which determine a good dynamic range for the system and the short duration of the received ultrasonic signal provides a good axial resolution. This paper presents the simulation of 8-element linear array transducer with center frequency 10 MHz, using Field-II GUI program for ultrasonic measurements.

Keywords-Detected image, Field-II GUI, Linear array Transducer, medical imaging, TX/RX Fields, TX/RX Axial slice, TX/RX lateral slice, Ultrasonic.

1. INTRODUCTION

For the period of the second half of 20th current century the medical imaging is grown through Ultrasound tool speedily. The part of novel technology is the use of computers to decide problems by simulating theoretical models (Numerical simulations) that has taken place alongside pure theory and experiment during the last few decades. These numerical simulations permit one to resolve problems that may not be accessible to direct experimental study or too complex for theoretical analysis. Computer simulations can link the gap between analysis and experiment [2].

More than the last half century much development has been made in medical device technology. One particular medical technology that has enhanced speedily over the last 30 years is ultrasound. This advancement in technology however has brought with it the rapid obsolescence of systems.

VOLUME 6 ISSUE 6 2022

PAGE NO: 793

design. The accomplishment of modern electronics is built on the possibility to precisely predict system performance by the use of simulation tools. This model can be extended to components such as piezoelectric transducers attached to the electronics [3]. The ability to simulate both piezoelectric transducer and electronics together renders possible efficient optimizations at system level, i.e. minimizing size, price and power consumption [4].

II SPATIAL IMPULSE THEORY

The pressure field generated by the aperture is found by the Rayleigh integral [5]

$$p(\vec{r}_1, t) = \frac{\rho_0}{2\pi} \int \frac{\partial v_n(\vec{r}_2, t, |\vec{r}_1 - \vec{r}_2|)}{|\vec{r}_1 - \vec{r}_2|} d\vec{r}_2 \quad (1)$$

Where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then: where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

$$\Psi(\vec{r}_1, t) = v_n(t) * \int \frac{e^{i(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}}{2\pi |\vec{r}_1 - \vec{r}_2|} d\vec{r}_2 \quad (2)$$

Where * denotes convolution in time. The integral in this equation

$$h(\vec{r}_1, t) = \int \frac{e^{i(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}}{2\pi |\vec{r}_1 - \vec{r}_2|} d\vec{r}_2 \quad (3)$$

Represent the spatial impulse response. The continuous wave field can be found from the Fourier transform of

$$p(\vec{r}_1, t) = \rho_0 \frac{\partial \Psi(t)}{\partial t} * h(\vec{r}_1, t) \quad (4)$$



The impulse response includes the excitation convolved with both the transducers electro-mechanical impulse response in transmit and receive. The final signal for a collection of scatters is calculated as a linear sum over all signals from the different scatters [6-7].

III SIMULATION OF LINEAR ARRAY TRANSDUCER

The linear array is the fundamental type of multi-element transducer and it scans the region of interest by exciting the elements situated over the region. The field is focused on the region by introducing time delay in the excitation of the concerned individual elements, so initially concave beam is emitted. Here a Fig.1 shows general design format of 16 element linear array transducer having height, width and kerf of individual element are taken as 5 mm, 0.2 mm and 0.02 mm respectively. The transducers are situated at the center of the coordinate system. To achieve focal length of 30 mm from the center of transducer the electronic focusing is included.



Fig. (1) Design format of linear array transducer (Height=5mm, Width=0.25mm, Kerf=0.02mm)

In this paper a linear array transducer of 8 elements is simulated using FIELD-II program with center frequencies 7.5MHz. For this specified linear array transducer, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30)

IV RESULT AND DISCUSSION

The calculation of the impulse response is facilitated by projecting the field point onto the plane of the aperture. In this way, the problem became two-dimensional and the field point is given as a (x, y) coordinate set and a height z above the plane. The spatial impulse response is, thus, determined by the relative length of the part of the arc that intersects the aperture [8]. Thereby it is the crossing of the projected ultrasonic waves with the edges of the aperture that determines the spatial impulse responses as a function of time. In this paper by using FIELD-II program created a 32-element linear array transducer with center frequency $f_0 = 5\text{MHz}$. The speed of sound in tissue

is $c=f_0 = 1540\text{m/s}$. The sampling frequency used was $f_s = 100\text{MHz}$. The elements had a width and height of 0.25mm and 5mm respectively. The focal-point was set to 30mm .

Table: 1 shows the parameters for 8 element array transducer, excitation pulse and medium used, for this center frequency (f_0) used is 5 MHz While Figs. (a-m) shows; Element impulse response for 8 element array, TX Field image for 8 element array, TX Axial waveform for 8 element array, TX Lateral beam plot for 8 element array, TX/RX Field image for 8 element array, TX/RX Axial waveform for 8 element array, TX/RX Lateral beam plot for 8 element array, K-space TX/RX field image for 8 element array, K-space axial slice for 8 element array, K-space lateral slice for 8 element array, Detected image for 8 element array, Detected image axial slice for 8 element array and Detected image lateral slice for 8 element array.

Linear Array	
Number of Elements	8
Element Width (mm)	0.25
Element Height (mm)	5
Element Pitch (mm)	0.25
Element Delay (ns)	0
Element Focus (mm)	30
Element Frequency (MHz)	5
Element Wavelength (mm)	30
Element Delay (ns)	0
Element Focus (mm)	30
Element Frequency (MHz)	5
Element Wavelength (mm)	30
Element Delay (ns)	0
Element Focus (mm)	30
Element Frequency (MHz)	5
Element Wavelength (mm)	30

Table: 1

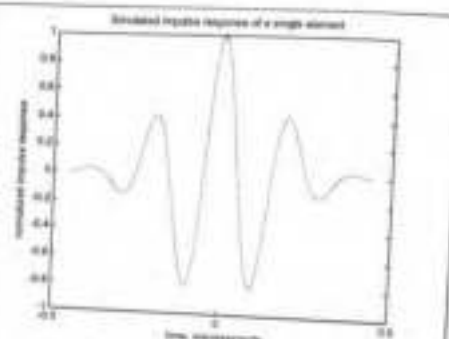


Fig. (a) Element impulse response

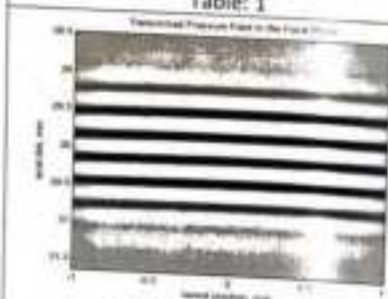


Fig. (b) TX Field image

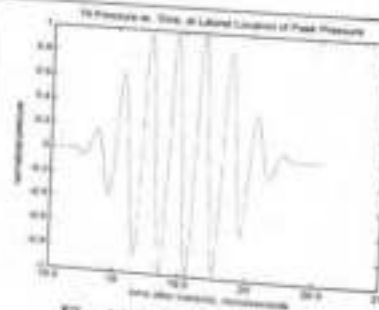
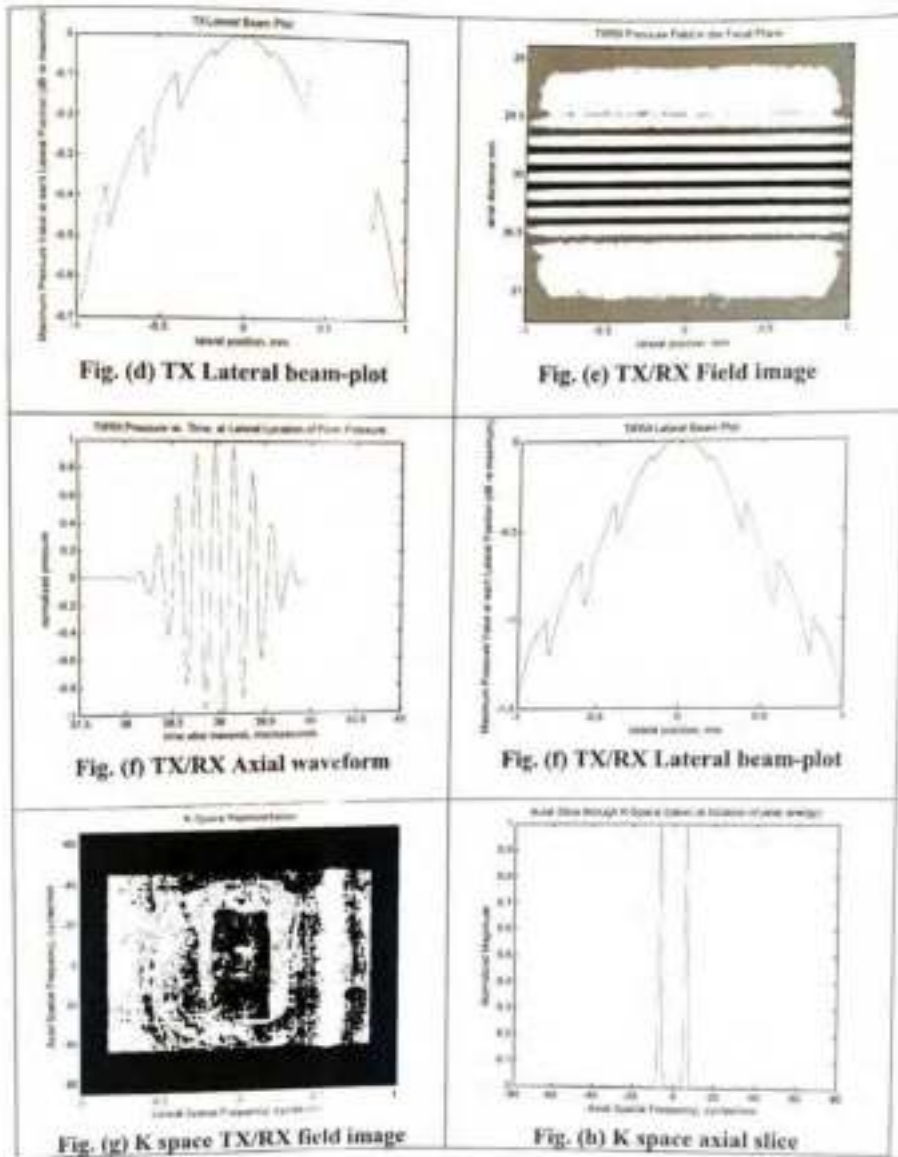
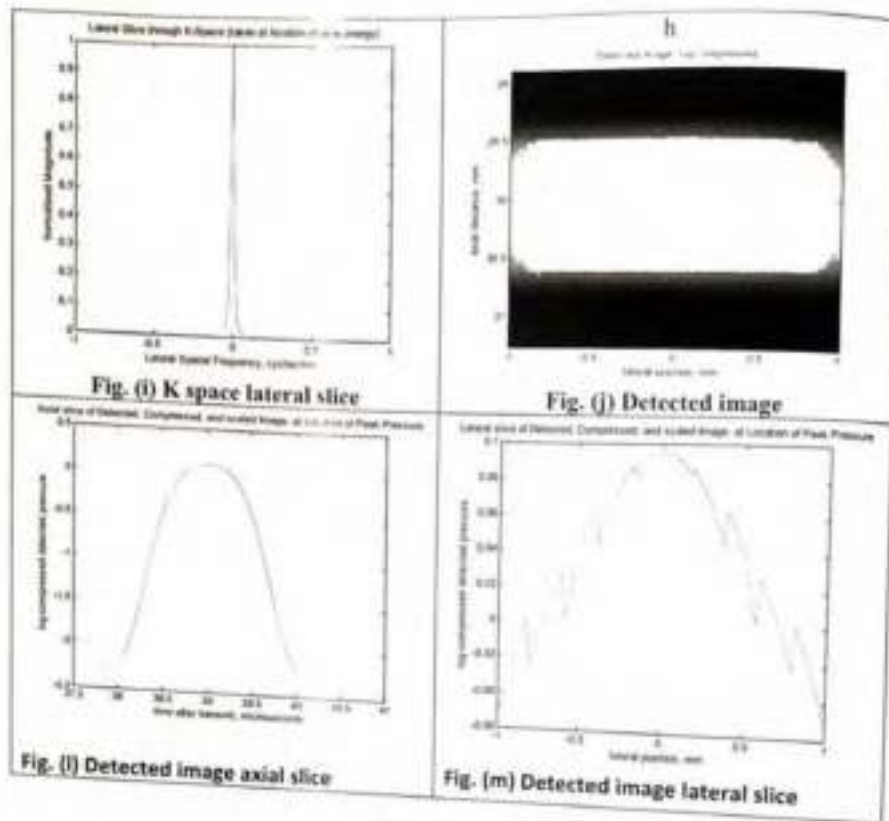


Fig. (c) TX Axial waveform





V CONCLUSION

The paper attempts to present a coherent analysis of the focusing strategies for 2-D array transducer design and properties, based on linear acoustics. The delays on the individual transducer elements and their relative weight or apodization are changed continuously as a function of depth. This yields near perfect focused images for all depths and has increased the contrast in the displayed image, thus, benefiting the diagnostic importance of ultrasonic imaging. If the center frequency and number of elements in transducer is increased then contrast in the detected image is increased, this also increases the diagnostic status of ultrasonic imaging.

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2021

VOLUME 8, ISSUE 12, 2021
Volume 8, Issue 12, December 2021

VOLUME 8, ISSUE 11, 2021
Volume 8, Issue 11, November 2021

VOLUME 8, ISSUE 10, 2021
Volume 8, Issue 10, October 2021

VOLUME 8, ISSUE 9, 2021
Volume 8, Issue 9, September 2021

VOLUME 8, ISSUE 8, 2021
Volume 8, Issue 8, August 2021

VOLUME 8, ISSUE 7, 2021
Volume 8, Issue 7, July 2021

VOLUME 8, ISSUE 6, 2021
Volume 8, Issue 6, June 2021

VOLUME 8, ISSUE 5, 2021
Volume 8, Issue 5, May 2021

VOLUME 8, ISSUE 4, 2021
Volume 8, Issue 4, April 2021

VOLUME 8, ISSUE 3, 2021
Volume 8, Issue 3, March 2021

VOLUME 8, ISSUE 2, 2021
Volume 8, Issue 2, February 2021

VOLUME 8, ISSUE 1, 2021
Volume 8, Issue 1, January 2021

2020

VOLUME 7, ISSUE 12, 2020
Volume 7, Issue 12, December 2020

VOLUME 7, ISSUE 11, 2020
Volume 7, Issue 11, November 2020

VOLUME 7, ISSUE 10, 2020
Volume 7, Issue 10, October 2020

VOLUME 7, ISSUE 9, 2020
Volume 7, Issue 9, September 2020

VOLUME 7, ISSUE 8, 2020
Volume 7, Issue 8, August 2020

VOLUME 7, ISSUE 7, 2020
Volume 7, Issue 7, July 2020

VOLUME 7, ISSUE 6, 2020
Volume 7, Issue 6, June 2020

VOLUME 7, ISSUE 5, 2020
Volume 7, Issue 5, May 2020

VOLUME 7, ISSUE 4, 2020
Volume 7, Issue 4, April 2020

VOLUME 7, ISSUE 3, 2020
Volume 7, Issue 3, March 2020

VOLUME 7, ISSUE 2, 2020
Volume 7, Issue 2, February 2020

VOLUME 7, ISSUE 1, 2020
Volume 7, Issue 1, January 2020

2019

VOLUME 6, ISSUE 12, 2019
Volume 6, Issue 12, December 2019

VOLUME 6, ISSUE 11, 2019
Volume 6, Issue 11, November 2019

VOLUME 6, ISSUE 10, 2019
Volume 6, Issue 10, October 2019

VOLUME 6, ISSUE 9, 2019
Volume 6, Issue 9, September 2019

VOLUME 6, ISSUE 8, 2019
Volume 6, Issue 8, August 2019

VOLUME 6, ISSUE 7, 2019
Volume 6, Issue 7, July 2019

VOLUME 6, ISSUE 6, 2019
Volume 6, Issue 6, June 2019

VOLUME 6, ISSUE 5, 2019
Volume 6, Issue 5, May 2019

VOLUME 6, ISSUE 4, 2019
Volume 6, Issue 4, April 2019

VOLUME 6, ISSUE 3, 2019
Volume 6, Issue 3, March 2019

VOLUME 6, ISSUE 2, 2019
Volume 6, Issue 2, February 2019

VOLUME 6, ISSUE 1, 2019
Volume 6, Issue 1, January 2019

2018

VOLUME 5, ISSUE 12, 2018
Volume 5, Issue 12, December 2018

VOLUME 5, ISSUE 11, 2018
Volume 5, Issue 11, November 2018

VOLUME 5, ISSUE 10, 2018
Volume 5, Issue 10, October 2018

VOLUME 5, ISSUE 9, 2018
Volume 5, Issue 9, September 2018

VOLUME 5, ISSUE 8, 2018
Volume 5, Issue 8, August 2018

VOLUME 5, ISSUE 7, 2018
Volume 5, Issue 7, July 2018

VOLUME 5, ISSUE 6, 2018
Volume 5, Issue 6, June 2018

VOLUME 5, ISSUE 5, 2018
Volume 5, Issue 5, May 2018

VOLUME 5, ISSUE 4, 2018
Volume 5, Issue 4, April 2018

VOLUME 5, ISSUE 3, 2018
Volume 5, Issue 3, March 2018

VOLUME 5, ISSUE 2, 2018
Volume 5, Issue 2, February 2018

VOLUME 5, ISSUE 1, 2018
Volume 5, Issue 1, January 2018

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VOLUME 5, ISSUE 4, 2018 (Volume 5, Issue 4, 2018)

VOLUME 5, ISSUE 3, 2018 (Volume 5, Issue 3, 2018)

Comparison of Responses of 2 Element and 4 Element Array Transducer at Center Frequency 5 MHz

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ABSTRACT

The systems for images processing in the medical field are very important calling for new techniques, much more advanced and performing than they used to be, in order to provide a correct analysis and diagnosis. Among the medical techniques using computer sciences, it can be mentioned: scintigraphy, echography, tomography, radiography, quantitative microscopy, nuclear magnetic resonance. Ultrasound, widely used in many areas of medicine, provides a safe and efficient means for diagnosis and therapy. When the medium becomes complex solving the wave propagation formula becomes virtually impossible. Modeling becomes much more complex inside the body because the ultrasound propagation speed is different for each tissue and it is known that tissues are not a homogeneous medium for ultrasound wave propagation. Therefore, it is important to know how the ultrasound wave is generated and the ultrasound wave beam shaped. This paper represents the comparison of responses of 2-element 4-element linear array transducers

Key words: Ultrasonic, Linear array Transducer, Field-II GUI, TX/RX Fields, detected image.

1.INTRODUCTION

For the period of the second half of 20th current century the medical imaging is grown through Ultrasound tool speedily. The part of novel technology is the use of computers to decide problems by simulating theoretical models (Numerical simulations) that has taken place alongside pure theory and experiment during the last few decades. These numerical simulations permit one to resolve problems that may not be accessible to direct experimental study or too complex for theoretical analysis. Computer simulations can link the gap between analysis and experiment [1]. These numerical simulations have emerged as a new branch in science and technology complementing both experiments and theory. A simulation can sometimes replace physical experiments, even though most often a simulation and an experiment are complementary. The results of scientific experiments are often explained by simulations and simulations are often calibrated by experiments. The experiments provide input for the simulations, which are viewed as experimenting with theoretical models. The feedback of numerical results into theoretical

modeling and continues interaction with laboratory experiments and analytical theory makes computing a vital tool for science. Consequently, the increased in computing power in both speed and storage has given computational electronics its significance. Improved computer capacity and the solution algorithms themselves, have a big outcome on the excellence of solution obtained. A numerical model can be used to understand measurements and observations enlarge existing analytical models into new parameter regimes and quantitatively test existing theories that can be done by comparing model predictions to experimental information.

The mutual weak point of both experiment and theory is cover up by the numerical simulations' examination and experiment. A third dimension in ultrasonic measurements, of equivalent status and significance to experiment and analysis is nothing but the simulation determination [1]. It has taken an everlasting place in every one aspect of ultrasonic measurements from basic research to engineering design.

II SPECIAL IMPLUSE THEORY

The pressure field generated by the aperture is found by the Rayleigh integral [5]

$$p(\vec{r}_1, t) = \frac{\rho_0}{2\pi} \int_S \frac{\partial v_n(\vec{r}_2, t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{|\vec{r}_1 - \vec{r}_2|} dS \quad (1)$$

Where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then: where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

$$\Psi(\vec{r}_1, t) = v_s(t) * \int \frac{\partial(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{2\pi|\vec{r}_1 - \vec{r}_2|} \quad (2)$$

Where * denotes convolution in time. The integral in this equation

$$h(\vec{r}_1, t) = \int \frac{\partial(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{2\pi|\vec{r}_1 - \vec{r}_2|} \quad (3) \text{Represent the spatial impulse}$$

response. The continuous wave field can be found from the Fourier transform of

$$p(\vec{r}_1, t) = \rho_0 \frac{\partial v(t)}{\partial t} * h(\vec{r}_1, t) \quad (4)$$

The impulse response includes the excitation convolved with both the transducers electro-mechanical impulse response in transmit and receive. The final signal for a collection of scatters is calculated as a linear sum over all signals from the different scatters [6-8].

II SIMULATION OF LINEAR ARRAY TRANSDUCER

The linear array is the fundamental type of multi-element transducer and it scans the region of interest by exciting the elements situated over the region. The field is focused on the region by introducing time delay in the excitation of the concerned individual elements, so initially concave beam is emitted. Here a Fig.1 shows general design format of 16 element linear array transducer having height, width and kerf of individual element are taken as 5 mm, 0.2 mm and 0.02 mm respectively. The transducers are situated at the center of the coordinate system. To achieve focal length of 30 mm from the center of transducer the electronic focusing is included.



Fig. (1) Design format of linear array transducer (Height=5mm, Width=0.25mm, Kerf=0.02mm)

In this paper a linear array transducer of 16 elements is simulated using FIELD-II program with center frequencies 5MHz. For this specified linear array transducer, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30)

IV. RESULT AND DISCUSSION

The calculation of the impulse response is facilitated by projecting the field point onto the plane of the aperture. In this way, the problem became two-dimensional and the field point is given as a (x, y) coordinate set and a height z above the plane. The spatial impulse response is, thus, determined by the relative length of the part of the arc that intersects the aperture [8]. Thereby it is the crossing of the projected ultrasonic waves with the edges of the aperture that determines the spatial impulse responses as a function of time. In this paper by using FIELD-II program created a 2-element and 4-element linear array transducer using field -II GUI with center frequency $f_0 = 5\text{MHz}$. The speed of sound in tissue is $c = 1540\text{m/s}$. The sampling frequency used was $f_s = 100\text{MHz}$. The elements had a width and height of 0.25mm and 5mm respectively. The focal-point was set to 30mm.

Table: 1 and Table: 2 shows the parameters for 2 element and 4- element array transducer, excitation pulse and medium used for this centre frequency (f_0) used is 5MHz. Figs. (a-m) and figs. (a'-m') shows; Element impulse response for 2 and 4-element arrays, TX Field image for 2 and 4 element arrays, TX Axial waveform for 2 and 4 element arrays, TX Lateral beam plot for 2 and 4 element arrays, TX/RX Field image for 2 and 4 element arrays, TX/RX Axial waveform for 2 and 4 element arrays, TX/RX Lateral beam plot for 2 and 4 element arrays, K- space TX/RX field image for 2 and 4 element arrays, K-space axial slice for 2 and 4 element arrays, K-space lateral slice for 2 and 4 element arrays, Detected image for 2 and 4 element arrays, Detected image axial slice for 2 and 4 element arrays and Detected image lateral slice for 2 and 4 element arrays.

IV. RESULT AND DISCUSSION

Tx Field Parameters		Rx Field Parameters	
Carrier Frequency (MHz)	100	Carrier Frequency (MHz)	100
Carrier Power (dBm)	30	Carrier Power (dBm)	30
Carrier Phase (deg)	0	Carrier Phase (deg)	0
Carrier Polarization	Linear	Carrier Polarization	Linear
Carrier Wavelength (m)	3	Carrier Wavelength (m)	3
Carrier Frequency (Hz)	100000000	Carrier Frequency (Hz)	100000000
Carrier Wavelength (m)	3	Carrier Wavelength (m)	3
Carrier Frequency (Hz)	100000000	Carrier Frequency (Hz)	100000000
Carrier Wavelength (m)	3	Carrier Wavelength (m)	3
Carrier Frequency (Hz)	100000000	Carrier Frequency (Hz)	100000000
Carrier Wavelength (m)	3	Carrier Wavelength (m)	3
Carrier Frequency (Hz)	100000000	Carrier Frequency (Hz)	100000000
Carrier Wavelength (m)	3	Carrier Wavelength (m)	3

Table: 1

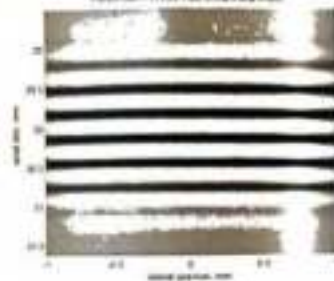


Fig.(b) TX Field image

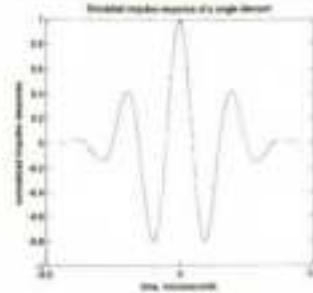


Fig. (a) Element impulse response

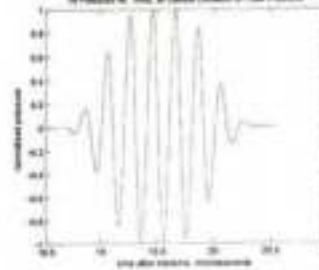


Fig. (c) TX Axial waveform

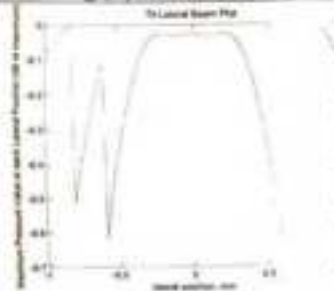


Fig. (d) TX Lateral beam-plot

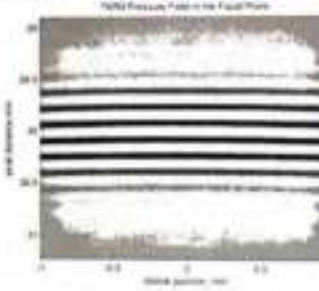


Fig. (e) TX/RX Field image

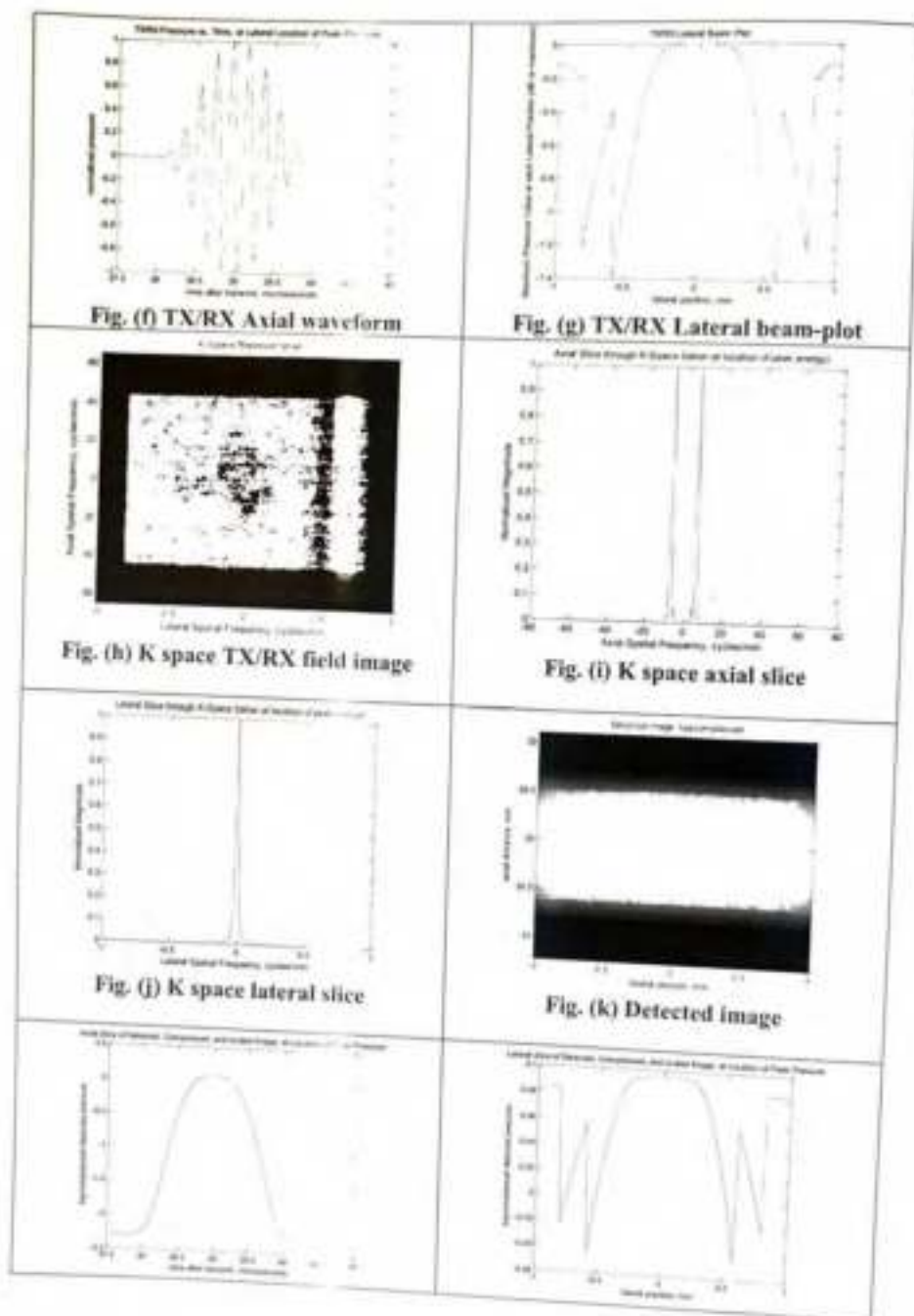


Fig.(m) Detected image lateral slice

Fig. (a') Element impulse response

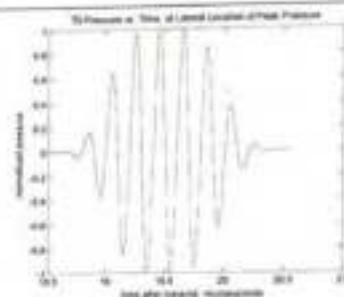


Fig. (c') TX Axial waveform

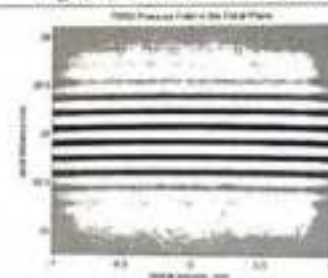
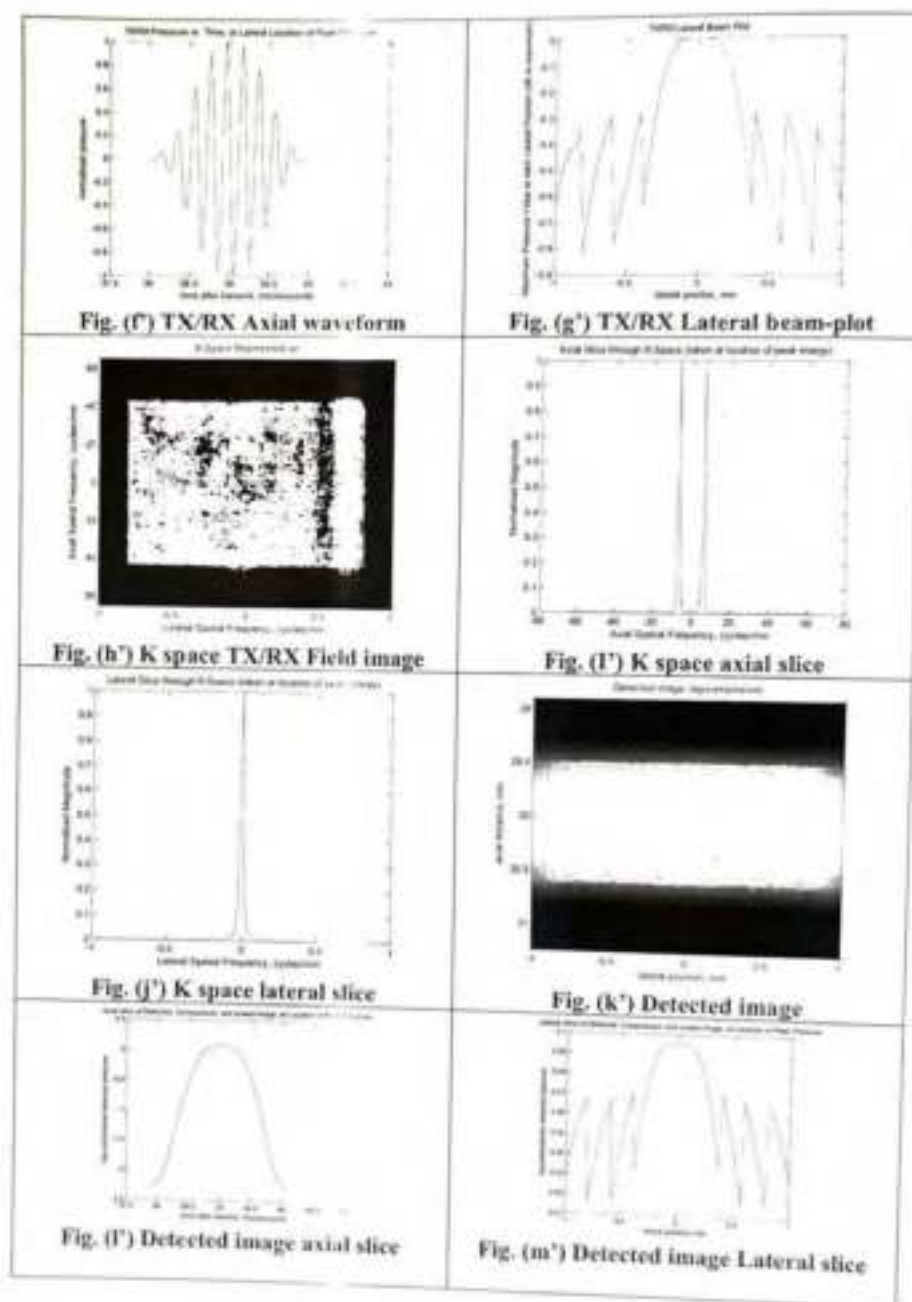


Fig. (e') TX/RX Field image





Comparative Study of 4-Element and 8-Element Array Transducers at Center Frequency 5 MHz

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ABSTRACT: Ultrasonic arrays are used in many applications including medical imaging. In this specific case is important to achieve precise information about the magnitude and position of the peak pressure, intensity, detected image and various pressure fields produced by the transducer probe. This paper presents the study of responses of 4 element and 8 element linear arrays with center frequency 5MHz for ultrasonic measurements.

KEY WORDS: Detected Image, Field-II GUI, Linear array transducer, TX/RX Pressure field.

I. INTRODUCTION

More than the last half century much development has been made in medical device technology. One particular medical technology that has enhanced rapidly over the last 30 years is ultrasound. This advancement in technology however has brought with it the rapid obsolescence of system design. The achievement of modern electronics is built on the possibility to precisely predict system performance by the use of simulation tools. This model can be extended to components such as piezoelectric transducers attached to the electronics [1]. The capability to simulate both piezoelectric transducer and electronics jointly renders possible efficient optimizations at system level, i.e. minimizing size, price and power consumption [2].

II. SPATIAL IMPULSE THEORY

The pressure field generated by the aperture is found by the Rayleigh integral [5]

$$p(\vec{r}_1, t) = \frac{P_0}{2\pi} \int_s \frac{\partial v_n(\vec{r}_2, t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{|\vec{r}_1 - \vec{r}_2|} ds \quad (1)$$

Where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, v_n is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_1$, then: where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, v_n is the velocity normal to the transducer surface. Using the velocity potential,

and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

$$\Psi(\vec{r}_1, t) = v_s(t) * \int \frac{\partial(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{2\pi |\vec{r}_1 - \vec{r}_2|} \quad (2)$$

Where * denotes convolution in time. The integral in this equation

$$h(\vec{r}_1, t) = \int \frac{\partial(t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{2\pi |\vec{r}_1 - \vec{r}_2|} \quad (3)$$

Represent the spatial impulse response. The continuous wave field can be found from the Fourier transform of

$$p(\vec{r}_1, t) = \rho_0 \frac{\partial v(t)}{\partial t} * h(\vec{r}_1, t) \quad (4)$$

The impulse response includes the excitation convolved with both the transducers electro-mechanical impulse response in transmit and receive. The final signal for a collection of scatters is calculated as a linear sum over all signals from the different scatters [3-7].

III. SIMULATION OF LINEAR ARRAY TRANSDUCER

The linear array is the fundamental type of multi-element transducer and it scans the region of interest by exciting the elements situated over the region. The field is focused on the region by introducing time delay in the excitation of the concerned individual elements, so initially concave beam is emitted.

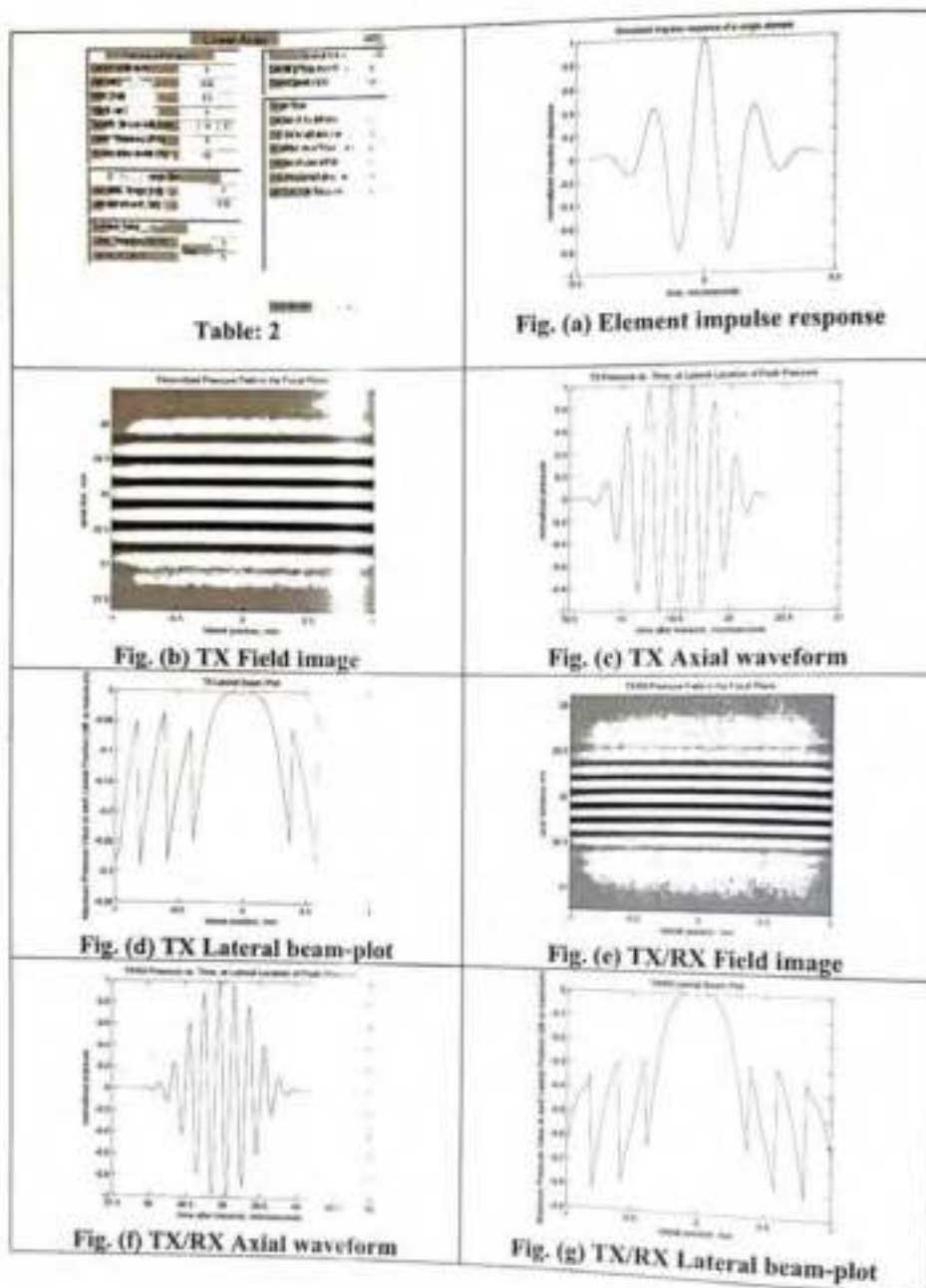
Here a Fig.1 shows general design format of 16 element linear array transducer having height, width and kerf of individual element are taken as 5 mm, 0.2 mm and 0.02 mm respectively. The transducers are situated at the center of the coordinate system. To achieve focal length of 30 mm from the center of transducer the electronic focusing is included.

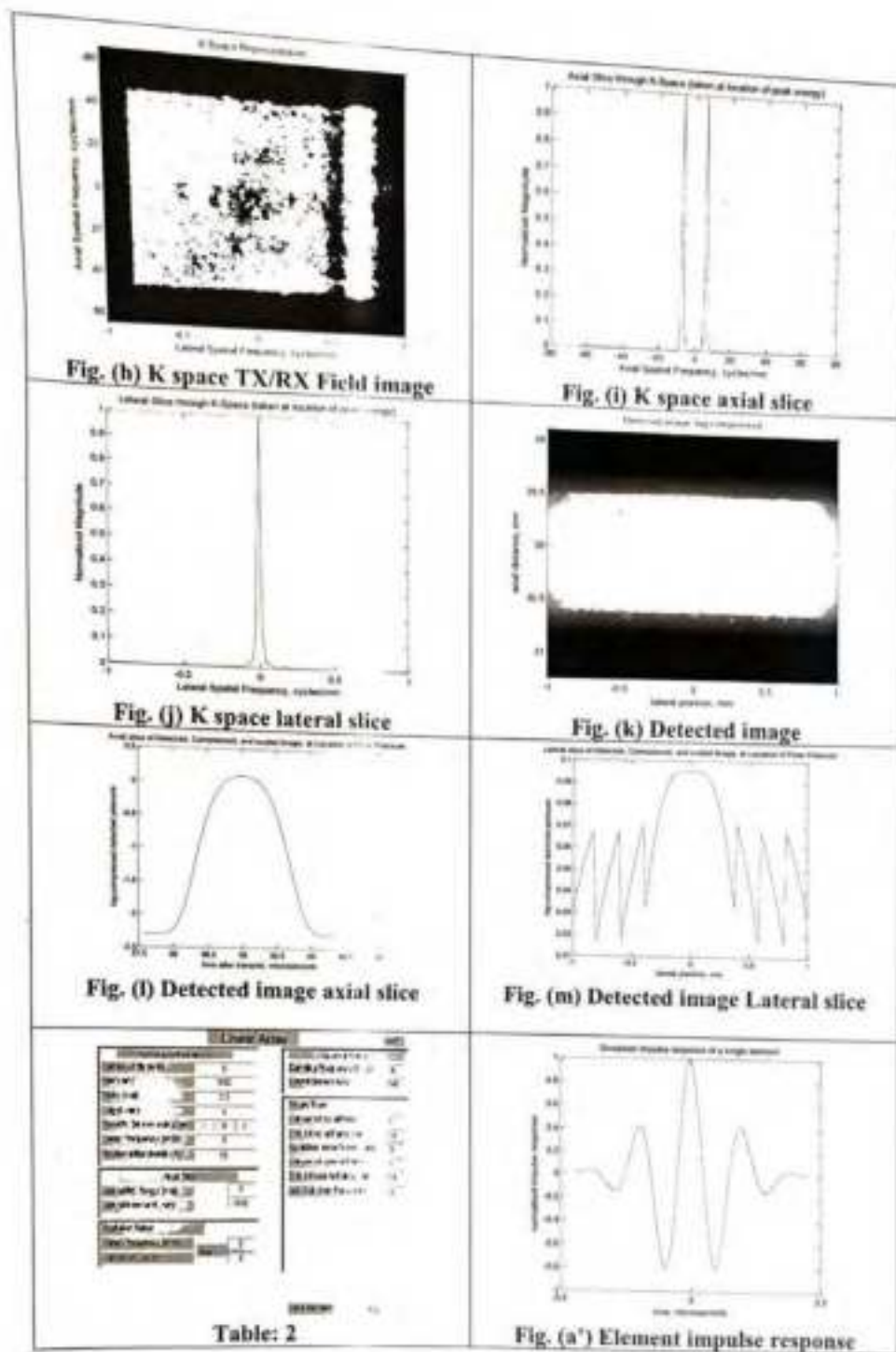


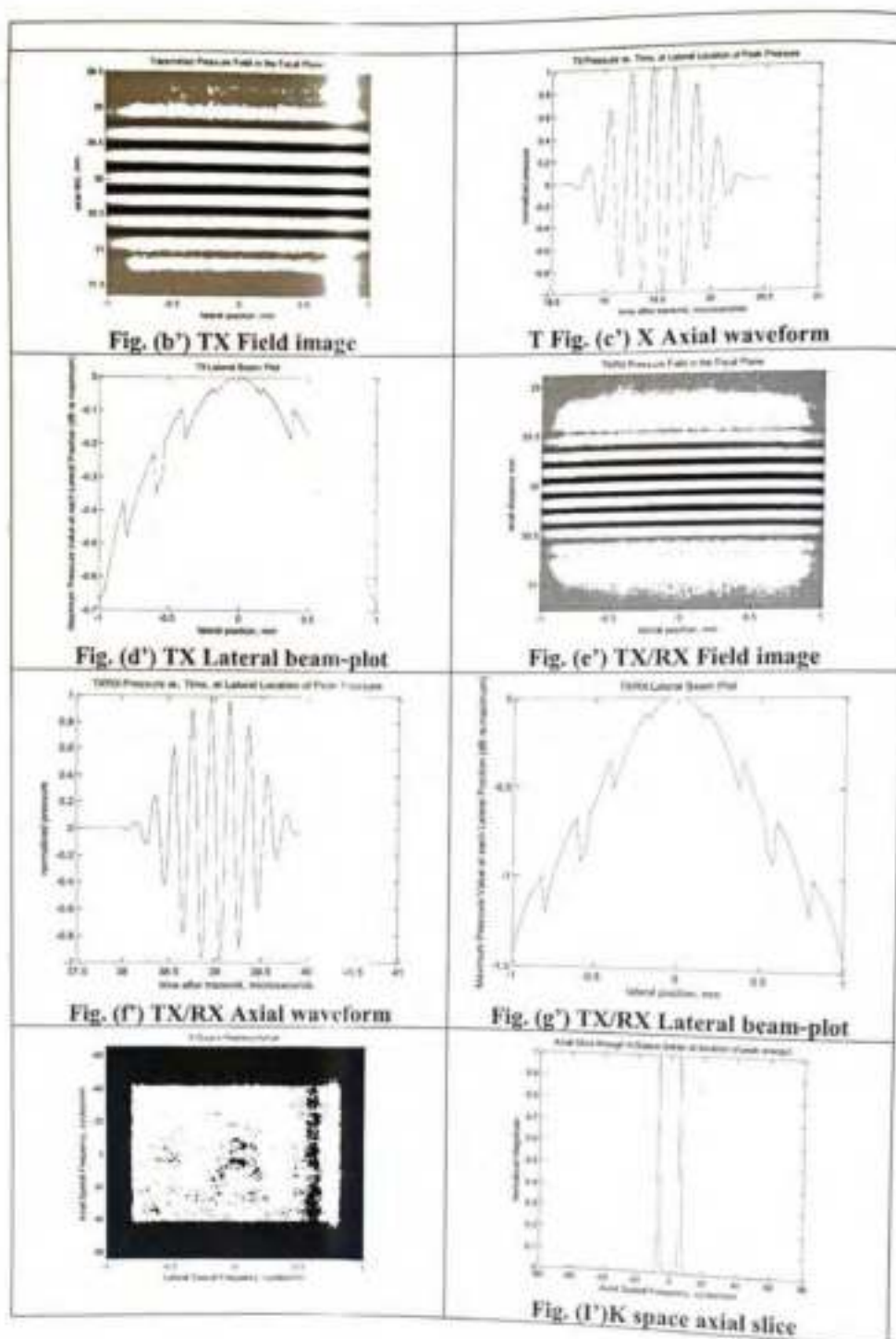
In this paper a linear array transducer of 4-elements and 8-elements is simulated by using FIELD-II GUI program with center frequencies 5MHz. For these specified linear array transducers, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30)

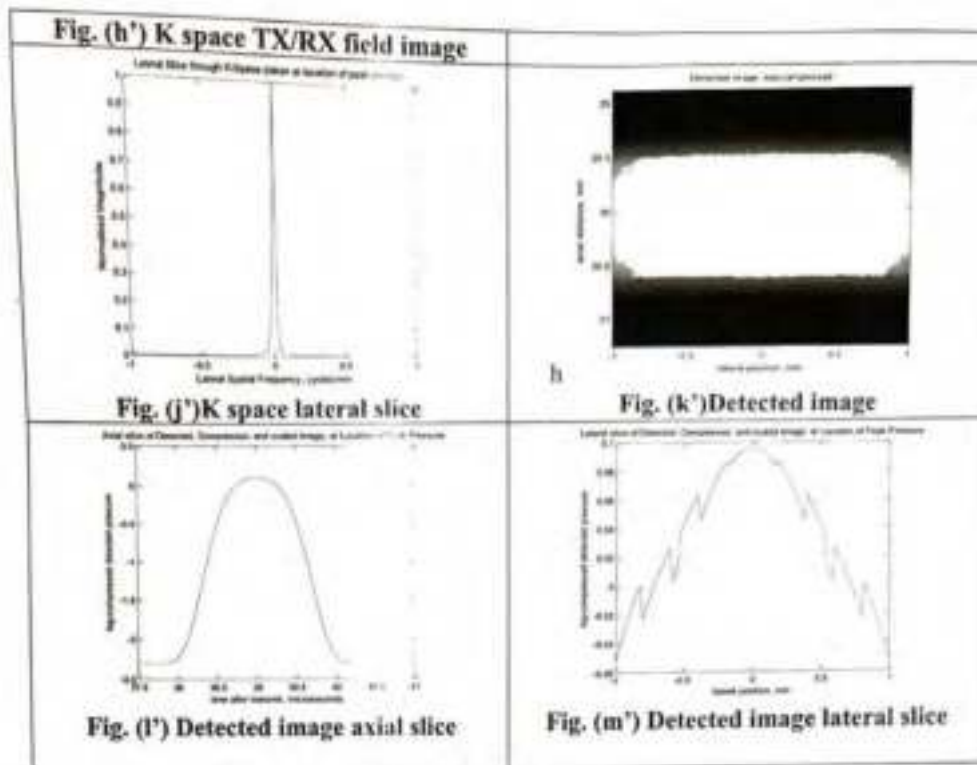
IV. RESULT AND DISCUSSION

The calculation of the impulse response is facilitated by projecting the field point onto the plane of the aperture. In this way, the problem became two-dimensional and the field point is given as a (x, y) coordinate set and a height z above the plane. The spatial impulse response is, thus, determined by the relative length of the part of the arc that intersects the aperture [8]. Thereby it is the crossing of the projected ultrasonic waves with the edges of the aperture that determines the spatial impulse responses as a function of time. In this paper by using FIELD-II program simulated 4-element and 8-element linear array transducers with center frequency $f_0 = 5\text{MHz}$. The speed of sound in tissue is $c = 1540\text{m/s}$. The sampling frequency used was $f_s = 100\text{MHz}$. The elements had a width and height of 0.25mm and 5mm respectively. The focal-point was set to 30mm. Table: 1 and Table: 2 shows the parameters for 4-element array transducer and 8-element array transducer, excitation pulse and medium used for these center frequency (f_0) used is 5 MHz. Figs. (a-m) shows; Element impulse response for 4-element array and figs. (a'-m') Element impulse response for 8-element array transducer, TX Field image, TX Axial waveform, TX Lateral beam plot, TX/RX Field image, TX/RX Axial waveform, TX/RX Lateral beam plot, K-space TX/RX field image, K-space axial slice, K-space lateral slice, Detected image, Detected image axial slice and Detected image lateral slice for 4-element array transducers and 8-element array transducers.









V. CONCLUSION

The paper attempts to present a coherent analysis of the focusing strategies for 2-D array transducer design and properties, based on linear acoustics. The delays on the individual transducer elements and their relative weight or apodization are changed continuously as a function of depth. This yields near perfect focused images for all depths. Similarly, if number of arrays in the transducer are increased then contrast of displayed image, is increased thus, benefitting the diagnostic importance of ultrasonic imaging. The detected image response for 8-element array has higher contrast than 4-element array also the pressure field generated by 8-element is high this increases diagnostic value.

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Development of 16 elements linear transducer using field II :Dr. S. A. Rodge; R. D. Chaudhary





Development of 16 Element linear array transducer Using Field-II

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Abstract: Piezoelectric transducers are significant elements of many broadband ultrasonic systems, either pulse-echo or through-transmission, used for imaging and detection purposes. In ultrasonic broadband applications such as medical imaging, or non-destructive testing, piezoelectric transducers should generate/receive ultrasonic signals with good efficiency over a large frequency range. This implies the use of piezoelectric transducers with high sensitivity, broad bandwidth and short-duration impulse responses. High sensitivity provides large signal amplitudes which determine a good dynamic range for the system and the short duration of the received ultra-sonic signal provides a good axial resolution. This paper presents the simulation of linear array transducers for ultrasonic measurements.

Keywords—Ultrasonic, Linear array Transducer, medical imaging, Field-II GUI, TX/RX Fields, detected image, TX/RX Axial slice, TX/RX Axial slice.

1. INTRODUCTION

For the period of the second half of 20th current century the medical imaging is grown through Ultrasound tool speedily. The part of novel technology is the use of computers to decide problems by simulating theoretical models (Numerical simulations) that has taken place alongside pure theory and experiment during the last few decades. These numerical simulations permit one to resolve problems that may not be accessible to direct experimental study or too complex for theoretical analysis. Computer simulations can link the gap between analysis and experiment [1].

These numerical simulations have emerged as a new branch in science and technology complementing both experiments and theory. A simulation can sometimes replace physical experiments, even though most often a simulation and an experiment are complementary. The



results of scientific experiments are often explained by simulations and simulations are often calibrated by experiments. The experiments provide input for the simulations, which are viewed as experimenting with theoretical models. The feedback of numerical results into theoretical modeling and continues interaction with laboratory experiments and analytical theory makes computing a vital tool for science. Consequently the increased in computing power in both speed and storage has given computational electronics its significance. Improved computer capacity and the solution algorithms themselves, have a big outcome on the excellence of solution obtained. A numerical model can be used to understand measurements and observations enlarge existing analytical models into new parameter regimes and quantitatively test existing theories that can be done by comparing model predictions to experimental information.

The mutual weak point of both experiment and theory is cover up by the numerical simulations examination and experiment. A third dimension in ultrasonic measurements, of equivalent status and significance to experiment and analysis is nothing but the simulation determination [1]. It has taken an everlasting place in every one aspect of ultrasonic measurements from basic research to engineering design.

A novel and potentially powerful tool is the computer experiment. One can resolve novel and uncertain aspects of usual process, by combining predictable theory, experiment and computer simulation [1]. Such aspects could frequently neither have been understood nor revealed by analysis or experiments alone.

More than the last half century much development has been made in medical device technology. One particular medical technology that has enhanced speedily over the last 30 years is ultrasound. This advancement in technology however has brought with it the rapid obsolescence of system design. The accomplishment of modern electronics is built on the possibility to precisely predict system performance by the use of simulation tools. This model can be extended to components such as piezoelectric transducers attached to the electronics [2]. The ability to simulate both piezoelectric transducer and electronics together renders possible efficient optimizations at system level, i.e. minimizing size, price and power consumption [3].

The systems for images processing in the medical field are very important calling for new techniques, much more superior and performing than they used to be, in order to provide a

acceptable analysis and diagnosis. Amongst the medical techniques using computer sciences, it can be mentioned: scintigraphy, echography, tomography, radiography, quantitative microscopy, nuclear magnetic resonance. Ultrasound, widely used in many areas of medicine, provides a secure and efficient means for diagnosis and therapy. When the medium becomes complex solving the wave propagation formula becomes virtually not possible. Modeling becomes much more complex inside the body because the ultrasound propagation speed is different for each tissue and it is known that tissues are not a homogeneous medium for ultrasound wave propagation [4]. Therefore, it is important to know how the ultrasound wave is generated and the ultrasound wave beam shaped.

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Where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, v_n is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then: where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, v_n is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

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$$h(\vec{r}_1, t) = \int_S \frac{\partial (t - |\vec{r}_1 - \vec{r}_2|/c)}{2\pi |\vec{r}_1 - \vec{r}_2|} dS \quad (3) \text{Represent the spatial impulse}$$

response. The continuous wave field can be found from the Fourier transform of

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Fig. (1) Design format of linear array transducer (Height=5mm, Width=0.25mm, Kerf=0.02mm)

In this paper a linear array transducer of 16 elements is simulated using FIELD-II program with center frequencies 5MHz. For this specified linear array transducer, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30)

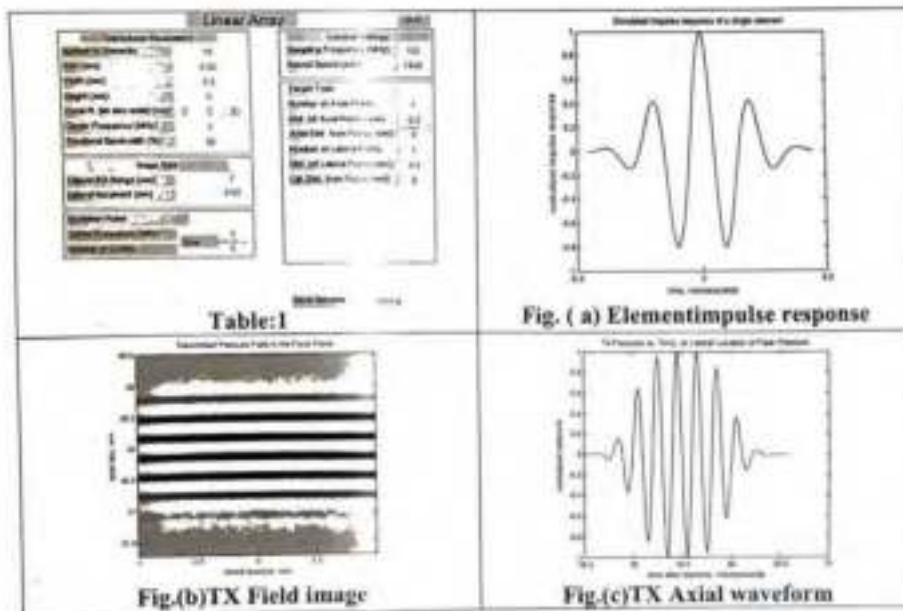
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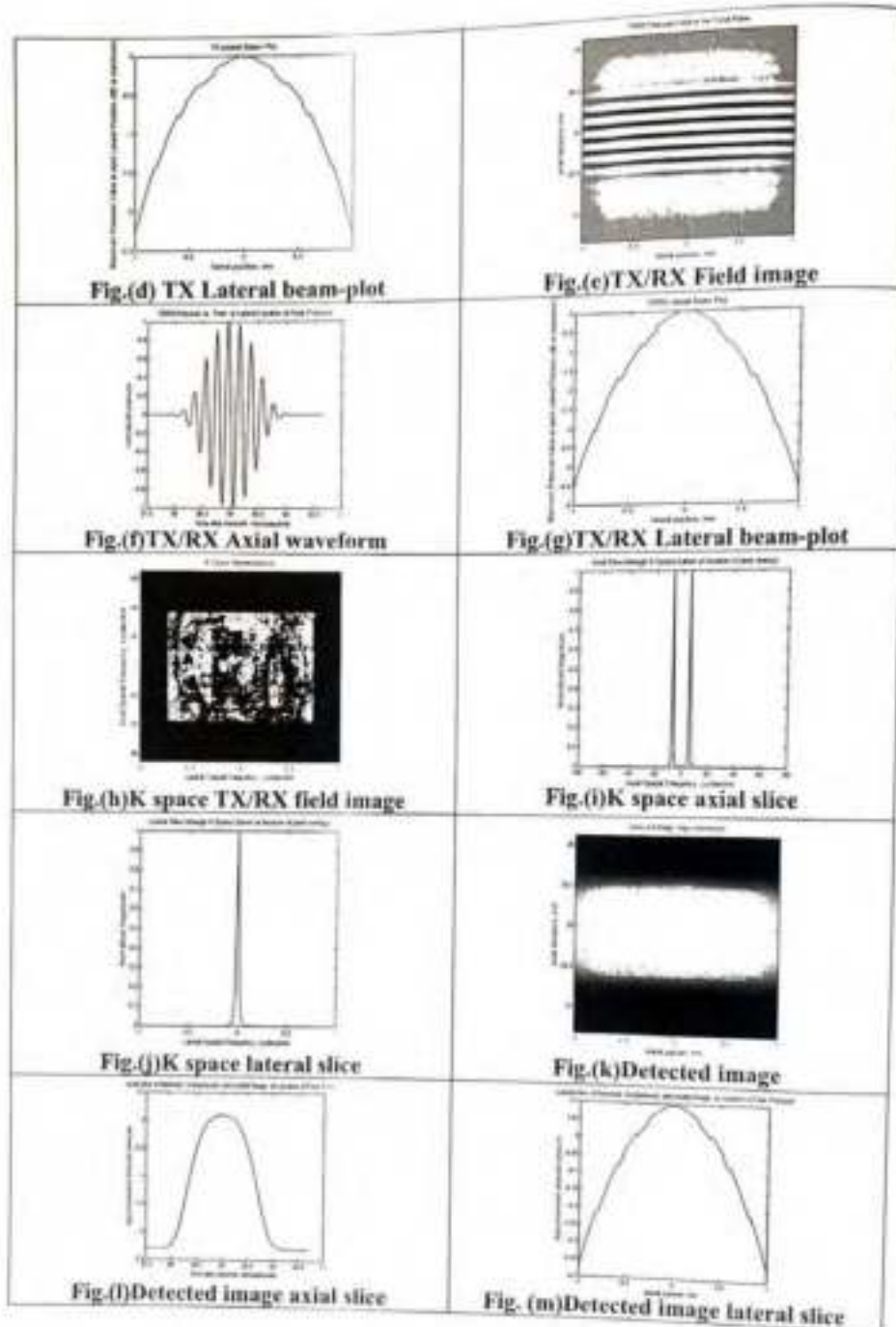
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Table: 1 shows the parameters for 16 element array transducer, excitation pulse and medium used for this centre frequency (f_0) used is 5MHz. Figs. (a-m) shows; Element impulse response for 16 element array, TX Field image for 16 element array, TX Axial waveform for 16 element array, TX Lateral beam plot for 16 element array, TX/RX Field image for 16 element array, TX/RX Axial waveform for 16 element array, TX/RX Lateral beam plot for 16 element array, K- space TX/RX field image for 16 element array, K-space axial slice for 16 element array, K-space lateral slice for 16 element array, Detected image for 16 element array, Detected image axial slice for 16 element array and Detected image lateral slice for 16 element array.

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The paper attempts to present a coherent analysis of the focusing strategies for 2-D array transducer design and properties, based on linear acoustics. The delays on the individual transducer elements and their relative weight or apodization are changed continuously as a function of depth. This yields near perfect focused images for all depths. Similarly if number arrays in the transducer are increased then contrast of displayed image, has increased thus, benefitting the diagnostic importance of ultrasonic imaging.

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Linear Array Transducer with Center Frequency 7.5 MHz

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ABSTRACT

Over the past decades, ultrasound imaging technology has satisfied outstanding improvement in obtaining significant diagnostic information from patients in a fast, noninvasive approach. Piezoelectric transducers are important elements of various broadband ultrasonic systems, either pulse-echo or through-transmission, used for imaging and detection purposes[1]. In ultrasonic broadband applications such as medical imaging, or non-destructive testing, piezoelectric transducers should generate/receive ultrasonic signals with good efficiency over a large frequency range. This implies the use of piezoelectric transducers with high sensitivity, broad bandwidth and short-duration impulse responses. High sensitivity provides large signal amplitudes which determine a good dynamic range for the system and the short duration of the received ultra-sonic signal provides a good axial resolution. This paper presents the simulation of 8-element linear array transducers with center frequency 10 MHz, using Field-II GUI program for ultrasonic measurements.

Keywords-Detected image, Field-II GUI, Linear array Transducer, medical imaging, TX/RX Fields, TX/RX Axial slice, TX/RX lateral slice, Ultrasonic.

INTRODUCTION

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Where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then: where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

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Fig. (1) Design format of linear array transducer (Height=5mm, Width=0.25mm, Kerf=0.02mm)

In this paper a linear array transducer of 8 elements is simulated using FIELD-II program with center frequencies 7.5MHz. For this specified linear array transducer, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30).

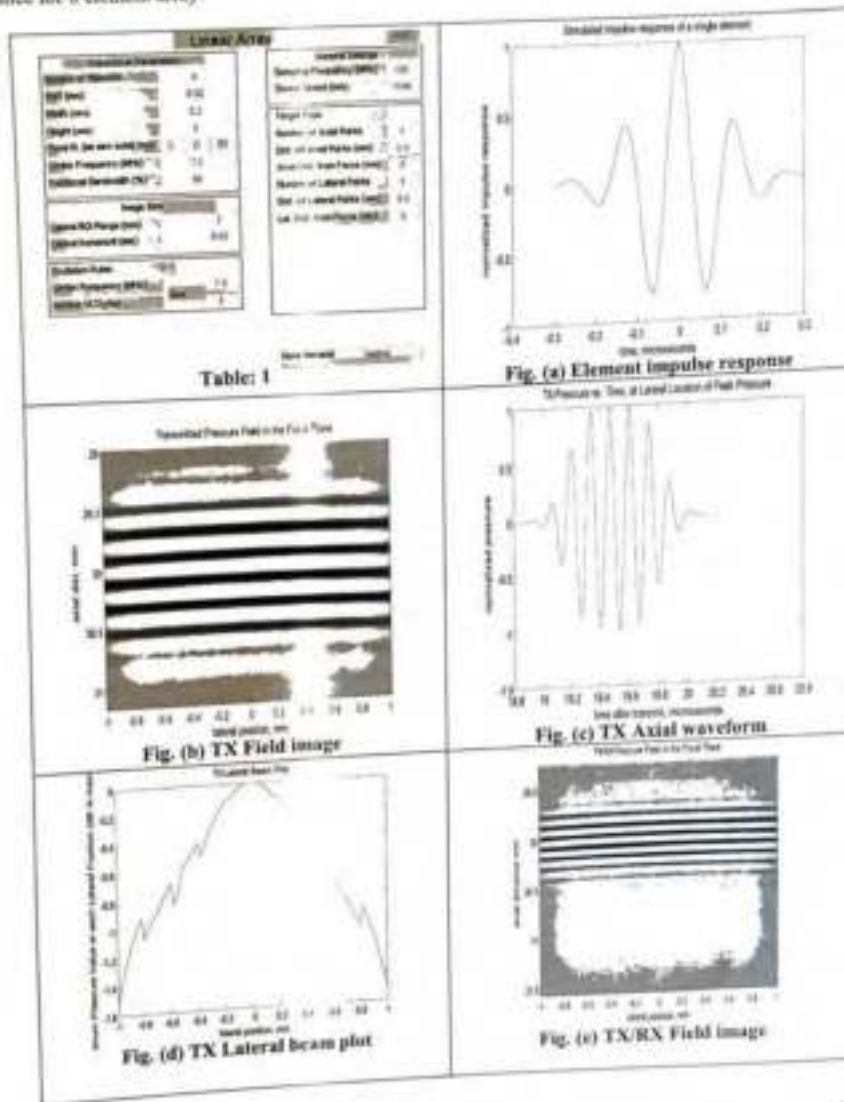
RESULT AND DISCUSSION

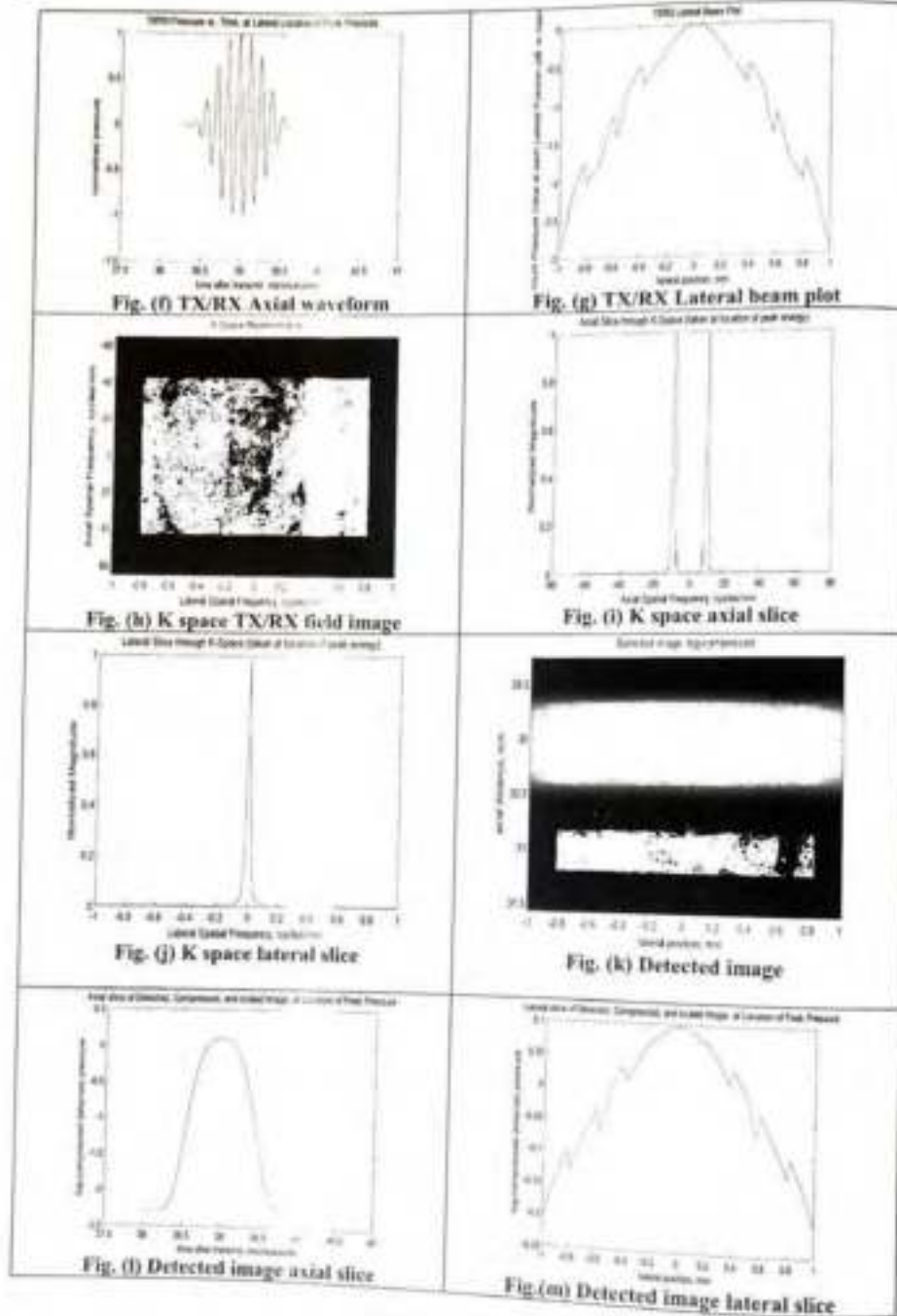
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frequency used was $f_0 = 100\text{MHz}$. The elements had a width and height of 0.25mm and 5mm respectively. The focal-point was set to 30mm .

Table: 1 shows the parameters for 8 element array transducer, excitation pulse and medium used, for this centre frequency (f_0) used is 7.5MHz . Figs. (a-m) shows: Element impulse response for 8 element array, TX Field image for 8 element array, TX Axial waveform for 8 element array, TX Lateral beam plot for 8 element array, TX/RX Field image for 8 element array, TX/RX Axial waveform for 8 element array, TX/RX Lateral beam plot for 8 element array, K-space TX/RX field image for 8 element array, K-space axial slice for 8 element array, K-space lateral slice for 8 element array, Detected image for 8 element array, Detected image axial slice for 8 element array and Detected image lateral slice for 8 element array.





CONCLUSION

The paper attempts to present a coherent analysis of the focusing strategies for 2-D array transducer design and properties, based on linear acoustics. The delays on the individual transducer elements and their relative weight or apodization are changed continuously as a function of depth. This yields near perfect focused images for all depths and



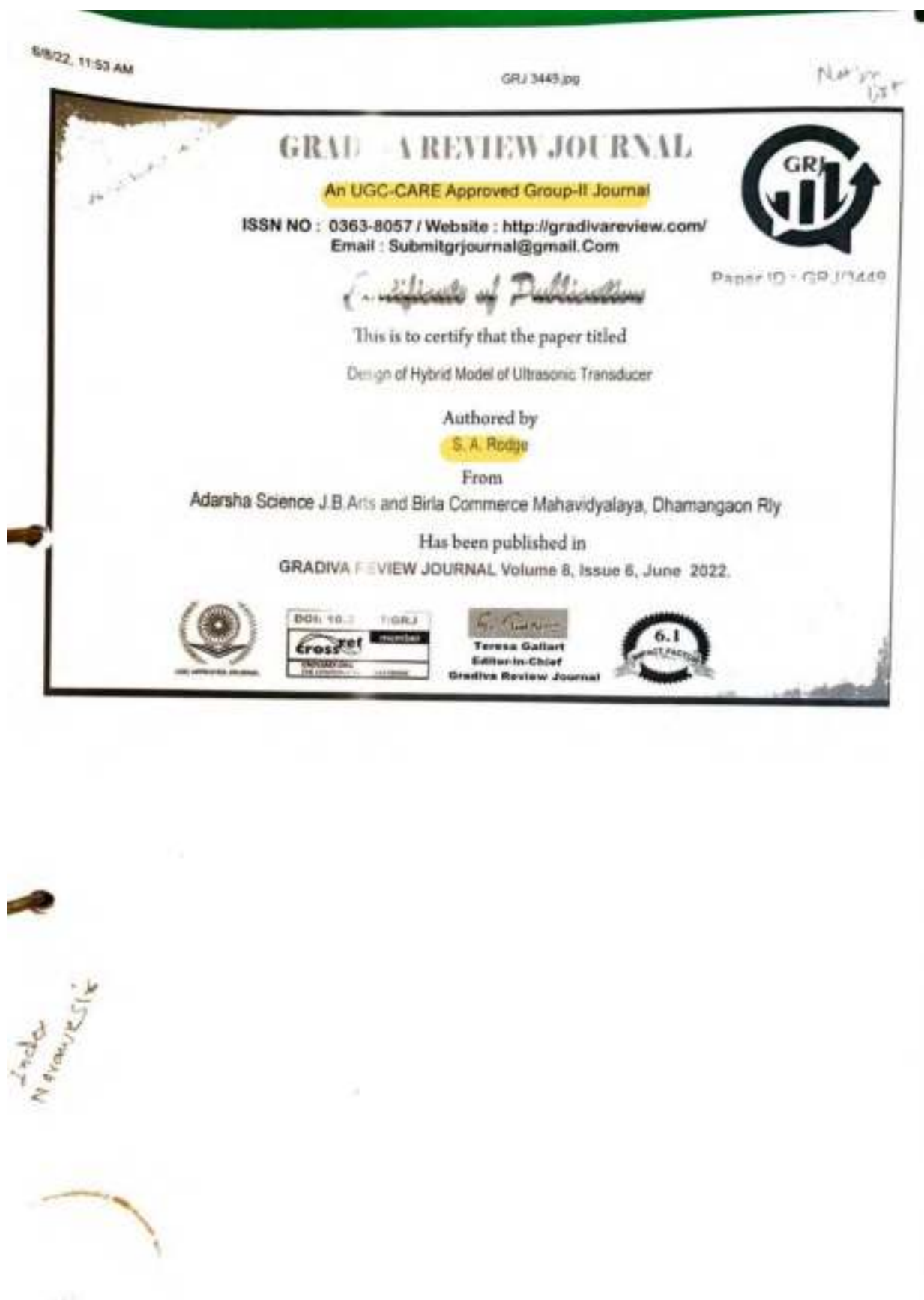
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has increased the contrast in the displayed image, thus, benefitting the diagnostic importance of ultrasonic imaging. If the center frequency and number of elements in transducer is increased then contrast in the detected image is increased, this also increases the diagnostic status of ultrasonic imaging.

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ABSTRACT: Newly, ultrasound transducer projecting in several applications especially in medical field to improve the health services either for diagnostic. The advent of ultrasound applications raises the need of reliable transducer to comply with that purpose. As polymer material being popular in medical ultrasound, there are chances in combining it with former piezoelectric ceramic material in designing diagnostic transducer to get hybrid characteristics required for multi-frequency application. In this work, SPICE model of ceramic-polymer piezoelectric has been developed. With signal conditioner circuit, complete analog system for ultrasound has also been developed. Initially transducer test for ceramic and polymer model were generated. By filtering and amplifying frequency range from 1 MHz until 10 MHz, the system bargains wideband medical ultrasonic acceptance. It gives smooth result of ultrasound signal for medical purposes.

KEY WORDS: Piezoelectric transducing, SPICE model, Hybrid transducer model

Introduction

Ultrasonic sensing techniques have become mature and are extensively used in the numerous fields of engineering and basic science. Actually, many types of conventional ultrasonic instruments, devices and sophisticated software are commercialized and used for both industrial and medical applications. One of advantages of ultrasonic sensing is its outstanding capability to probe inside objectives nondestructively because ultrasound can propagate through any kinds of media including solids, liquids and gases except vacuum. In typical ultrasonic sensing the ultrasonic waves are travelling in a medium and often focused on evaluating objects so that a useful information on the interaction of ultrasonic energy with the objects are acquired as ultrasonic signals that are the waveforms variations with transit time. Such ultrasonic data provides the fundamental basis for describing the outputs of ultrasonic sensing and evaluating systems. In this work, SPICE model of ceramic-polymer piezoelectric has been described. With signal conditioner circuit, complete analog system for ultrasound has also been developed. Initially transducer test for ceramic and polymer model were generated. By filtering and amplifying frequency range from 1 MHz until 10 MHz, the system offers wideband medical ultrasonic acceptance. It gives smooth result of ultrasound signal for medical purposes.

The model developed for piezoelectric transducer is shown in fig.1. The block T_1 represents the transmission line. Independent sources V_1 and V_2 are zero value sources, which are used as ammeters in the circuit. F_1 and F_2 are dependent current sources. The value of F_1 is given by $F_1 = k C_d \times I(V_1)$, where $I(V_1)$ is the current through V_1 . The voltage across the dependent voltage source E_1 is given by $E_1 = V(4)$, where $V(4)$ is the voltage at node 4 i.e. the voltage across C_d . The dependent current source F_2 which charges C_f is given by $F_2 = k \times I(V_2)$, where $I(V_2)$ is the current through V_2 and k is the ratio of the piezoelectric stress constant in the direction of propagation and the permittivity with constant strain. Resistor R_f is included to prevent node 4 from being a floating node. From the mechanical side (i.e. transmission line T_1), the difference between the velocity of each surface normal to the propagation path, represented by the currents u_1 and u_2 controls the current source F_2 . The node labels E, B and F, respectively, denote the electrical, back, and front ports.

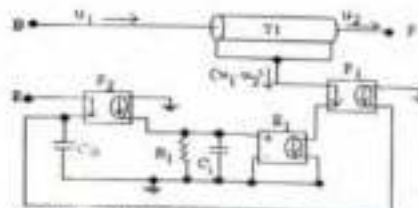


Fig. 1 Model of a piezoelectric transducer
MODEL OF PZT 5A- PVDF HYBRID TRANSDUCER

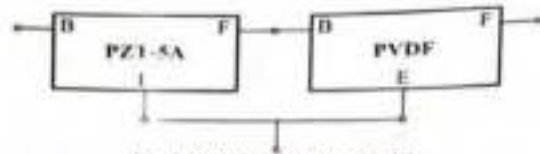


Fig. 2 : Hybrid Transducer Model

Fig. 2 shows series configuration of two material's equivalent models. Although in this model lossy characteristics (mechanical, dielectric, and electromechanical) of piezoelectric are considered, it must be taken to note that polymer material has complex additional losses than those of ceramic material. The piezoelectric material PZT-5A and PVDF whose material data was obtained from [1], [2], [3], and [6] was chosen and given in table 1

TABLE 1. PHYSICAL PROPERTIES OF TRANSDUCERS AT 25 °C

S.No	Physical properties at 25°C	PZT-5A	PVDF
1	Density (ρ) (kg/m ³)	7750 [3]	1780 [2]
2	Mechanical Q (Q_m)	75 [3]	19 [6]
3	Sound velocity (c) (m/s)	4350 [3]	2200 [2]
4	Permittivity with constant strain (ϵ^T) (C ² /Nm ²)	7.35×10^{-9} [3]	55.78×10^{-9} [6]
5	Piezoelectric stress constant (e^{ij}) (C/m ²)	15.8 [3]	0.16 [6]
6	Acoustic Impedance (MRayl)	33.7 [3]	2.7 [2]
7	Piezoelectric Constant (10^{-12} C/N)	$d_{31} = 374$ [1] $d_{32} = 584$ [1]	$d_{31} = 23$ [6] $d_{32} = 4$ [6] $d_{33} = 33$ [6]
8	Coupling factor (K_{31})	0.66 [3]	0.2 [6]

Assisted with the definition of the low loss characteristic impedances equation, following relationships can be obtained

$$L = A \cdot \rho \quad (1)$$

$$C = \frac{1}{A \rho c^2} \quad (2)$$

$$R = 2 \rho c A \alpha_s \quad (3)$$

$$G = \frac{2 \alpha_s}{\rho c A} \quad (4)$$

Mechanically, a transmission line T of length len (m) represents the acoustical layer. The length is selected to achieve the desired center frequency f (Hz) of the transducer. With fixed ends, the piezoelectric plate has a fundamental resonant frequency as

$$f = \frac{c(T)}{2 \cdot len} \quad (5)$$

Where $c(T)$ is the velocity of sound through it at temperature T .

Using equations (1), (2) and (3) the piezoelectric material density ρ , required for transmission line, L and C values can be calculated. The mechanical factor Q_m describes the shape of the resonance peak in the frequency domain. The relation between angular frequency ω , inductance L and the resistance R is given as [5]:

$$Q_m(T) = \frac{\omega L}{R} \quad (6)$$

In the electrical section, the static capacitance $C_0(T)$ at temperature T is calculated as

$$C_0(T) = \frac{\epsilon^T(T) \cdot A}{len} \quad (7)$$

where $\epsilon^T(T)$ (C²/Nm²) is the permittivity with constant strain at temperature T [3]. The latter is related to the

permittivity with constant stress (free) ϵ^T as :

$$\frac{\epsilon^T(T)}{\epsilon^T(T)} = \frac{1}{1 - k^2(T)} \quad (8)$$

Where $k(T)$ is the piezoelectric coupling constant at temperature T .

The mechanical and electrical sections interact with two current controlled sources. From the mechanical side, the deformation itself is not measurable, but the current representing the rate of deformation is the difference between the velocity of each surface normal to the propagation path, represented by the currents u_1 and u_2 , is the rate of deformation. This current $(u_1 - u_2)$ controls the current source F_1 . It has a gain equal to the product of the transmitting constant h (N/C), and the capacitance C_0 . h is the ratio of the piezoelectric stress constant e^{31} (C/m²) in the direction of propagation and the permittivity with zero or constant strain ϵ^T . In the thickness mode it is [3].

$$h(T) = \frac{e^{31}(T)}{\epsilon^T(T)} \quad (9)$$

This source's output is in parallel with the capacitor $C_0(T)$. The result is a potential difference across the capacitor that is proportional to the deformation. In the electrical section, the current through the capacitor $C_0(T)$ controls the current source F_2 . The gain for this second current source is $h(T)$. Its output needs to be integrated to obtain the total charge on the electrodes that proportionally deforms the transducer. The integration is performed by the capacitor C_1 . The voltage controlled voltage source E_1 with unity gain is a one-way isolation for the integrator.

To evaluate the model, the model parameters of PZT-5A and PVDF transducers were calculated using equations (1, 2, 3, 5, 6, 7, 9) and given in table 2.

TABLE 2 MODEL PARAMETERS OF TRANSDUCERS

S.No.	Model parameters	PZT-5A	PVDF
<i>Physical parameters</i>			
1	Diameter (mm)	12.7	12.5
2	Cross sectional area (A) (m ²)	0.0001267	0.0001227
3	Center frequency (MHz)	5MHz	5MHz
<i>Equivalent lossy transmission line parameters (Mechanical section)</i>			
4	C	53.8nF	945.3nF
5	R	411kΩ	361.18 kΩ
6	L	981mH	218 mH
7	G	0	0
8	len	435μm	220 μm
<i>Electrical section parameter</i>			
9	Static capacitance C_0	2.14nF	31.14nF
<i>Controlled sources parameter</i>			
10	Transmitting constant (h) (N/C)	2.15×10^5	2.87×10^6
11	Current source gain (F_2)	2.15×10^3	2.87×10^6
12	Dependant current source gain (F_1)	4.60	0.09
13	Voltage control voltage source gain (E_1)	1	1
14	R_1	1 KΩ	1 KΩ
15	C_1	1F	1F

SIMULATION SETUP FOR ULTRASONIC SYSTEM

The analogous simulation schematic setup is described in figure 3, with the transducer sub circuit shown in figure 1. In this schematic an ultrasonic probe with acoustic matching layer is symbolized by the two three-port blocks X1 and X2, which involve established PSPICE piezoelectric model. The measuring cell is modeled using lossy transmission line.

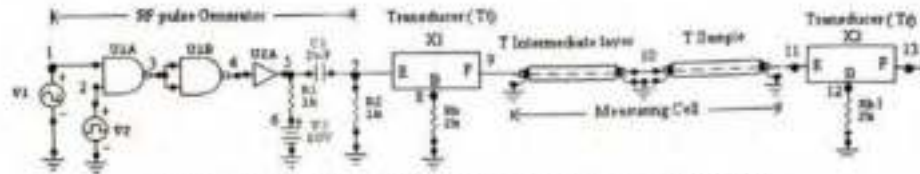


Fig.3 : Simulation setup Schematic for ultrasonic test system.

RESULT AND DISCUSSION

Transient analysis of the transducer model was done with the configuration shown in figure 4. An oscillation was observed after excitation of the piezoelectric crystal. The received signal was compared in the time domain. (Fig. 5, 6, 7)

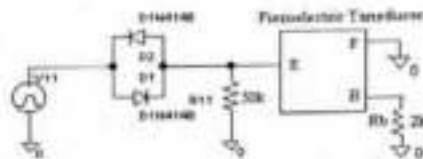


Fig. 4: Simulation setup for analysis of transient behavior of transducers.

Certain polymer characteristic losses are neglected to simplify the preliminary design at this stage. Fig. 6 shows the transient response of series configuration model.

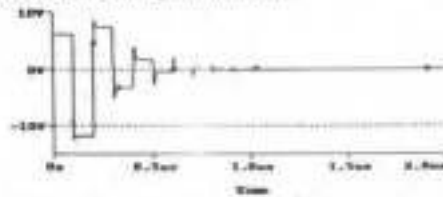


Fig 5 : Transient response of PZT-5A piezoelectric Transducer.

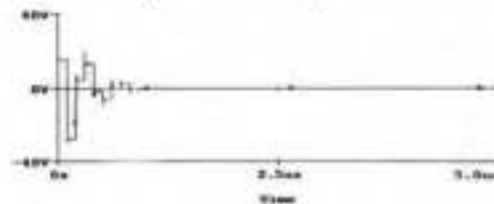


Fig 6: Transient response of PVDF Transducer

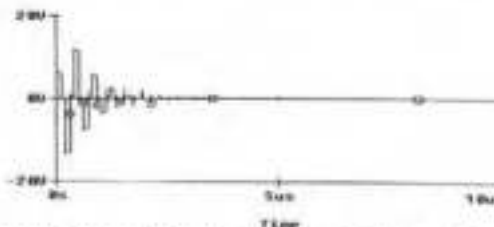


Fig. 7: Transient response of ceramic-polymer piezoelectric transducer (PZT-5A+PVDF) for multi-frequency ultrasonic system

Fig. 8 shows frequency response of transducer. AC analysis was conducted to observe frequency behavior from 1 MHz to 10 MHz. There are three peaks of power spectrum: at 2.8 MHz, 5.5 MHz, and 8 MHz. The last

spectrum is higher than another, but for overall dB, bandwidth from 2.5 MHz to 8.5 MHz is considered flat.

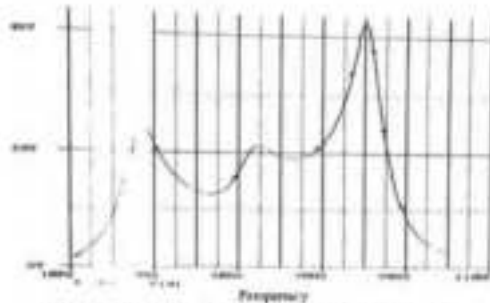


Fig. 8: Frequency response of Transducer

Electrical impedance of transducer was observed as in Fig 9. It gives turning point at about 1 MHz.

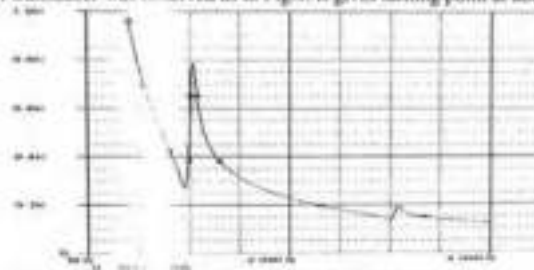


Fig. 9: Electrical Impedance of Transducer

Experimental validation for water sample is shown in fig. 10

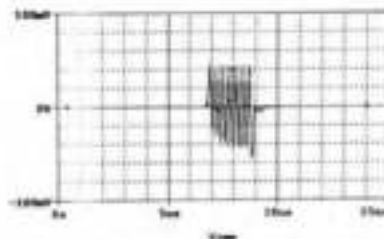


Fig. 10: Complete transient received by 5MHz ceramic-polymer piezoelectric transducer (PZT-5A+PVDF) for multi-frequency ultrasonic system at 25°C in distilled water at d= 1 cm.

CONCLUSION

A model of ceramic-polymer piezoelectric has been described. Simulation level shows that by hybridization, characteristics of both materials are providing a satisfying performance for multi-frequency transducer. Future work would be in detailed design which includes matching element, backing and loading consideration, also single or array configuration. Furthermore, the model could be improved so that it would be prepared for fabrication of hybrid multi-frequency ultrasound transducer.

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Analysis of 4 element and 16 element linear array Transducers 5 MHz

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ABSTRACT: Ultrasonic arrays are used in numerous applications including medical imaging. In this specific case is imperative to achieve precise information about the magnitude and position of the peak pressure, intensity, detected image and various pressure fields produced by the transducer probe. This paper presents the analysis of 4-element and 16-element linear arrays with center frequency 5MHz for ultrasonic measurements.

KEY WORDS: Detected Image, Field-II GUI, Linear array transducer, TX/RX Pressure field.

I. INTRODUCTION

More than the last half century widely development has been made in medical device technology. One particular medical technology that has improved rapidly over the last 30 years is ultrasound. This development in technology however has brought with it the rapid obsolescence of system design. The achievement of modern electronics is built on the possibility to precisely predict system performance by the use of simulation tools. This model can be extended to components such as piezoelectric transducers attached to the electronics [1]. The capability to simulate both piezoelectric transducer and electronics jointly renders possible efficient optimizations at system level, i.e., minimizing size, price and power consumption [2].

II. SPATIAL IMPULSE THEORY

The pressure field generated by the aperture is found by the Rayleigh integral [5]

$$p(\vec{r}_1, t) = \frac{\rho_0}{2\pi} \int \frac{\partial v_n(\vec{r}_2, t - \frac{|\vec{r}_1 - \vec{r}_2|}{c})}{|\vec{r}_1 - \vec{r}_2|} ds \quad (1)$$

Where the field point is denoted by $\vec{r}_1$ and the aperture by $\vec{r}_2$, v_n is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then: where the field point is denoted by $\vec{r}_1$ and

the aperture by $\vec{r}_2$, is the velocity normal to the transducer surface. Using the velocity potential, and assume that the surface velocity is uniform over the aperture making it independent of $\vec{r}_2$, then:

$$\Psi(\vec{r}_1, t) = v_n(t) * \int \frac{\partial \left(1 - \frac{|\vec{r}_1 - \vec{r}_2|}{c}\right)}{2\pi |\vec{r}_1 - \vec{r}_2|} \quad (2)$$

Where * denotes convolution in time. The integral in this equation

$$h(\vec{r}_1, t) = \int \frac{\partial \left(1 - \frac{|\vec{r}_1 - \vec{r}_2|}{c}\right)}{2\pi |\vec{r}_1 - \vec{r}_2|} \quad (3)$$

Represent the spatial impulse response. The continuous wave field can be found from the Fourier transform of

$$p(\vec{r}_1, t) = \rho_0 \frac{\partial v(t)}{\partial t} * h(\vec{r}_1, t) \quad (4)$$

The impulse response includes the excitation convolved with both the transducers electro-mechanical impulse response in transmit and receive. The final signal for a collection of scatters is calculated as a linear sum over all signals from the different scatters [3-7].

III. SIMULATION OF LINEAR ARRAY TRANSDUCER

The linear array is the fundamental type of multi-element transducer and it scans the region of interest by exciting the elements situated over the region. The field is focused on the region by introducing time delay in the excitation of the concerned individual elements, so initially concave beam is emitted.

Here a Fig.1 shows general design format of 16 element linear array transducer having height, width and kerf of individual element are taken as 5 mm, 0.2 mm and 0.02 mm respectively. The transducers are situated at the center of the coordinate system. To achieve focal length of 30 mm from the center of transducer the electronic focusing is included.

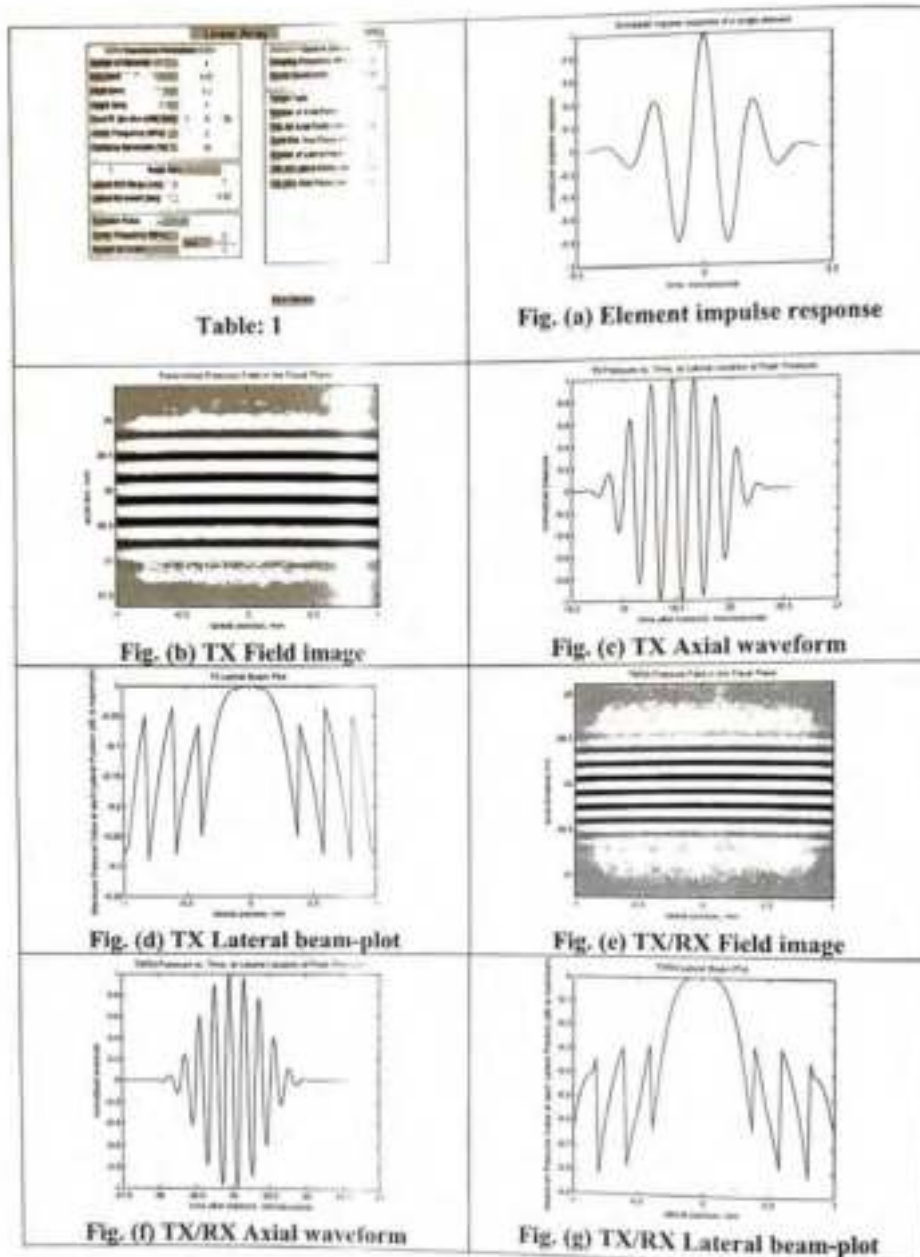


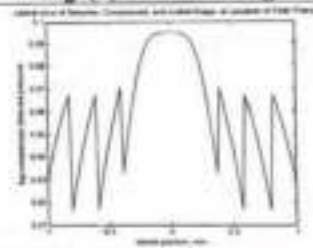
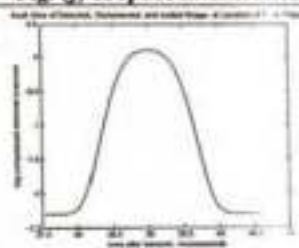
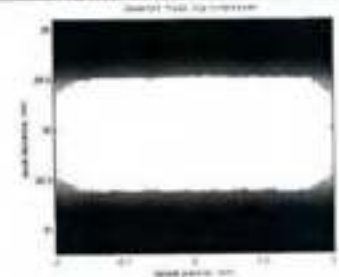
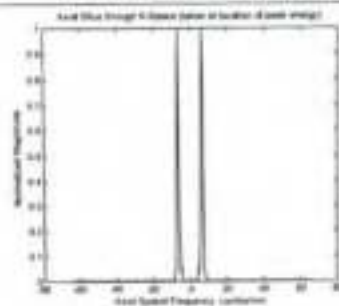
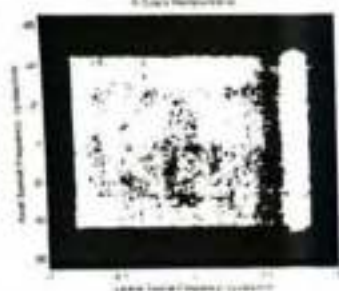
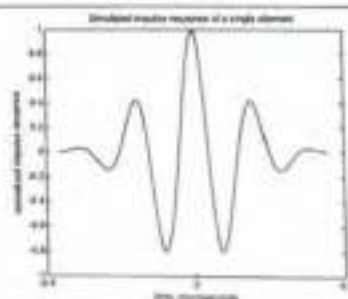
In this paper a linear array transducer of 4-elements and 16-elements is simulated by using FIELD-II GUI program with center frequencies 5MHz. For these specified linear array transducers, acoustic field generated is propagated through human body tissues and is observed at a focal distance i.e. (0, 0, 30)

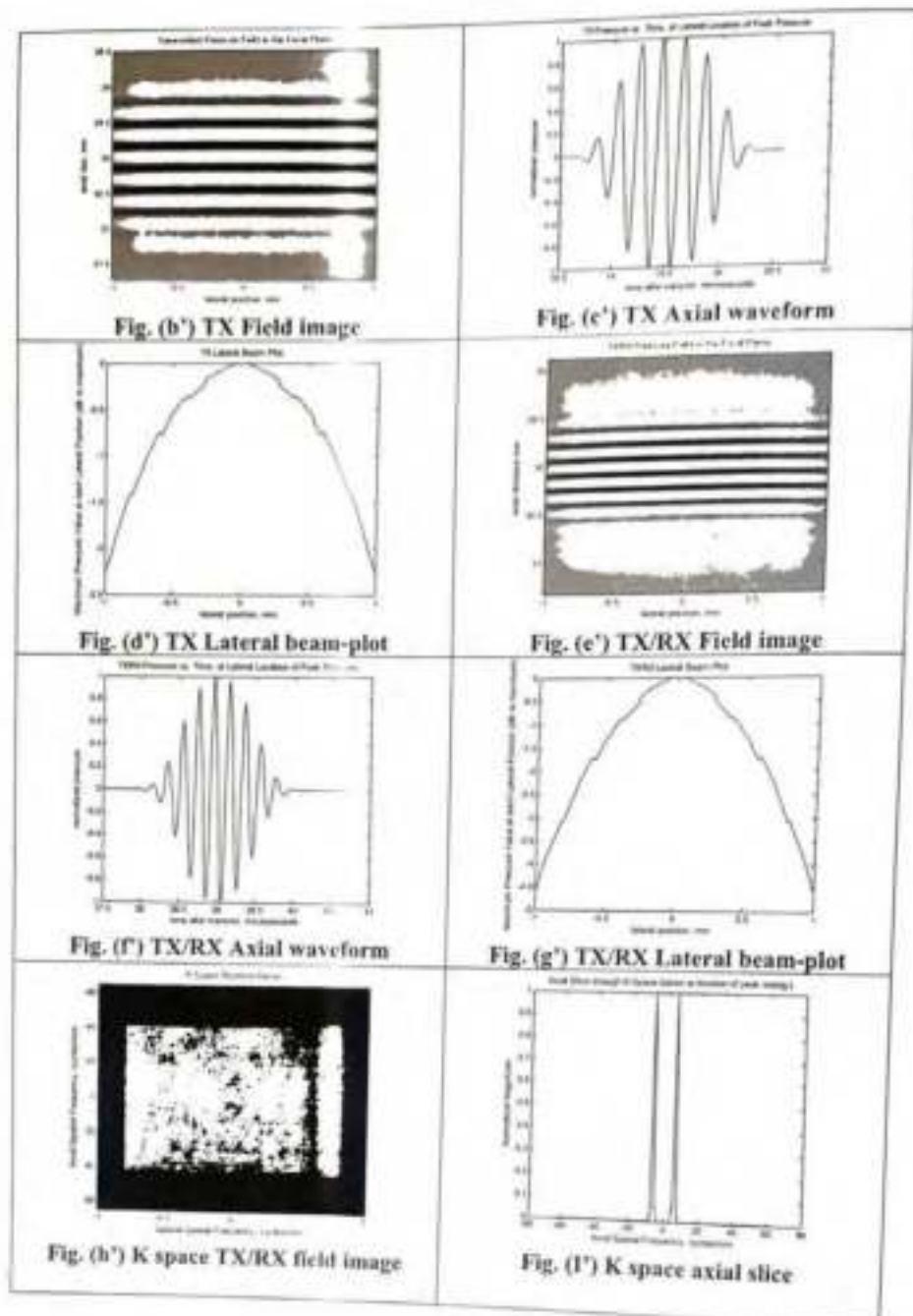
IV. RESULT AND DISCUSSION

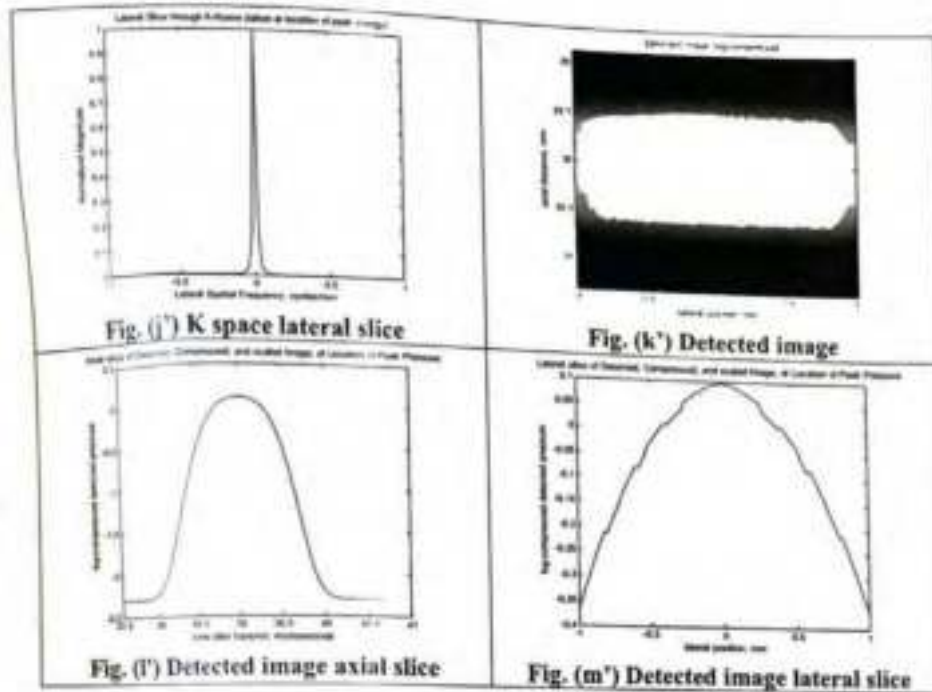
The calculation of the impulse response is eased by projecting the field point onto the plane of the aperture. In this way, the problem became two-dimensional and the field point is given as a (x, y) coordinate set and a height z above the plane. The spatial impulse response is, thus, determined by the relative length of the part of the arc that intersects the aperture [8]. Thereby it is the crossing of the projected ultrasonic waves with the edges of the aperture that determines the spatial impulse responses as a function of time. In this paper by using FIELD-II program simulated 4-element and 16-element linear array transducers with center frequency $f_0 = 5\text{MHz}$. The speed of sound in tissue is $c = f_0 \lambda = 1540\text{m/s}$. The sampling frequency used was $f_s = 100\text{MHz}$. The elements had a width and height of 0.25mm and 5mm respectively. The focal-point was set to 30mm.

Table: 1 and Table: 2 shows the parameters for 4-element and 16-element linear array transducers respectively, excitation pulse and medium used for this center frequency (f_0) used is 5 MHz. Figs. (a-m) shows; element impulse responses for 4-element array and figs. (a'-m') shows; element impulse responses for 16-element array transducer (i.e. TX Field image, TX Axial waveform, TX Lateral beam plot, TX/RX Field image, TX/RX Axial waveform, TX/RX Lateral beam plot, K-space TX/RX field image, K-space axial slice, K-space lateral slice, Detected image, Detected image axial slice and Detected image lateral slice).



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V. CONCLUSION

The paper attempts to present a coherent analysis of the focusing strategies for 2-D array transducer design and properties, based on linear acoustics. The delays on the individual transducer elements and their relative weight or apodization are changed continuously as a function of depth. This yields near perfect focused images for all depths. Similarly, if number of arrays in the transducer are increased then contrast of displayed image, is increased thus, benefitting the diagnostic importance of ultrasonic imaging. The detected image response for 16-element array has higher contrast than 4-element array also the pressure field generated by 16-element is high this increases diagnostic value.

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Surface charge dependent structure of ionic liquid/alkali halide interfaces investigated by atomic force microscopy

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Ionic liquid (IL)/solid interfaces are relevant to a broad range of physicochemical phenomena and technological processes such as catalysis, corrosion, electrochemistry, and lubrication. Hence, understanding the effect of substrate surface nature on the interfacial properties has a significant impact on improving technological processes in which interfacial properties are dominant. In this work, we investigated interfacial structures between 1-butyl-3-methylimidazolium hexafluorophosphate (BMI- PF_6) IL and KBr crystal surfaces by frequency modulation atomic force microscopy utilizing a quartz tuning fork sensor. KBr(100) and (111) surfaces were used as the substrates, where the (100) surface is electrically neutral, and the (111) surface is highly charged. We investigated the influence of surface charge on their surface structures and interfacial solvation structures by atomic-scale topographic imaging and frequency shift versus distance curve measurement. The behavior of IL at these two interfaces was found to be significantly different due to these different surface properties. © 2022 The Japan Society of Applied Physics

1. Introduction

A new class of ionic salts, ionic liquids (ILs), have been proven to possess considerable potential in electrochemistry, catalysis, solar cell, lubrication applications and also used as green alternative solvents to organic solvents.^{1–5} Furthermore, they exhibit several advantages over conventional solvents because they have remarkable properties such as high thermal and chemical stability, low vapor pressure, and high ionic conductivity.^{6–9} Therefore, ILs have attracted great interest in the scientific community. In general, when a liquid is in contact with a solid substrate, the properties of the liquid at the interface are different from those of the bulk liquid.¹⁰ The interfaces between the solids and ILs play a crucial role for some of the applications mentioned above. To enhance the efficiency and selectivity of a particular process, detailed knowledge of the solid/IL interface at the molecular level is required for not only a fundamental understanding point of view but also the industrial applications.

Numerous approaches, such as X-ray reflectivity, resonance shear measurements, neutron reflectometry, sum-frequency generation spectroscopy, and scanning probe microscopy, including scanning tunneling microscopy and atomic force microscopy (AFM), have been applied to study the IL/solid interfaces.^{6–11} In particular, AFM is capable of investigating the density distribution of ions composing the IL near the substrate by AFM-based force curve measurement, which revealed the existence of layered structure of ILs, so-called solvation layers.^{12–13} However, there is limited knowledge regarding the interfacial structure between ILs/solid and the effects of the nature of solid substrates on the formation of solvation layers. Thus, this subject still has a demand for discussion.

Frequency modulation (FM) AFM, where an AFM force sensor vibrates at its resonance frequency and the force acting to the sensor is detected as its resonance frequency shift (Δf), has a higher resolution imaging capability than the classical AFM mentioned above. Thus, it is gaining particular interest to achieve true atomic-resolution imaging on various

materials in different environmental conditions such as in vacuum, ambient conditions, and in liquids.^{14–15} It can also characterize the density distribution of liquid molecules on liquid/solid interfaces, and the presence of solvation layers, has been revealed.^{26–27} These studies revealed that the FM-AFM technique significantly impacts various research fields like surface science and nanotechnology. However, FM-AFM imaging of the substrates and interfaces in ILs is considered more difficult than in other types of liquid because the quality factor (Q) of the force sensors (Si cantilevers are widely used in FM-AFM) is heavily suppressed due to the high viscosity of ILs. Yokota et al. decreased the sensor noise in FM-AFM by improving the optical beam deflection system and achieved molecular-resolution imaging in IL using a Si cantilever.¹⁶ In contrast, Ichii et al. took another approach; a quartz tuning fork with a sharpened metal probe, the so-called qPlus sensor, was used as a force sensor. Just a tip apex was immersed into the IL to keep the Q factor high, and atomic/molecular-resolution imaging was successfully achieved.^{23,24}

Ichii et al. also demonstrated the atomic-resolution imaging and structural analysis of an interface between a KCl (100) surface and 1-butyl-1-methylpyrrolidinium tris(pentafluoromethyl)trifluorophosphate (Py_{14}FAP) IL.²⁵ The two-dimensional Δf mapping revealed a different interfacial structure from other IL/solid interfaces. This difference was explained from the viewpoint of surface charge, i.e. a KCl (100) surface is electrically neutral, whereas charged substrates, such as mica, were used in other reports.^{29,30}

In this work, we extended our study with this background to understand the effects of surface nature on the interfacial structure. The KBr(100) and (111) crystal surfaces were investigated in a hydrophobic IL, 1-butyl-3-methylimidazolium hexafluorophosphate (BMI- PF_6), using FM-AFM, in which the (100) surface is electrically neutral, and the (111) surface is highly charged. Both the surface structures in BMI- PF_6 IL were characterized on the atomic scale. Since (100) surfaces of alkali halide crystals can be easily obtained by cleavage, many AFM studies have been carried out on

these surfaces.^{27–32} On the other hand, their (111) surfaces are highly charged and have high surface energy, which is difficult to prepare. However, Y. Matsumoto's group has recently developed a method to prepare (111)-oriented alkali halide crystals via IL flux and found that BMI-PF₆ is suitable for this process.^{33,34} In addition, the hydrophobicity of the BMI-PF₆ IL minimizes the effect of water molecules even in atmospheric experiments, which is beneficial for AFM analysis. For these reasons, BMI-PF₆ IL was chosen in this study. There have been few examples of AFM observation of alkali halide (111) surfaces, and thus, their atomic-scale structural analysis is both challenging and crucially important in the field of surface science. Furthermore, we carried out Δf versus distance curve measurements on these interfaces and discussed the influence of surface charges on their solvation structures.

2. Experimental

We used an FM-AFM instrument based on a commercial AFM (JEOL JSPM-5200), and the original AFM head was replaced by a home-built AFM head for a qPlus sensor.³⁵ The qPlus sensor was prepared by attaching one prong of the commercially available quartz tuning fork (STATEK Co. TFW-1165) to the substrate with the help of epoxy adhesive. The probe was made from a tungsten wire with a diameter of 0.1 mm (Nilaco Co.), which was electrochemically etched in a potassium hydroxide solution (1.2 mol L⁻¹) and then glued at the other prong of the tuning fork. The resonance frequency and spring constant of the tuning fork before the probe attachment were 32.768 kHz and 1884 N m⁻¹, respectively. After attaching the tungsten probe, the resonance frequency was typically decreased to 13–16 kHz. The qPlus sensor was mechanically vibrated by a piezoelectric zirconate titanate (PZT) plate. The deflection of the qPlus sensor was detected by a transimpedance amplifier with a 100 M Ω feedback resistor. Then, the deflection signal was amplified by an inverting amplifier with a gain of ~ 10 . These electronic circuits were embedded in the AFM head. Δf of the qPlus sensor was detected by a commercial FM demodulator (Kyoto Instruments KI-2001) with some modifications. The vibrating amplitude was kept constant using an amplitude feedback system. Because the vibrating amplitude was detected using root-mean-square (RMS)-DC converter with a bandwidth of more than a few hundred kHz, a band-pass filter was inserted between the transimpedance amplifier and the RMS-DC converter for precise amplitude detection.

All the AFM experiments were performed at room temperature. The AFM topographic images were obtained as the two-dimensional tip trajectories during the tip scanning parallel to the sample surface plane (*xy* plane), where Δf was kept constant, and were processed by WSxM software.³⁶ Δf versus distance curves were obtained by changing the tip-to-sample distance without the Δf feedback at a constant *xy* position. The atomic models of KBr crystals [Figs. 1 and 2(c), 2(f)] were processed by VESTA software.³⁷

BMI-PF₆ IL (Tokyo Chemical Industry Co., LTD., >98%) was purchased and used without further purification. First, we prepared the saturated solution of KBr in BMI-PF₆ IL by adding 1 wt % KBr powder (Nacal Tesque Inc., >99%). After that, the KBr(100) crystal (Funachi Chemical Co.) was cleaved using a sharp knife, and the solution droplet (volume:

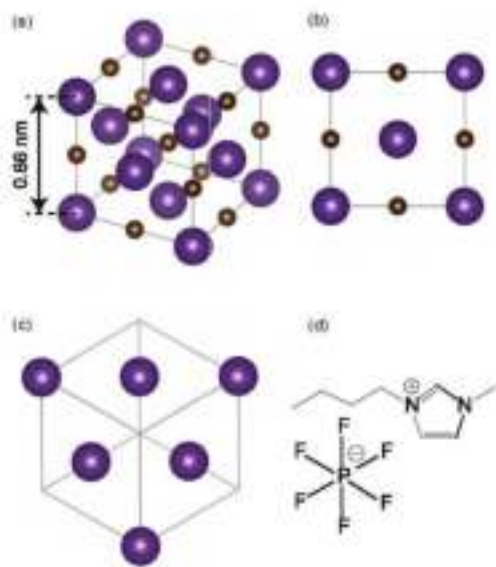


Fig. 1. (Color online) (a) Crystal structure of KBr crystal (purple: Br⁻, brown: K⁺). (b) Atomic arrangement of KBr(100) surface. (c) Atomic arrangement of KBr(111) surface assuming the exposure of Br⁻ ions and (d) molecular structure of BMI-PF₆ IL.

0.2 μ L) was immediately placed on the cleaved KBr(100) sample with a syringe. This sample containing the IL droplet was kept in a drying chamber (Daikin Industries, Ltd., dew point < -50 °C) for 1 day. In contrast, the KBr(111) sample cannot be obtained by cleavage. It was fabricated by Y. Matsumoto's group in Tohoku University using the IL-assisted vacuum deposition method.^{33,34} Then, the KBr(111) sample was transferred to H. Sagisura's group in Kyoto University by post. Note that the surface of the KBr(111) sample was covered with BMI-PF₆ IL, which protected the surface from contaminants. The ion arrangement on the (100) and (111) crystal surfaces of KBr is shown in Fig. 1. Alternate K⁺ and Br⁻ ions are present on the KBr(100) surface, while on the KBr(111) surface either K⁺ or Br⁻ ions are present. The KBr-saturated BMI-PF₆ droplet (0.5 μ L) was placed on the KBr(111) sample and left in the drying chamber for several weeks. Finally, both the samples were used for topographic imaging and Δf versus distance curve measurement with the FM-AFM instrument.

3. Results and discussion

BMI-PF₆ IL possesses a much higher viscosity (308 cP) than water (0.89 cP). We measured the *Q* factor of a qPlus sensor in air and BMI-PF₆, which were found to be 1700 and 55, respectively. Although the measured *Q* factor was heavily suppressed in the IL compared to air, it was still much higher than that of Si cantilevers in viscous ILs (~ 1).^{38,39} The low *Q* factor of the force sensor increases the frequency noise and reduces the force sensitivity.⁴⁰ In this study, only the tip apex was inserted into the IL, so that the *Q* factor was relatively kept high and high force sensitivity was achieved.²⁷ When the tip apex was sufficiently close to the

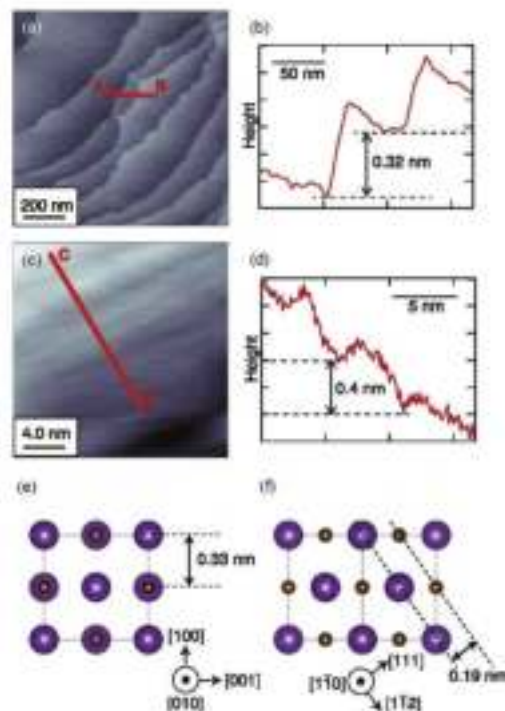


Fig. 2. (Color online) FM-AFM topographic images of (a) KBr(100) [$\Delta f = +5$ Hz, $A = 1.0$ nm] and (c) KBr(111) [$\Delta f = +5$ Hz, $A = 1.6$ nm] obtained in BMI-PP₆. (b) and (d) are the cross-sectional plots obtained along lines AB and CD, respectively. (e) and (f) are the atomic models representing inter planar distance of (100) and (111) KBr crystal surfaces, respectively.

sample surface, the resonance frequency of the qPlus sensor generally changed due to tip-to-sample interaction. The electrostatic and van der Waals forces are suppressed in conductive liquids, and hence positive Δf was usually observed. The Δf of the sensor was responsible for obtaining the topographic images in FM-AFM.

The KBr(100) is known as a natural cleavage plane because it has zero net charges. Figure 3(a) shows a topographic FM-AFM image of (100) surface obtained in BMI-PP₆, in which atomic steps were clearly seen with a step height of ~ 0.32 nm [Fig. 2(b)]. This value is in good agreement with the half of the lattice constant $a_0/2$, where $a_0 = 0.66$ nm, as shown in the atomic model of KBr(100) [Fig. 2(e)] and also consistent with the previously reported values by several researchers using AFM.^{22,28} As we used the KBr-saturated solution of the IL, the possibility of further dissolution of the KBr(100) crystal in IL was highly suppressed, and hence the stable atomic steps were imaged. In contrast, the KBr(111) surface is electrostatically unstable due to high surface energy and becomes quite difficult to prepare. Y. Matsumoto's group fabricated this sample by growing the KBr(111) microcrystal on α -sapphire(0001) substrate via IL thin film through vacuum deposition method.⁴⁴ In our study, we used this KBr(111) sample for the topographic imaging and the interfacial study. We kept the sample in a drying chamber for several weeks because the

IL/KBr(111) interface was unstable soon after placing the IL droplet. Figures 2(c) and 2(d) show the topographic image of the (111) surface and the cross-sectional plots obtained along line C-D in Fig. 2(c), respectively. A step structure with a height of ~ 0.4 nm was imaged, which was in good agreement with twice of interplanar spacing in the KBr(111) crystal surface, as shown in the atomic model Fig. 2(f). Although we cannot conclude which type of ion (K^+ or Br^-) was exposed to the surface, this result indicates that only one type of ion was exposed. From these low-resolution images of the surface topography and atomic arrangement,

Furthermore, we have carried out high-resolution imaging on these surfaces in the IL. Figure 3(a) shows an atomic-resolution topographic image of the KBr(100) surface obtained in KBr-saturated BMI-PP₆. The square lattice with a spacing of 0.46 nm was clearly seen, corresponding to the distance between the nearest equally charged ions (theoretically 0.47 nm). The previous FM-AFM studies on ionic crystals, such as NaCl, KCl, and CaF₂, carried out in UHV and aqueous solution demonstrated that either cations or anions are visible in most cases.^{28–32,41} Our previous study on KCl(100) in Py_{1,4}-FAP IL and KBr(100) in BMI-PP₆ showed the same result.^{28,42} Therefore, the bright spots in the atomic-resolution images could be assigned to K^+ or Br^- ions. Figure 3(b) shows a high-resolution topographic image of the (111) surface of KBr. As shown in Fig. 1(c), on KBr(111), either K^+ or Br^- ions form a triangular lattice with a lattice constant of 0.47 nm. However, the obtained atomic distance was approximately 0.4 nm, revealing the high distortion of atomic arrangement. Figure 3(c) shows another topographic image obtained on a different area on the KBr(111) in BMI-PP₆ IL. It also shows the atomic-scale contrast. The lattice was highly distorted, and the distance between the

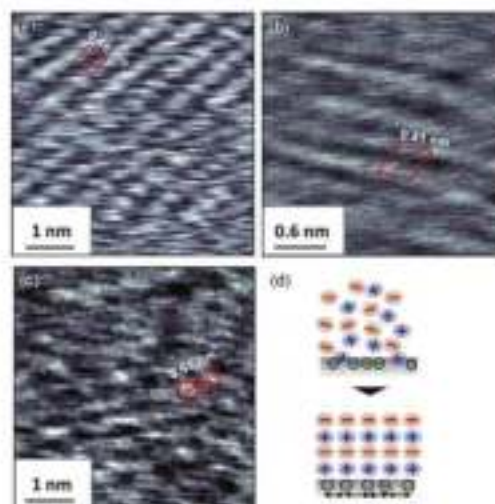


Fig. 3. (Color online) High-resolution AFM topographic images of (a) KBr(100) [$\Delta f = +11$ Hz, $A = 0.8$ nm], (b), (c) KBr(111) [$\Delta f = +5$ Hz, $A = 1.6$ nm] for (b) and $\Delta f = +104$ Hz, $A = 1.6$ nm for (c), obtained in BMI-PP₆. (d) Schematic illustration for explaining the distortion of atomic arrangement of KBr(111) surface in BMI-PP₆.

nearest equally charged ions was ~ 0.51 nm, obviously not matching the theoretical value (0.47 nm). The (111) surface consists of alternate anions and cations layers, and hence this surface possesses high surface energy, resulting in a distorted surface by surface rearrangement or reconstruction as schematically shown in Fig. 3(d). This may be one of the reasons why the distance between the closest bright spots did not match the theoretical value. It should be noted that from this experimental result, we cannot deny the possibility that the counterions derived from the IL strongly adsorbed on the KBr(111) were imaged, rather than the outermost ions of the KBr(111) surface. However, even if this were the case, it would be due to the high surface charge density of the KBr(111) surface. Thus, it is reasonable to conclude that the difference in the interfacial structure owing to the difference in the physical properties of the (100) and (111) surfaces was imaged on the atomic or molecular scale. At such interfaces where the surface atom reconstruction occurs, the interfacial solvation structure is also likely to be strongly influenced by the surface. Therefore, we analyzed the behavior of the BMI-PP₆ IL at the IL/KBr(100) and IL/KBr(111) interfaces by Δf versus tip-to-sample distance curve measurement.

Figures 4(a) and 4(b) present the Δf versus tip-to-sample distance curves which were obtained at the BMI-PP₆/KBr(100) and BMI-PP₆/KBr(111) interfaces, respectively. At the longer tip-sample distance, no Δf was observed on both the interfaces. On the BMI-PP₆/KBr(100) interface, Δf was positively and monotonically increased without oscillatory profile as the tip approached the surface. In contrast, on the BMI-PP₆/KBr(111) interface, an oscillatory Δf profile was observed. The period of oscillation was 0.65–0.73 nm, which agreed well with the theoretical ion pair diameter of BMI-PP₆ (~ 0.7 nm). The previous AFM studies

also reported that the detected thickness of the layered structure at solid/IL interface matched well with the theoretical ion-pair diameter of the ILs.^{11,13–15,26} This is often explained by a model in which the cation and anion layers of the IL are alternately distributed at the interface. Furthermore, the theoretical study on AFM force curves, including Δf versus distance curves in FM-AFM, also supports this model.⁴³ Thus, our results indicate that the layered distribution of the ions was present only on the BMI-PP₆/KBr(111) interface. The difference in Δf profiles between BMI-PP₆/KBr(100) and BMI-PP₆/KBr(111) interfaces would be due to the different surface charges. The (100) surface is a natural cleavage plane with zero net charges since the number of cations and anions present on the surface is equal. In contrast, the (111) surface of KBr possesses either cations or anions, which leads to a highly charged surface. This would be one of the reasons for the layered solvation structure formation at this interface. When obtaining topographic images at an interface Δf distance curve oscillates, the probe may scan on the solvation layer instead of the outermost surface of the sample because there exist multiple z -positions corresponding to a certain frequency shift. In other words, the KBr(111) surface may have been scanned in Fig. 3(c), which was imaged with the larger Δf , and the solvation layer may have been scanned in Fig. 3(b), which was imaged with the smaller Δf . However, we cannot conclude this at this stage because Figs. 3(b), 3(c), and 4(b) were all obtained with different probes. Topographic imaging at different Δf and the Δf versus distance curve measurements with the same probe and the same amplitude would be a promising way to evaluate the imaging position.

We previously reported two-dimensional Δf mapping on an interface between KCl(100) and Py₁₄-FAP IL, which were composed of 128 Δf versus distance curves.²⁵ In that experiment, some of them showed a monotonical increase of Δf as the tip approached the KCl(100) surface, which was similar to the BMI-PP₆/KBr(100) interfacial study (this study). It should be noted that some of Δf profiles on the Py₁₄-FAP/KCl(100) interfaces showed saw-tooth like oscillations with a period of ~ 0.3 nm, which agreed well to the height of the atomic step of KCl(100) surface, and obviously smaller than the ion-pair diameter of Py₁₄-FAP IL. These oscillations were explained by spontaneous and/or tip-induced dissolution of the KCl(100) surface and were considered unrelated to the interfacial solvation structures. Thus, the results on Py₁₄-FAP/KCl(100) and BMI-PP₆/KBr(100) interfaces both revealed the absence of a layered and alternating cation-anion density distribution at IL/alkali halide(100) interfaces. Recently, FM-AFM studies on water/alkali halide(100) crystal surfaces were reported. Interestingly, the presence of hydration layers at the interfaces was recognized.^{31,32} Moreover, the molecular dynamic simulation study also showed the presence of hydration structure at the interface.³³ Our experimental results in this study revealed that the mechanism of IL solvation layer formation is completely different from that of water hydration layers on alkali halide(100) surfaces, suggesting that surface charge is the critical factor for the formation of IL solvation layers.

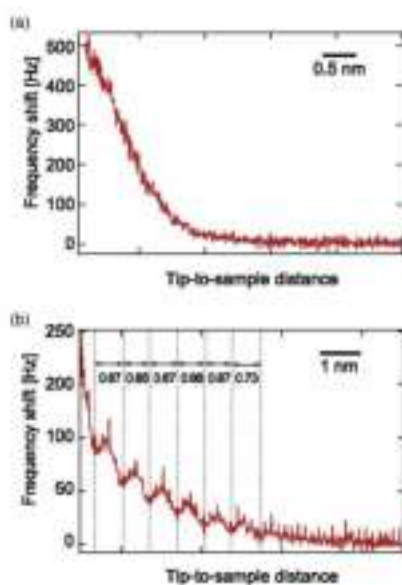


Fig. 4. (Color online) Δf versus tip-to-sample distance curves obtained on (a) a KBr(100)/BMI-PP₆ interface ($\lambda = 0.7$ nm) and (b) a KBr(111)/BMI-PP₆ interface ($\lambda = 0.7$ nm).

4. Conclusion

In conclusion, we performed atomic-scale topographic imaging on (100) and (111) surfaces of ionic crystal KBr in viscous BMI- PF_6 IL by FM-AFM utilizing a qPlus sensor. The (100) surface showed a periodic square lattice corresponding well to its atomic arrangement model, whereas the (111) surface showed a strongly distorted atomic-scale contrast. This difference would be attributed to the higher surface charge density and higher surface energy of the (111) surface than the (100) surface. Also, the BMI- PF_6 IL/KBr interfaces for both the surfaces were studied by Δf versus distance curve measurement. The oscillatory Δf profile was only observed on the IL/(111) interface, while the monotonical increase of Δf was observed on the IL/(100) interface. That is, the layered density distribution of the ions was only found on the IL/(111) interface, which was also attributed to the high surface charge density of the (111) surface. To the best of our knowledge, this is the first report on atomic-scale imaging on a polar (111) surface of KBr in BMI- PF_6 IL and on surface charge effects on the solvation layers at the interface. The selection of particular solids and liquids and the understanding of their interfacial properties are essential for improving technological processes such as catalysis, lubrication, and electrochemistry. Therefore, we consider the interpretation of the influence of substrate surface nature on interfacial properties to be of great importance, and further knowledge is urgently needed for academic and development of technological processes.

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