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Synthesis, Characterization and Application of $AlTiZrO_4$ Nanomaterial

M. V. Patil[1], S. R. Bamane[2], S. M. Khetre[3]

Department of Chemistry, Miraj Mahavidyalaya, Miraj, India[1]

Sushila Shankarrao Gadave Mahavidyalaya, Khandala, Satara, India[2]

Department of Chemistry, Dahiwadi College, Dahiwadi, Satara, India[3]

Correspondence Author: pramanikpatil@rediffmail.com[1]

Abstract: *Oval shaped **$AlTiZrO_4$** nanoparticles were synthesized by wet chemical co precipitation and muffle ignition method. The oval shapes of nanomaterial were confirmed using SEM imaging and spinal packing in crystals were determined on the basis of XRD spectrum. The surface functionalities over nanomaterial was confirmed using FTIR spectrum elucidating hydroxyl and oxide groups over surface for future water wet ability. Furthermore the porous nature and electronic states in nanomaterial were elaborated on the basis of UV-Vis. And PL spectral transitions along with matching SEM and XRD data. The very high porosity of this ceramic nanomaterial was confirmed by BET measurements and future water remediation applications were demonstrated using antimicrobial testing on Proteus Vulgaris and membrane water purification activity. Overall this novel ceramic porous nano material has proved probable application in water purification membranes.*

Keywords: Oval ceramic, Nano material, Highly Porous, Water remediation, Absorbance

I. Introduction

Nowadays, nanotechnology is the most advanced technique in the field of science and technology. The great amount of research has been made to identify for utilization in organic and inorganic oxides content for ceramic membrane preparation. The basic matrix is prepared by adaptation of some volatile and carboneous matter with single or multiple, inorganic components to create porosity while sintering process.[1-4]

Titanium and zirconium oxides are very advanced material for future technology of thin layers of ceramic membrane because of their mechanical, thermal, and chemical properties. Titanium oxide (TiO_2) is a cheap, non-toxic and non-biodegradable material. Hence it is widely used in various industries. It acts as semi-conducting material under the form of thin films. It shows insensitivity to visible light to its band gap 3.14 eV, while it absorbs light near ultra-violet region. Due to its low efficiency, it can be sensitized by great number of dyes. Thus, various applications are available for titanium oxide. Mainly titanium (TiO_2) is used as thin films in solar cells, photo catalyst cells and electro chromic system; mainly it is used in preparation of optics. In addition, TiO_2 films are important optic films due to their high reflective index and transparency.[5-9]

Zirconium oxide (ZrO_2) has good dielectric and optical property. It has high refractive index (43). Also it has good transparency, great chemical stability. All these properties lead to miscellaneous applications in optical filter, laser mirror. Zirconium oxides are also applicable in super conducting ceramics like biomaterial gas sensors batteries. The band gap between metal oxide has wide range and it is in between range of 5.0 – 5.85 eV. To obtain nano materials with controlled properties it is mainly used in dyes, semi- conductors, and metal particles.

Thus, this ($AlTiZrO_4$) newly synthesized trio metal nano composite material has suitable application for antimicrobial and water purification. $AlTiZrO_4$ nano particles were synthesized by wet chemical co precipitation and muffle ignition method. The disc shaped nano material was confirmed by SEM analysis and spinal packing crystal lattice was determined by XRD method. The surface function was confirmed by using FTIR spectrum. The porous nature and electronic states in nano material were determined by UV-Visible spectroscopy, while PL emission spectra are used for spectral transition of oxide group and hydroxyl group. The porosity of ceramic nano material was confirmed by BET technique. Also, antimicrobial testing on Proteus Vulgaris microorganism.[10-15]

The ceramic nano composite material were demonstrated by using antimicrobial testing on Proteus Vulgaris. Proteus Vulgaris is a rod- shaped, nitrate-reducing indole-positive and catalane positive hydrogen sulfide-producing, Gram-negative bacterium that inhabits the intestinal tracts of humans and animals. It can be found in soil, water, and fecal matter. It is grouped with the Morganellaceae and is an opportunistic pathogen of humans. It is known to cause wound infections and other species of its genera are known to cause urinary tract infections.

This ceramic nano composite material shows water remediation activity. Therefore it becomes essential to improve water contamination treatment technology. This newly synthesized nano composite ceramic material creates zone of inhibition for Proteus Vulgaris indicates that this ceramic material kills such type of water born microorganism. Thus, it is applicable in water purification treatment.[16-18]

II. Experimental Section

2.1 Materials and Cell Cultures

All the chemicals used for synthesis nanocomposites and their *in vitro* biological screening such as aluminum nitrate, titanium chloride, calcium nitrate, Conc. HCl, ethanol were of A. R. grade. These chemicals were purchased from S. D. fine chem. Ltd. and Merck ltd and were used without further purification. The cell culture medium such as agar growth broth and bacterial culture, fetal bovine serum, trypsin buffer were obtained from Hi-media ltd. The double distilled water was obtained from millipore system and used throughout the synthesis and *in vitro* biological screening tests.

2.2 Synthesis of Oval Ceramic Nanoparticles (Co-Precipitation Method)

All the metal salts are mixed in 0.01M proportion in 25 ml. double distilled water and traces of HCl were added to the flask. The flask contents are vortexes on magnetic stirrer at 600 rpm. For 6 hours then visible color change was observed after formation of precipitate. The precipitate was washed with double distilled water and dried in oven at $92^{O}C$. The dried trio metal oxide nano composite ceramic powder was then crushed. This powder was characterized and used for antimicrobial studies for to use in water remediation.

2.3 Structural and Morphological Characterization of Oval Nanomaterial:

As mentioned in table 1.1, the structure, morphology, particle diameter range and types of bonding of functionalities in the ceramic nanocomposite was determined based on physicochemical characterization using UV-Vis., PL, FTIR, XRD spectrometry techniques and SEM microscopic analysis. The Spectronics double beam UV-Vis. spectrometer with water as blank was used to determine absorption spectrum of material. To confirm functionalities present in material and comparing with pallet form, Perkin Elmer series FTIR spectrometer was used with KBr pellet technique. The PL emission spectrum of nanocomposite was determined using Jasco type spectro fluorometer with excitation identity of material with same 25 ppm. concentrations. The X-ray diffraction pattern of material was determined using X-ray spectrometer by powder diffraction technique for to elaborate the packing f ions, hybridization and crystal system of nanocomposite entities. The composition of material, formation and phase of ceramic nanocomposite is proved here by this spectrometric analysis.

2.4 Antimicrobial Screening on Gram Positive Bacteria by Agar Well Disc Diffusion Method

The cell-particle interactions of materials demonstrating their reactivity and biocompatibility can be elaborated using simple *in vitro* antibacterial screening in buffer solutions to maintain physiological mimicking pH at material cell interactions. As cell pH affect on the biocompatibility of molecules. Here in this work 20 ppm. Concentrations of material were dosed on bacterial cell cultures grown in agar broth on discs, inside the wells bored on plates. The gram negative bacteria *Proteus Vulgaris* was grown on culture plates and inhibited by dosing of material solutions in buffer dispersions with physiological pH = 7.7 by use of phosphate buffer. The culture plates were incubated and zones of inhibition were measured, and biocompatibility/ antimicrobial property of nanocomposite was elaborated.

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III. Results and Discussion

3.1. Morphological and Structural Characterization of Ceramic Nanomaterial

A. UV-Vis. Absorption and PL Emission Spectrum: Figure 1.1 and 1.2

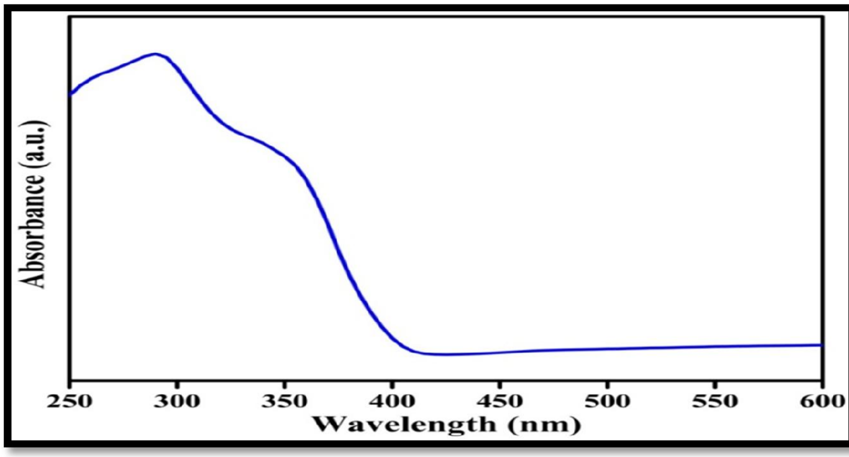

Figure 1.1: UV-Visible absorption spectrum of $AlTiZrO_4$ ceramic nanomaterial

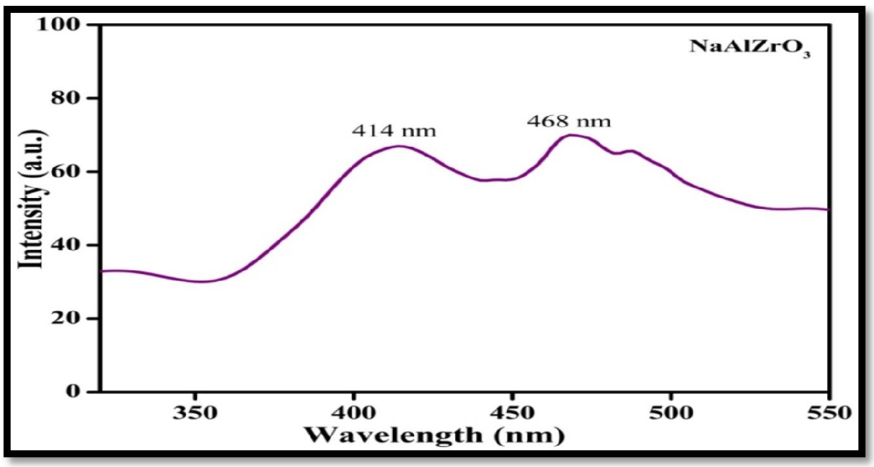

Figure 1.2: PL emmission spectrum of disc Nanomaterial

B. FTIR Spectrum of Ceramic Nanomaterial for Surface Functionalities

As mentioned in table 1.2, and figure 1.3, FTIR spectrum of nanocomposite were determined to estimate the functionalities present inside the materials and to confirm the formation of material on the basis of signals.

Table 1.2: Matching of FTIR signals elaborating the functionalities of Schiff base and CORM complex. Figure 1.3 as above mentioned.

Signal in FTIR spectrum	Functionality	Ceramic group functionality
1040 cm^{-1}	Zr-Al linkage	Metal-oxide bonds
1311 cm^{-1}	Al-O linkage	Presence of linked oxide metal species
1551 cm^{-1}	Ti-Al group	Metal (I) and Metal (III) interaction
1632 and 1711 cm^{-1}	Presence of hydroxide	Surface groups of oxide nanomaterial
2361 and 2794 cm^{-1}	Presence of –OH and Oxides	Presence of surface moisture for porosity

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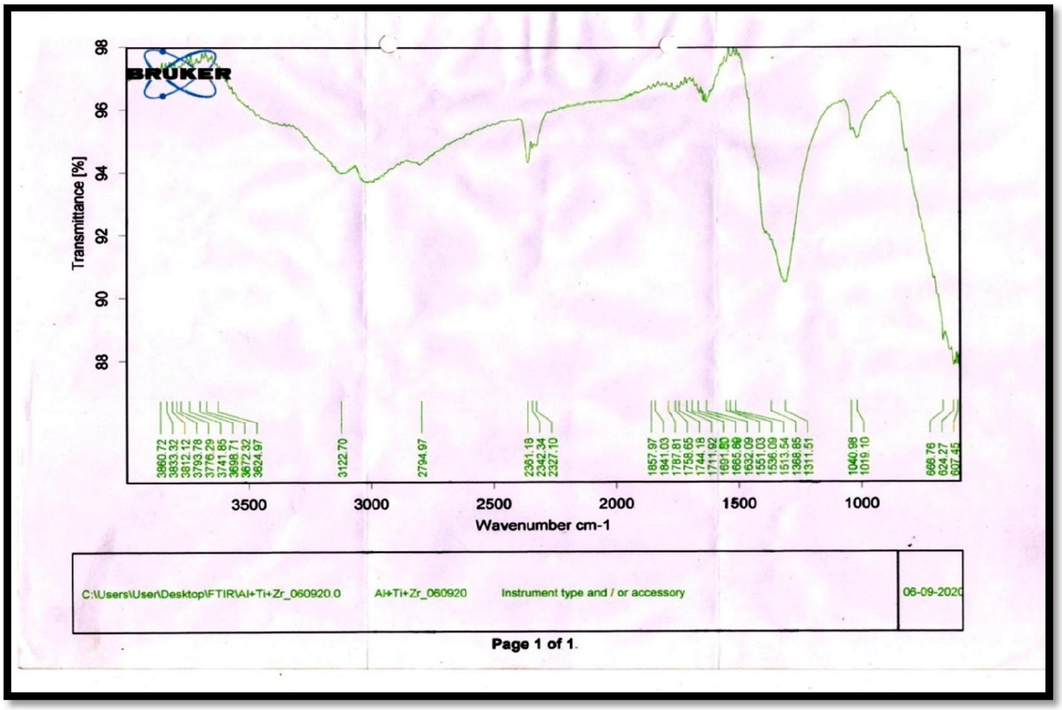

Figure 1.3: FTIR spectrum of $AlTiZrO_4$ ceramic nanomaterial for surface functionalities

C. XRD (X ray diffraction) pattern of trio metal nanomaterial :

Table 1.3: Crystal parameters of Ceramic nanocomposite with octahedral packing matched with known ceramic JCPDS card.

Crystallite planes (Miller Indices) (h,k,l) Main peaks of ceramic nano trio metal oxide	d Calculated A^0 $d = a/\sqrt{(h^2+k^2+l^2)}$ or $2d\sin\theta = n\lambda$	d Standard A^0 JCPDS card no.- 87-7799 for AiTiZrO4 matched	Lattice Constant a and b A^0 from main XRD peaks [242] at theta = 37.5 of disc ceramic nanomaterial
411	3.479	3.577	a =b=c 2.7652 A* Standard
666	2.719	2.644	
431	2.351	1.965	
544	1.876	1.654	a =b=c 2.7450 A* Calculated
633	1.627	1.602	

As per XRD data in figure 1.4 & table 1.3 it had been proved that the ceramic nanocomposite has octahedral packing of ions and cubic phase purity elaborating the 65 nm sizes from main peak using Scherer's equation.

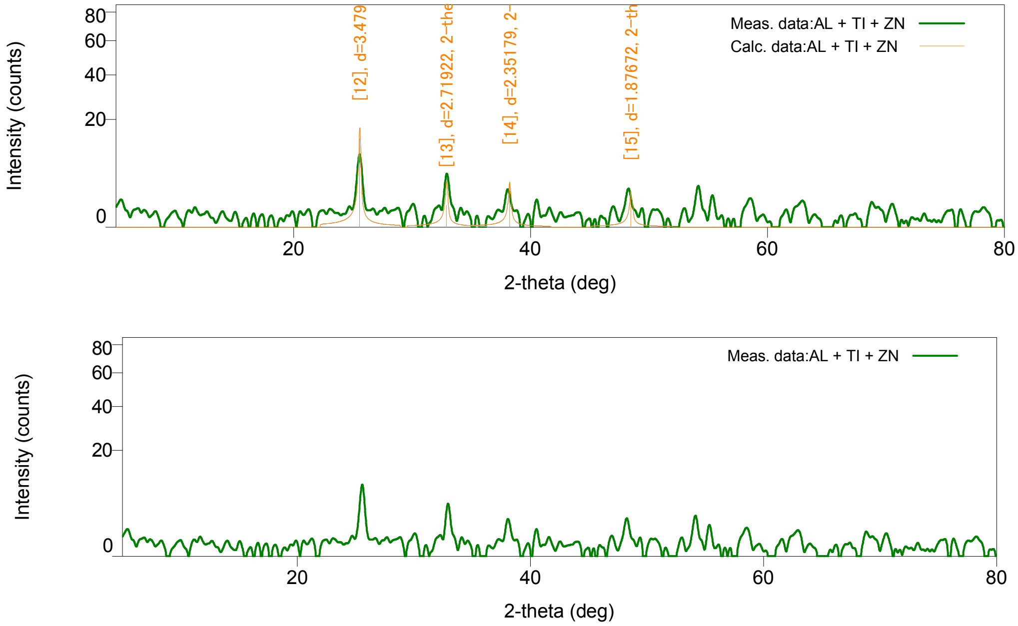

Figure 1.4 : XRD pattern of the disc $AlTiZrO_4$ ceramic nanomaterial

D. SEM Image for Disc Morphology

The SEM image of nanocomposite ceramic is as shown in figure 1.5. It throws light on oval shapes of material with some aggregation of particles. This trio metal oxide ceramic composite posses not only porosity but also exhibit vval shapes for better water loving nature and cell particle interactions.

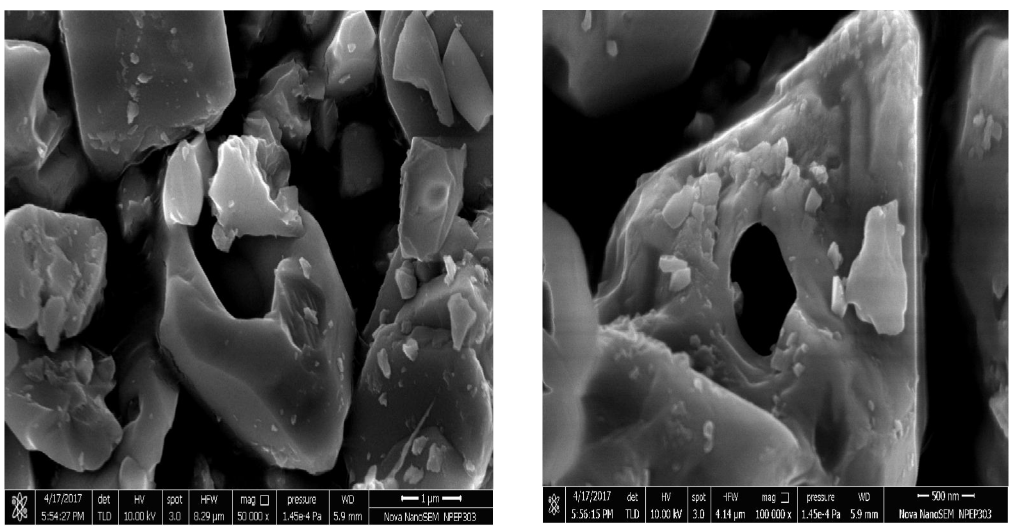

Figure 1.5 : SEM image of $AlTiZrO_4$ nanomaterial with oval morphology

E. BET Isotherm Elaborating Porosity of Disc Nanomaterial

As per figure 1.6, it is oberved that the ceramic nanocomposite show multi layer BET isotherm for nitrogen adsorpion hence it have good surface posrosity. The BET plots proves presence of porosity on surface of this nanocomposite ceramic material.

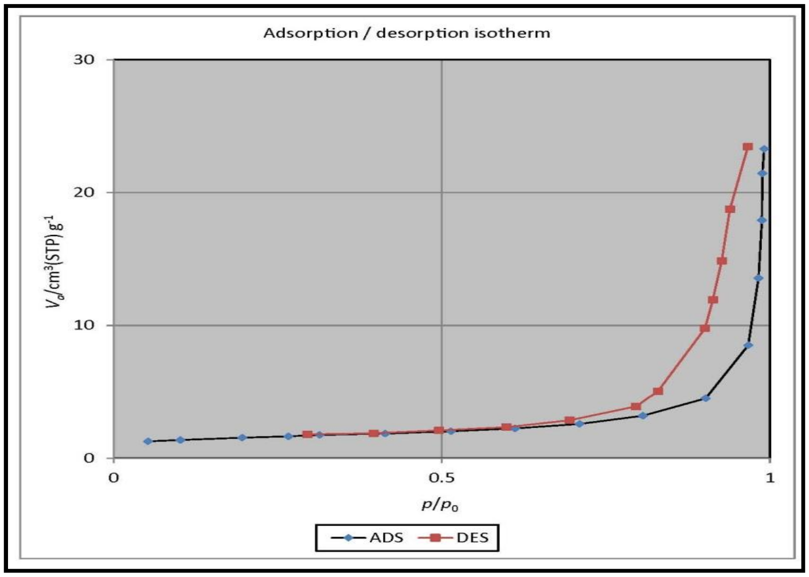

Figure 1.6: BET adsorption isotherm of $AlTiZrO_4$ nanomaterial elaborating surface porosity

IV. ANTIMICROBIAL PROPERTIES FOR WATER REMEDIATION FROM NANOMATERIAL

As per figure 1.7 and table 1.4, for antimicrobial activity of 20 ppm. material on Proteus Vulgaris. It had been demonstrated that good zone of inhibition with better antimicrobial activity.

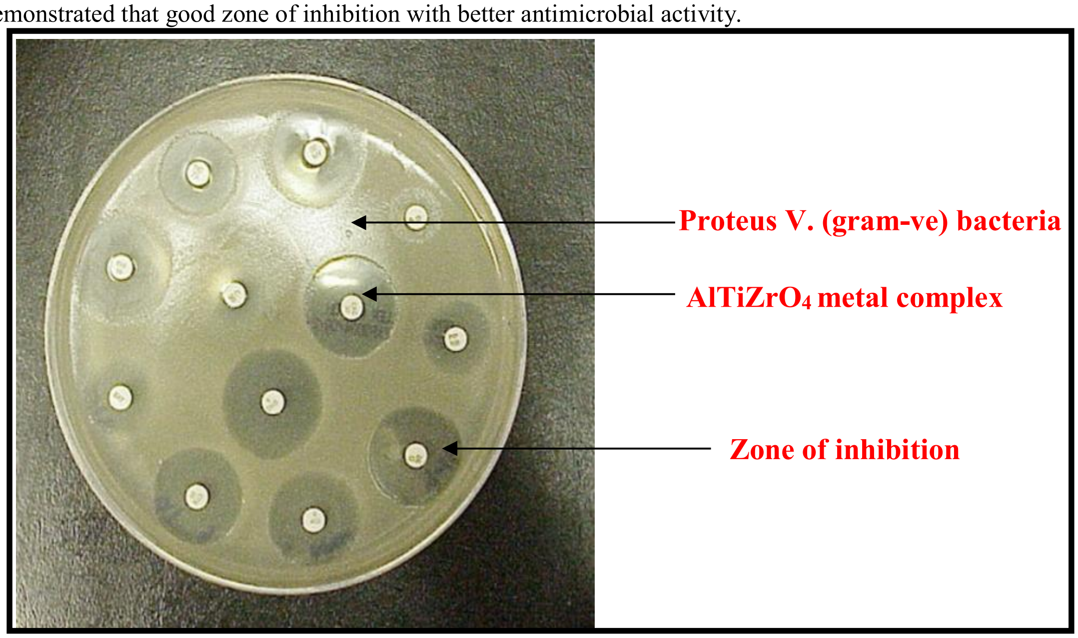

Figure 1.7: Anti microbial effects of $AlTiZrO_4$ ceramic nanomaterial on Proteus V. for zone of inhibition

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Table 1.4: Anti microbial activities of Schiff base and complex compared for gram positive and gram negative bacteria.

Type/ name of bacterial culture in Agar broth [as per figures]	Zones of inhibition for gram negative bacteria as zone diameter in mm. for Concentrations of drug/ dose of ceramic nanomaterial	
	At 10 ppm.	At 25 ppm. Fig. 8.7
Proteus Vulgaris *(gram -ve)*	13 mm.	25 mm.

V. Mechanism for Antimicrobial Activity and Water Remediation Activity

As per physicochemical and antimicrobial screening of material and elaboration in **scheme 1**, the disc nanomaterial trio metal oxide ceramic nanocomposite exhibit antimicrobial and water remediation potential at surface by material cell interactions. Here as material have surface porosity after reaction with cell membrane material and water the surface of material show adhesion to liquid and biomaterials which result in dissociation of nanocomposite to oxides on surface so result to production of peroxide on surface. This peroxide produced at surface of nanomaterial further can produce oxide and super oxide radicals to give antimicrobial effects for water remediation activity.

VI. Water Remediation Activity of AlTiZrO4

λ Max is 670 nm. At this λ max, methylene blue dye with concentration 20 ppm has been used. This concentration is prepared as 150mg / 100ml. It is photocatalyst amount. Sample is observed in total 180min. of an interval of 30min. PH of solution is maintained 7.7 and source of light is 365nm Hg Vapor lamp.

After 180min. it has been observed that the degradation of methylene blue dye rate is 82% indicates that this metal complex is very effective for water remedial activity.

Scheme 1

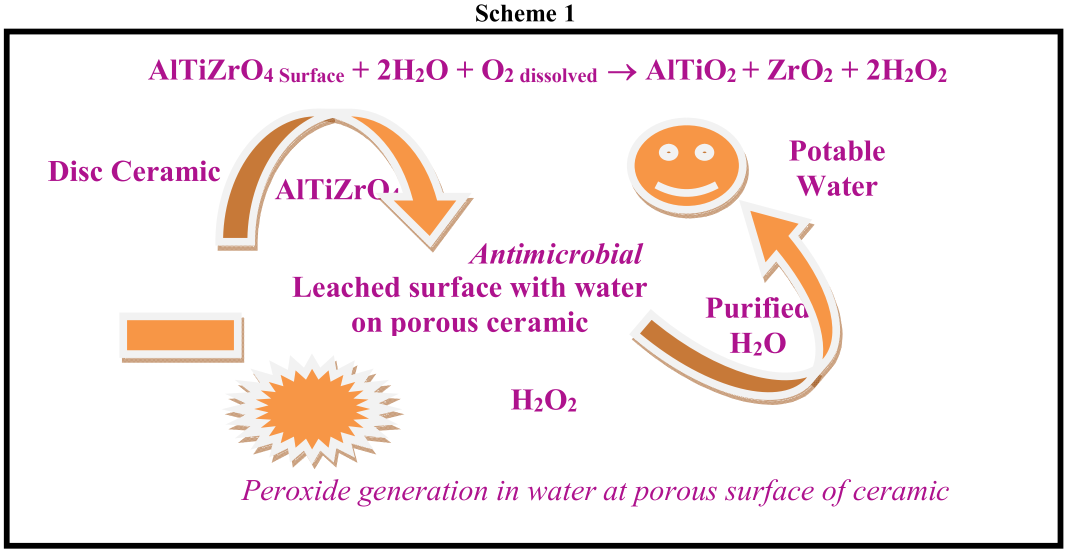

Table 1.5: Degradation Parameters

	Dye	Methylene blue dye
2.	Concentration	20 ppm
3.	Photocatalyst's amount	150mg/100mL
4.	Degradation Time	180 min
5.	Degradation Efficiency	82%
6.	pH	**7.7**
6.	Source of light	365 nm Hg Vapor lamp

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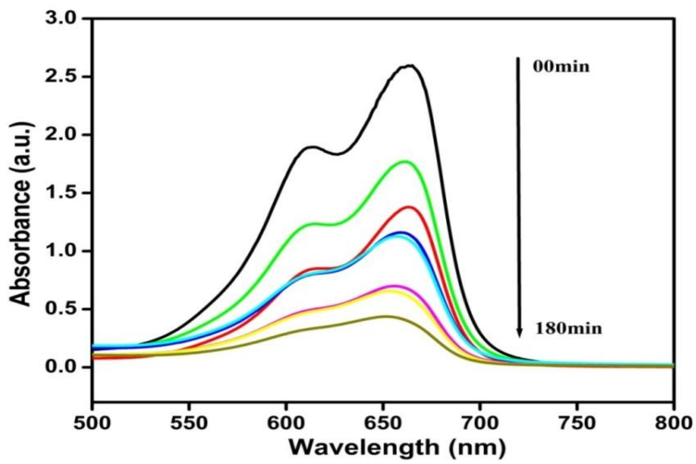
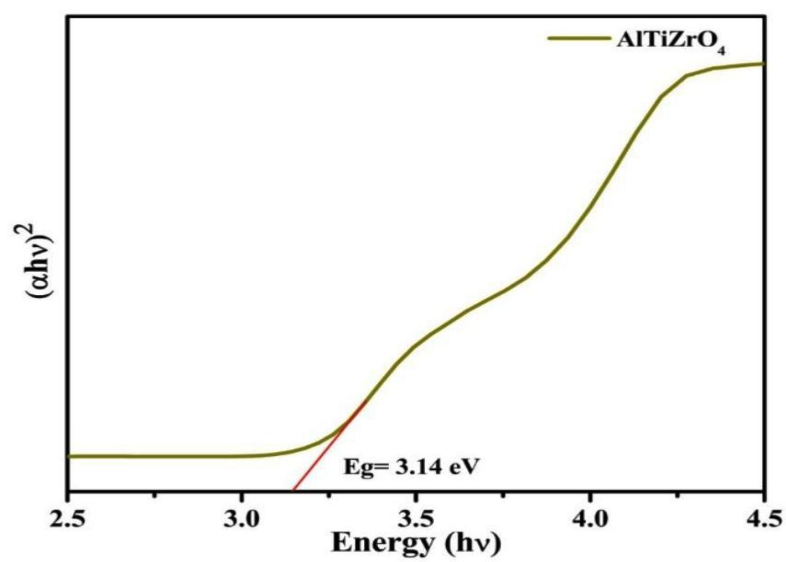
Table 1.6: Percentage degradation during course of time

Time	% Degradation of MB
Blank	00
Adsorption	32
30 min	47
60 min	55
90 min	58
120 min	70
150 min	76
180 min	**82**





In testing of water remedial activity of our ceramic composite nanomaterial $AlTiZrO_4$ with methylene blue dye, the maximum bandgap is 3.14 ev and it has been observed that maximum absortion is at 650nm at 180 min. indicates that colour of Mithylene blue dye disappears. Our nano composite material $AlTiZrO_4$ shows good water remedial activity

VII. Conclusion

A new oval shaped trio metal oxide based ceramic nanomaterial was prepared using simple wet chemical and drying route. This nanomaterial with 55 nm. mean size had exhibited surface porosity on the basis of BET isotherm N_2 adsorption. The absorption and emission spectra of nanomaterial had proved presence oxide free electrons on surface. The nanomaterial posses surface oxide and hydroxide species for water loving nature of material on the basis of FTIR analysis. Hence these evidences for nanomaterial had elaborated its properties for antimicrobial water remediation potential. On the basis of antimicrobial testing of the material it has been determined that this oval ceramic trio metal oxide nanomaterial finds applications in water purification and environmental fields.

References

[1]. Basheer A.A., Journal of Molecular Liquids, Nethelands**, (2018**), V.216, pp.583-593.
[2]. Burggraaf, A. J., Keizer, K., Synthesis of Inorganic Membrane 35; **(2009),** 38-38.
[3]. Buxton, G. V.; Greenstock, C. L.; Helman, W. P.; Ross, A. B. *J. Phys. Chem. Ref. Data (***1988)**, *17*, 513.
[4]. Connes, J. and Connes, P., Near Infrared Planetary spectra by Fourier Spectroscopy. I. Instruments and results**.(1966)** 56(7) : 896-910.
[5]. Egerton, R. F., Physical Principles of Election Microscopy : An Introduction to TEM, SEM and AEM. Springer**,(2005)** 202.
[6]. Giamello, E.; Calosso, L.; Fubini, B.; Geobaldo, F. *J. Phys. Chem*. (**1993)**, *97*, 5735.
[7]. Ghormely, J,A.; Stewart, A.C. *J.Am. Chem*. Soc.**(1956)**, 78,2934.
[8]. Haines, R. I.; McCracken, D. R.; Rasewych, J. B. In *Water Chemistry of Nuclear Reactor Systems 5*; British Nuclear Energy Society: London, (**1989**),p 309.
[9]. Khali A., Gondal M.A.,Dastageer M.A., Catalysis Communication**,(2009**),11(3),pp.214-219.
[10]. Li, W., Zhou, L.; Xing, W., Xu, N. Desalin. Water Treat. **(2010),** 18, 239-244.
[11]. Marcells A., Omole, Issac K., Owino, Sadik, (**2009**), pp.233-247.
[12]. Misra, P., Dubinski, M. eds., Ultraviolet Spectroscopy and UV Lasers. New York : Marcel Dekker (**2002**) ISBN 978-0-8267-0668-5.
[13]. Savage, N., Diallo M., Nanomaterials and Water Purification: Opportunities and Challenges. Journal of Nanoparticle Research**,(2005**) 7, pp.331-342.
[14]. Prabhu, V., Patwardhan, A. & Patwardhan, A.W., CIJCT). **(2016)** Vol.24, PP - 367-373.
[15]. Reimer, L., Scanning Electron Microscopy: Physics of Image formation and Microanalysis. (Springer), (**1998**), 527.P.
[16]. The Infracord double-beam spectrophotometer. Clinical Science. (**1957**) 16 (2).
[17]. Qu.X, Alvarez P.J.J., Li,Q.,. Water research, England, **(2013),**v.47,n.12,pp.3931-3946.
[18]. Zhi-ChuanW., Yong Z., Zhangb L., Fongb H., Applied Surface Science, (**2010)**,257(3),pp.1092-1097.