CATISIO$_5$ CERAMIC NANO OVALS: SYNTHESIS, PHYSICOCHEMICAL CHARACTERIZATION AND ANTIMICROBIAL ACTIVITY WITH WATER REMEDIATION APPLICATION

***M. V. Patil, **S. R. Bamane, #S. M. Khetre**

**Department of Chemistry, Miraj Mahavidyalaya,Miraj,416410 India,*
***Sushila Shankarrao Gadave Mahavidyalaya,Khandala,Satara,416416 India,*
#Department of Chemistry Dahiwadi College, Dahiwadi,Satara-416416, India.

ABSTRACT

*A new oval shaped **$CaTiSiO_5$** Nano particles were synthesized by wet chemical Co-precipitation and muffle ignition method. The oval shapes of Nano material were confirmed using SEM imaging and spinal packing in crystals were determined on the basis of XRD spectrum. The surface functionalities over Nano material 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 Nano material 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 Nano material was confirmed by BET measurements and future water remediation applications were demonstrated using antimicrobial testing on Klebsiella 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*

INTRODUCTION :

The most emerging branch of science is nano science and nanotechnology in which metallic nano particles plays an important role especially in the field of physics, chemistry, medicine pharmaceuticals etc. Metallic nano particles have great potential in its applications. Hence it is used in different spectroscopic and microscopic characterization methods. Metallic nano-particles have applications in drug, protein, tissue engineering, enzymology biosensors, different diagnostic devices, and production of ceramic membrane. With this idea in the field of ceramic nano- technology, a new class of trio-metal oxide ceramic nano composite material has been developed **$CaTiSiO_5$**. This material has good anti-microbial and water remediation property. In this nano composite material calcium and titanium based material are used along with silicate oxide. Then it can form stable mix metal oxide results it into trio-metal nano composite material with good porosity[1,2,3].

Titanium is a common mineral. Titanium also have industrial applications, because Titanium based ceramics are good waste for nuclear waste. In the production of nano composite material of mixed oxide contain mixture of calcium carbonate, Titania and Silica. This novel shape ceramic nano composite material has been confirmed with its applications in water purification membranes. Hydrogen peroxide is mainly used for production of ceramic nano composite material. Here H_2O_2 generates peroxide entities with porous surface in water. $CaTiSiO_5$ nano- particles were synthesized by WET chemical co-precipitation method and Muffle ignition method. The oval shaped nano- material was confirmed by SEM analysis and spinal packing crystal lattice was determined by XRD method. The surface functions was confirmed by using FTIR spectrum, in which hydroxyl group and oxide group over the surface was used to determined wettablity. The porous nature and electronic states in nano-material were determined by using UV-Viz spectroscopy method. While PL absorbance is used for spectral transitition of hydroxyl and oxide group. The good quality of porosity of ceramic nano-material was confirmed by BET measurements. Also anti-microbial activity testing on Klebsiella proteus and water remediation property with its porous nature [4,5,9].

This ceramic nano- composite material shows water remediation activity 94%. Therefore it becomes essential to improve water contamination treatment technology. Photo catalysis is one of the best methods to obtain potable water. The term photo catalysis is a chemical reaction caused by photo absorption behavior of materials commonly known as photo catalysis. Water molecules which absorb over the surface of photo catalyst are used to participate in an electron transfer reaction which breaks down the molecules absorbed along with water over the surface of catalyst. Thus this mixed nano- composite material is best for water remediation property[9].

This ceramic nano- composite material were demonstrated by using anti-microbial testing on Klebsiella and Serratia. Klebsiella proteus is a genes of Gram- negative proteobacteria. Proteus bacilli are widely distributed in nature as saprophytes, being found in decomposing animal matter, sewage, manure soil, the mammalian intestine, and human and animal feces. They are opportunistic pathogens, commonly responsible for urinary and septic infections, often nosocomial. This newly synthesized nano composite ceramic material creates zone of inhibition for Klebsiella indicates that this ceramic material kills such type of water born microorganism. Thus, it is applicable in water purification process.

EXPERIMENTAL SECTION:

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.

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 are 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 and bonded with gluteraldehyde binder to form pallet. This powder and pallet was characterized and used for antimicrobial studies for to use in water remediation.

Structural and morphological characterization of oval nanomaterial: As mentioned in Table 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 [6,7,12,15]. The PL emission spectrum of nano composite 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 nano composite entities.

The composition of material, formation and phase of ceramic nano composite was proved here by this spectrometric analysis.

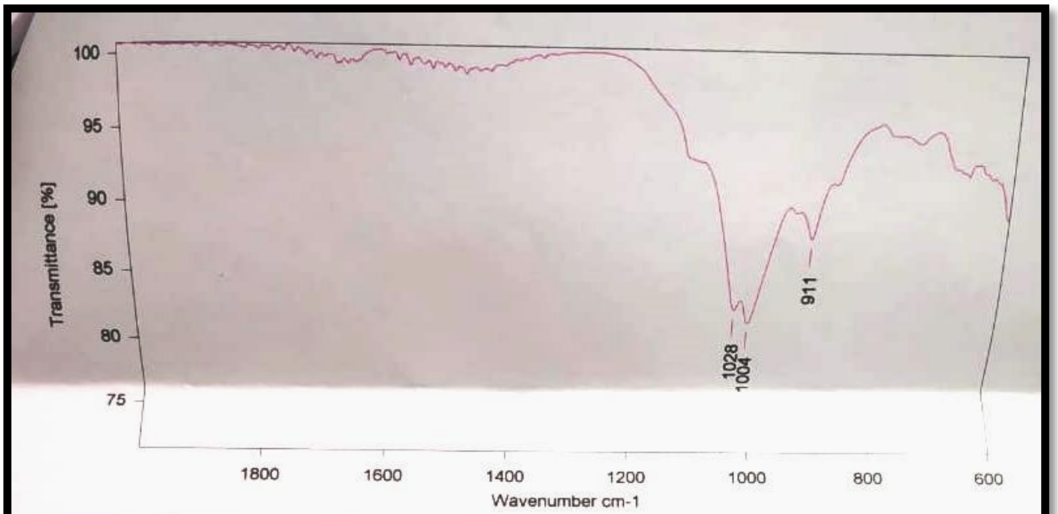
Antimicrobial screening on gram positive bacteria by agar well disc diffusion method:

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 Klebsiella gram negative bacteria was grown on culture plates and inhibited by dosing of material solutions in buffer dispersions with physiological pH = 7.4 by use of phosphate buffer. The culture plates were incubated and zones of inhibition were measured, and biocompatibility/ antimicrobial property of nanocomposite was elaborated.

RESULTS AND DISCUSSION:

Morphological and structural characterization of Ceramic nanomaterial :

A) UV-Vis. absorption and PL emission spectrum: Figure 1 and 2



Wavenumber	Abs. intensity	Rel. intensity	Width	Found if threshold <	Shoulder
1027.7208	0.828	0.009	10.9486	8.122723	0
1004.0444	0.818	0.150	96.6377	230.420853	0
910.9765	0.881	0.038	208.3734	56.551479	0

Figure 1 : UV-Visible absorption spectrum of **$CaTiSiO_5$** ceramic nanomaterial

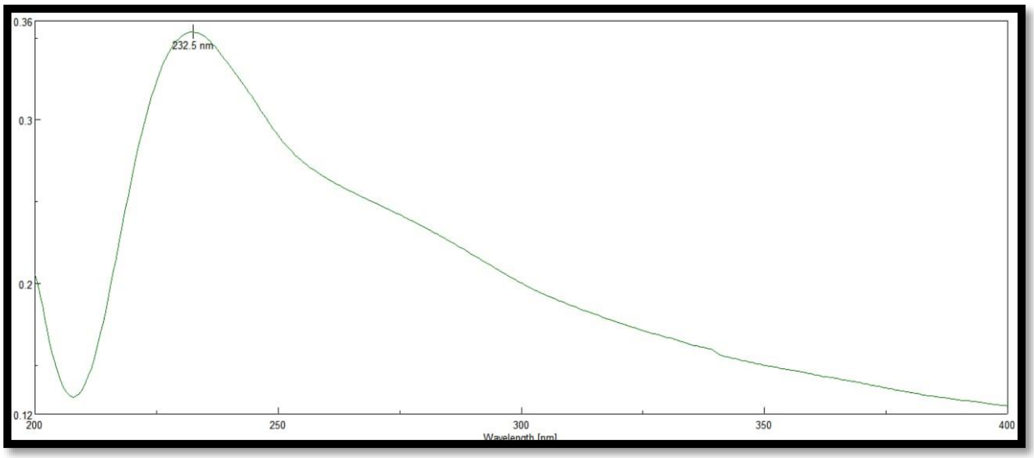


Figure 2 : PL emmission spectrum of **$CaTiSiO_5$** disc Nanomaterial

B) FTIR spectrum of ceramic nano material for surface functionalities :

As mentioned in Table 2, and figure 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 2: Matching of FTIR signals elaborating the functionalities of Schiff base and CORM complex. Figure 3 as above mentioned.

Signal in FTIR spectrum	Functionality	Ceramic group functionality
483 cm^{-1}	Ca-Ti linkage	Metal-oxide bonds
828 cm^{-1}	Ti-O linkage	Presence of linked oxide metal species
1050 cm^{-1}	Ti-Si group	Metal (I) and Metal (III) interaction
1385 and 1663 cm^{-1}	Presence of hydroxide	Surface groups of oxide nanomaterial
1764 and 3222 cm^{-1}	Presence of –OH and Oxides	Presence of surface moisture for porosity

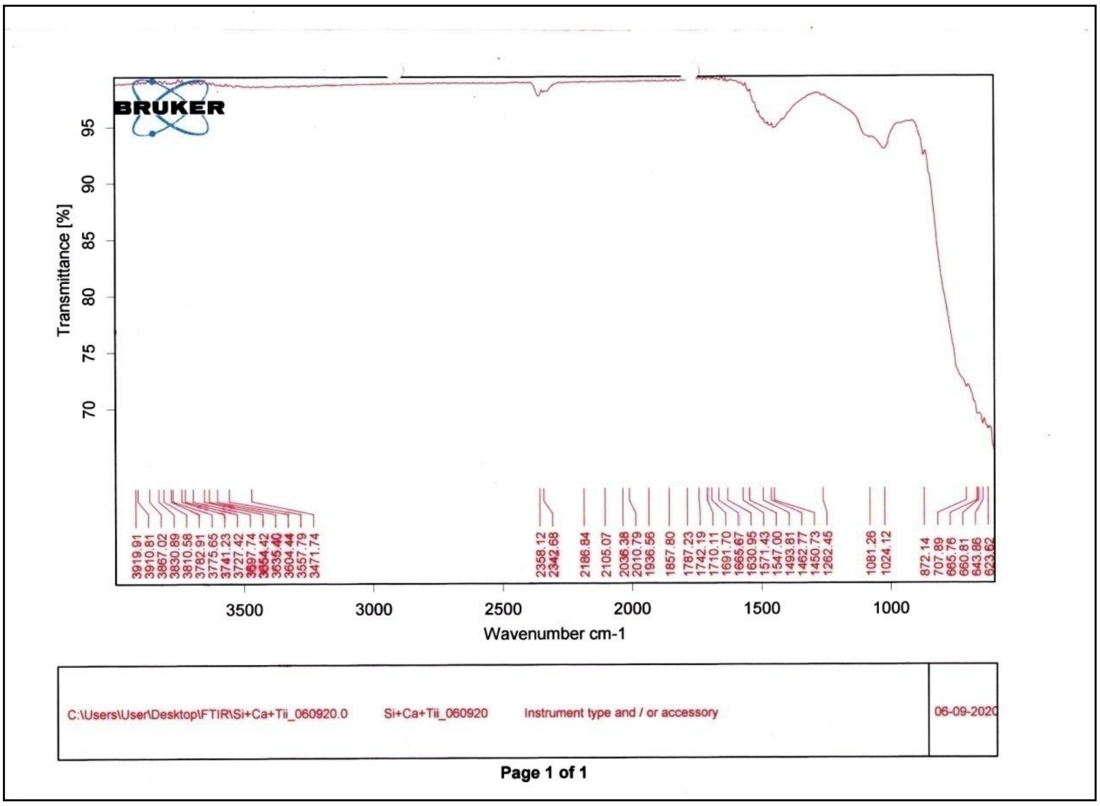


Figure 3 : FTIR spectrum of **$CaTiSiO_5$** ceramic nanomaterial for surface functionalities

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

Table 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 Calcium Titanate silicate oxide	Standard "d" (A) JCPDS Card No.01-073-2066 for **$CaTiSiO_5$** matched	Calculated "d" (A)	Lattice Constant (a, b, c)(A)
(111)	3.4107	3.5017	a = 7.0730 A b = 8.7180 A c = 6.5550 A (Standard)
(20-2)	**2.8478**	**2.7349**	
(211)	2.3651	1.8909	
(231)	1.8763	1.6650	a =6.6600 A b =8.6022 A c =6.5170 A (Calculated)
(400)	1.6157	1.6650	
(033)	1.6456	1.6650	

As per XRD data in figure 4 & Table 3 it had been proved that the ceramic nanocomposite has octahedral packing of ions and cubic phase purity elaborating the 65 nm size from main peak using Scherer's equation.

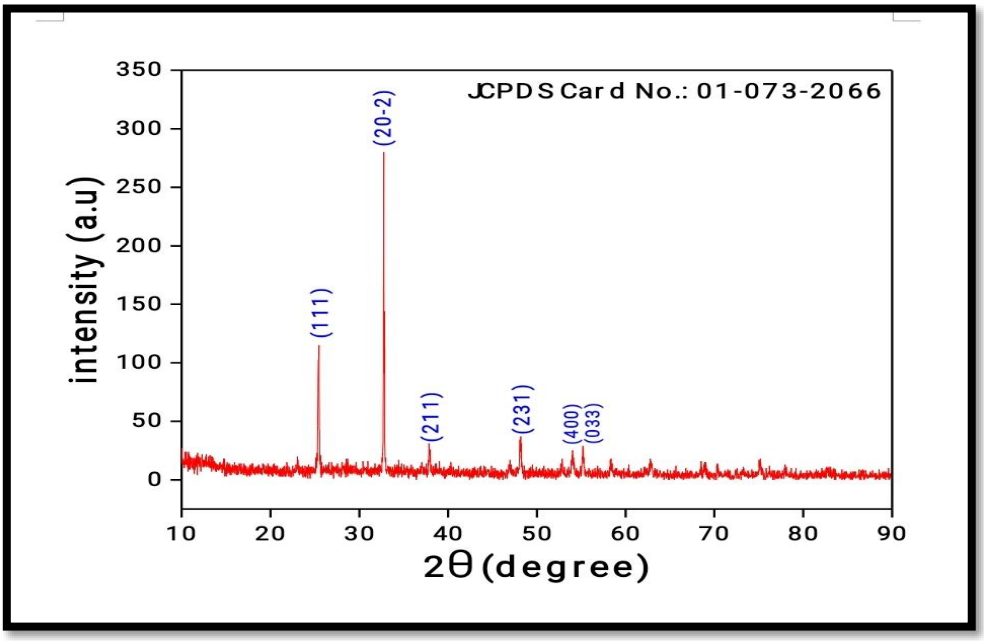


Figure 4 : XRD pattern of the disc **$CaTiSiO_5$** ceramic nanomaterial

D)SEM image for disc morphology : The SEM image of nanocomposite ceramic is as shown in figure 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 Oval shapes for better water loving nature and cell particle interactions.

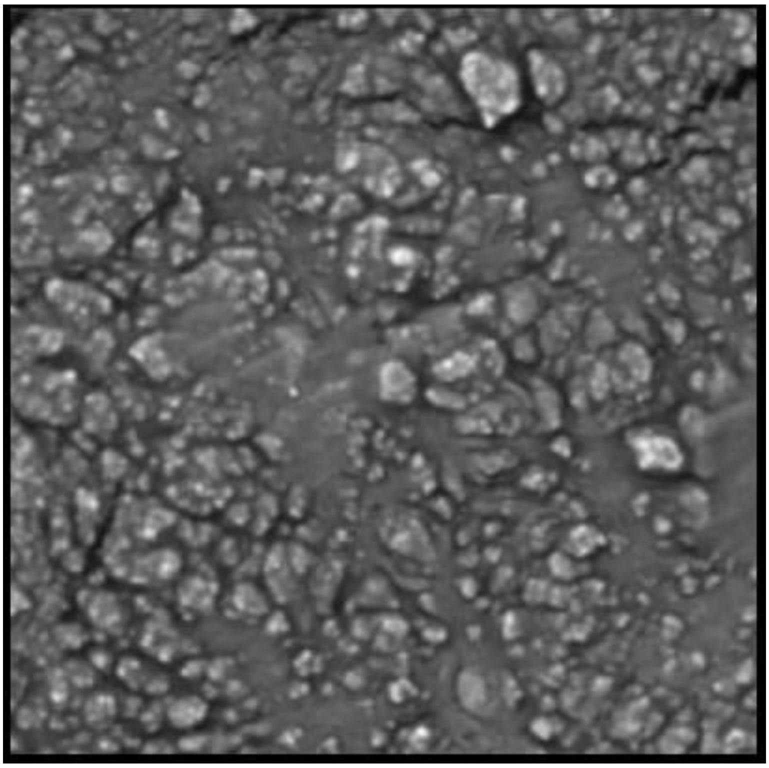


Figure 5 : SEM image of **$CaTiSiO_5$** nanomaterial with oval morphology

E)BET isotherm elaborating porosity of disc nanomaterial : As per figure 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.

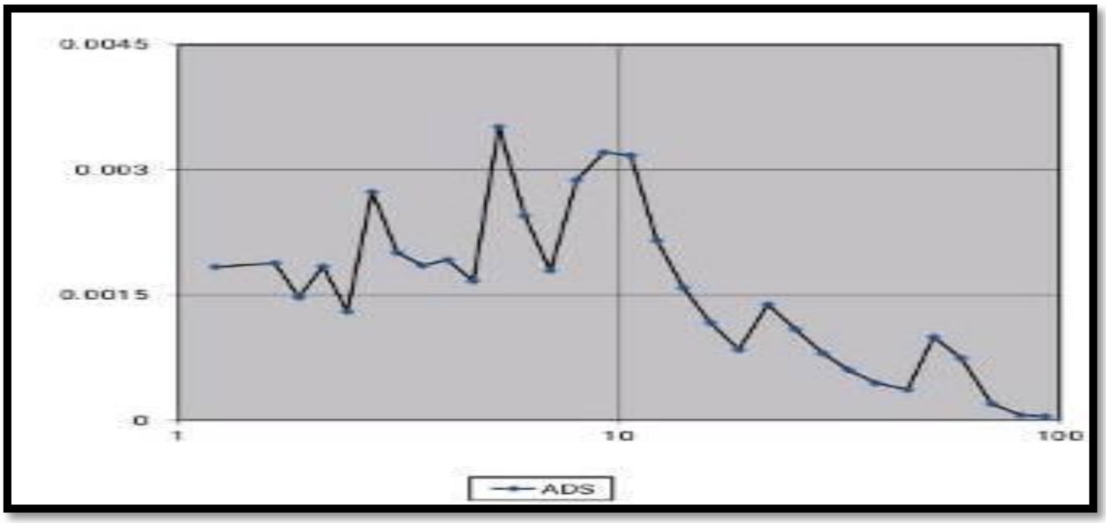


Figure 6 : BET adsorption isotherm of **$CaTiSiO_5$** nanomaterial elaborating surface porosity

7.4 Antimicrobial properties for water remediation from nanomaterial :

As per figure 7 and Table 4, for antimicrobial activity of 20 ppm. material on Klebsiella. It had been demonstrated that good zone of inhibition with better antimicrobial activity[9].

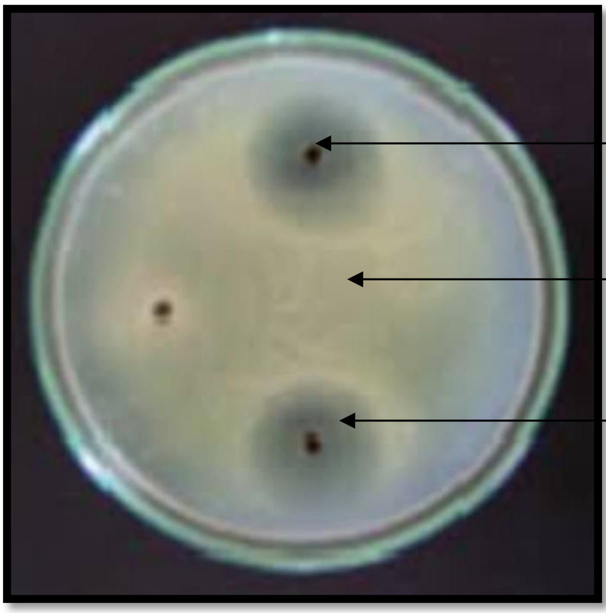


Figure 7: Anti microbial effects of ceramic nanomaterial on Klebsiella for zone of inhibition at 20 ppm.

Table 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. 7
Klebsiella *(gram -ve)*	15 mm.	27 mm.

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[8,9].

Water Remediation Activity of $CaTiSiO_5$:

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 wss observed in total 180min. of an interval of 30min. PH of solution is maintained 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 94% indicates that this metal complex is very effective for water remedial activity[9].

Table 5 Degradation Parameters

1.	Dye	Methylene Blue
2.	Concentration	20 ppm
3.	Photocatalyst’s amount	150mg/100mL
4.	Degradation Time	30 min
5.	Degradation Efficiency	94%
6.	pH	7
6.	Source of light	365 nm Hg Vapor lamp

Table 6 % degradation during course of time

Time	% degradation
Blank	00
Adsorption	10
30 min	94

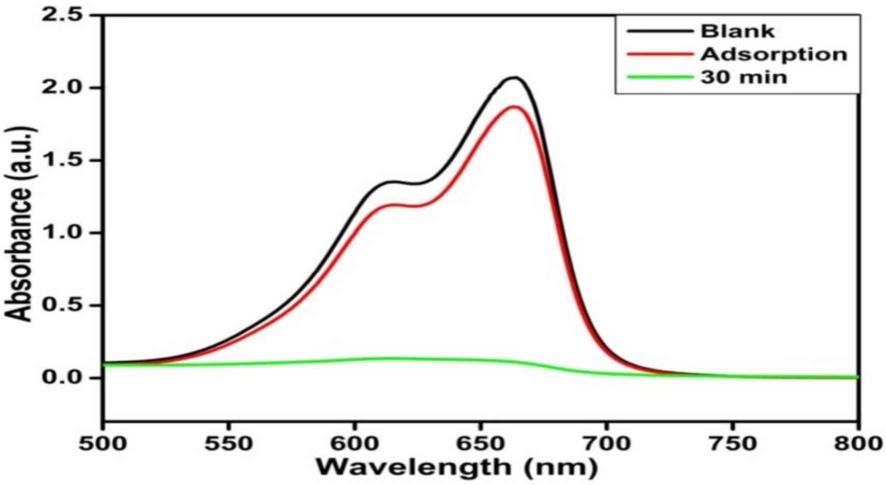


Figure 8 : Absorption Spectrum

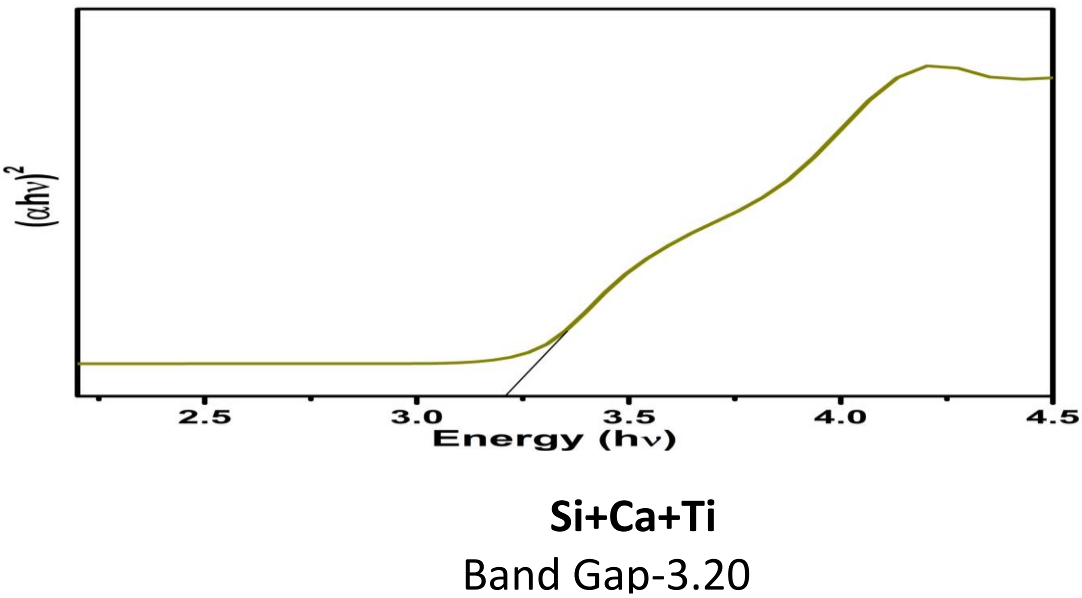


Figure 9: Band Gap

CONCLUSIONS:

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.

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