



THE SYNTHESIS MECHANISM AND CHANGE IN OPTICAL PROPERTY WITH DOPANT CERIA (CeO₂) in Al₂CeO₅ BASED NANOSTRUCTURED MATERIALS

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ABSTRACT

In the present world, Nano science in itself is a massive field for conducting research and bringing out interesting insights due to the superior behavioral traits exhibited by nanoparticles. The properties associated with their dependencies on size are an excellent property that makes it different from other types of research. In this research paper, the Nano stuff (NP's) structures of Al doped CeO₂ with varying dopant concentrations, i.e. 5%, 10%, 20% are manufactured prosperously by using the uncomplicated co-precipitation mechanism where the mixture is calcinated at 600°C for a total of 2 hours. The structure and morphology of the formed metal oxide Nano stuff samples and calcined as synthesized samples has characterized by the diffraction through P-XRD (X-Ray), UV-visible spectroscopy, FTIR spectrum, Transmission electron micrographs signify that the metal oxide Nano stuff sample are crystalline in nature and undergoes a grain size calculated by Debye Scherer's method is 30 nm and investigated structure is face central cubic structure. The UV visible adsorption spectrum is procured to examine the optical (energy) band gap which is showing the shifting range from 236 eV to 238 eV in the Nano metal oxide samples with variation of dopant concentration respectively. The Fourier transform IR spectrum of given samples disclose that the incorporation of Ce ion with the no. of oxidation state by replacing the position of Al in Al₂O₃ structure with metal oxide Nano stuff at 484.9 for CeO₂ and 633.3 for Al₂O₃.

KEYWORDS: Nano stuff, FCC, XRD, FTIR.

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1. Introduction:

A wide range of alumoxidalopates (Al metal oxides

with varying concentrations and cerium oxide) nanomaterial show different physical parameters,



magnetic, antibacterial, and anti-pollution characteristics, which helps to provide bacteria with new places to compete against them, as well as non-pollutant opposition properties. These characteristics change based on the composition of dopant. One thing researchers ought to take into consideration is field usage of nanoparticles: one can pour the particles into the vials used in the vials. It is required to have the term Expand ability. This means you have to identify an atomic number of the term "Al" and mark it as well as a widely used chemical sign for it, which are both Al. More has been produced in the upper earth crust than any other two of the three elements, namely oxygen and silicon. The aluminum is still bound to the oxygen, fluoride, and silicon particle in the alloy, though it is present in the Free State, is seldom observed. Other components can have special qualities including lightness, ductility, conductivity, and mechanical power, but only aluminum has a unique combination of these three: aluminum has corrosion, recyclability, reality, and ductility. A class of nanomaterial that is made from aluminum oxides has different biomedical application can be described as another expansion reason, as the material's price and texture were less demanding and favorable than for further study. There is no realistic distinction between accommodation and real estate since it's important to the running of the economy. In the medical industry, these drugs have multiple applications, most of which are as a coating or distribution material. Placed in usage in the environment, including cars, catalyst and in different ways like it, in microsatellite construction, satellites, and space, gamma-alumina often sees use as a catalyst and as a catalyst. Now in the Nano-based materials oxide, which has been incredibly intriguing owing to its antibacterial properties, is attracting considerable attention. The increase in the growth in the field of semiconductor or magnetic/magnet-pored nanoparticles has been well beyond what might be defined as a moderate of a scientifically or technologically (Paul -2012). The methods used in previous years had either magnetic or non-magnetic properties, which were discovered through research eISSN1303-5150

into the behavior of Nano materials (Ohno 2010). many physical characteristics have a large impact on the amount of the composition of the particles that are present in a Nano-system, chemical defects which is present in a system, and physical grain limits, which may be attributed to their varying concentrations in the delivery, and nature of the grain, or whether there is or lack of oxygen in the system. Nano materials have earned the spotlight because of interest due to their magnetic and electronic properties currently; these products are being used in various forms in the lab tests and procedures such as magnetic beads and electrodes in Bio applications like bio imaging and electrophoresis ology, and endocrinology. Nano et al., 2012), (Manna et al., - 2008 and Layek& Verma-2016). (as compared to other methods) the chemical process is simpler, faster, less complex this involves adding cerium oxide solution to co-precipitate and is less labor intensive than the lower amounts of aluminum oxide, thereby enabling different ceria concentration to be applied at a variety of temperatures than through co-preprocessing, which results in less time savings. property is a standard: The aim of this research is to build on established knowledge, leverage existing knowledge, and discover unknown properties as well It's been seen that cerium oxide content has antioxidant properties because of it's a re-regeneration. The reduced form in the 4+3 state is generated as well as the oxidized form when the redox (cycling) reaction is run. An almost exclusive research effort has been put into exploring the usage of germanium oxides in medical applications where it was discovered that increasing the oxygen level was an effective for treating various types of disease where greater quantities of cerium oxides were needed. This cerium is a part of the rare-earth metals family. To put it another way, we may assume that it is in the first block (F). Ce is a chemical which is represented by the capital letter C. It has a silky-elastic nature, softness, ductility, and a silver sheen. The atomic number is 58. The atomic number (number of total protons) is 140.116 it can be expanded to two additional states: it has two states: plus 4 oxidation

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states are known as the best state of cerium. Oxidothermutation does not take place under the presence of oxygen it is of minor industrial importance since it is a rare-earth metal Cerium oxide has little biochemical impact in the body, but is utilized for its de-acidification properties. In that regard, it is really safe to take-your-all natural. is part of a club of six melting stage 1463 degrees Fahrenheit Its crystal arrangement is one hexagonal prism layers with two hexagonal circles of close-packed hexagons. Cerium nitrate has shown to be an efficient in the work that has been done on the discovery of antimicrobial agents in this project so far, and further studies could be conducted in order to determine how effective it is against disease-causing bacteria. In the recent study, where concentrated cerium (NO₃) and aluminum (NO₂) nitrate solution is employed, findings show that cerium (III) oxide (CN) Nano (organic compound) precipitation is achieved by way of method withalic acid (inorganic amine base). Equal to the total elimination of phosphorus from water samples that are of varying degrees of purity. The creation of this Nano-manufacturing processes is eco-friendly There should be no interaction with hazardous chemical while one is prepared. Because of its importance to human wellbeing, phosphorus is mostly used as a pesticide, as well as for fruit. Plants started to fail because of the over-enrich water source due to phosphorus toxicity, which accelerated algae or plant growth, resulting in their consumption of excess oxygen. Because of a shortage of oxygen requirement, this water species' mortality, a hypoxic condition set of conditions has arisen because of this species' phosphorus. Since the phosphorus is just very long-term and permanent, then the safest way is to eliminate it as part of an overall fertilization plan. Nano materials of distinguish with respect to various components are formed for getting rid of impurities. Such as aluminum dioxide would be needed to act as an alkaline adj. and manganese complex bi-reducing bimetallic oxides will accumulate the impurities which will involve aluminum, like this (mulligan et al -2001). Cerium may be combined with the same volume of copper, eISSN1303-5150

and the property of eliminating contaminants differs. become far more secure with the business you won't be stressed to stay slim or pretty and you have more time for food now that you're on your full schedule can contribute to more relaxed meal plans become available for you now you will have more time to eat] Nano crystalline particles are sometimes used in thermal expansion applications, where expanded pellets or pieces of Lanthanum oxide or activated Al-Alumina mina are usually cause failure due to failure (xie et al - 2014). Magnetite nanoparticle and other Nano-constituents which are nontoxic, bio-reactive, and/deac-detectable As far as the experimental design work in this project is concerned, this section of the study was accomplished using micro particles, for removal of impurities Additional info on decontaminate the water, as well as using Nano-material surgical methods to decontaminate the water the specific conductivity and electrical resistance of tin oxide are also important characteristics. This research indicates that room temperature based ferromagism can be found in 3d transition metal doped materials: In₂O₃, CuO, and TiO₂O₂ and SnO₂ (Vadivel et al., 2011, Venkatesan et al., 2004, Hong et al., 2005 and Dubowik et al., 2007). A number of research groups have demonstrated the existence of iron, manganese us oxide, and cobalt oxy TinO (i.e.ganous) oxide among them (Liu et al., 2009, Xiao et al., 2008, Kashyap et al., 2009, Junying Zhang et al., 2010 and Singhal et al., 2010).

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2. Synthesis mechanism of Ce doped Al₂O₃ structureNano stuff

The chemicals deployed as a part of this research work are extra pure which were acquired in their purest form from nature and no modifications were done with that. These were A.R. grade with 99.9% purity at lab scale. As mentioned above, n further modification was done. The appropriate concentration of 1 M Al(NO₃)₃.9H₂O or Aluminum nitrate Nona hydrate solution was supplied by Himedia which was used for the experimental work, similarly appropriate concentration of 0.1M, 0.2M,



0.3M of Ce(NO₃)₂.6H₂O (Cerium nitrate hexahydrate) too was supplied by Himedia for the

purpose of this experimental research

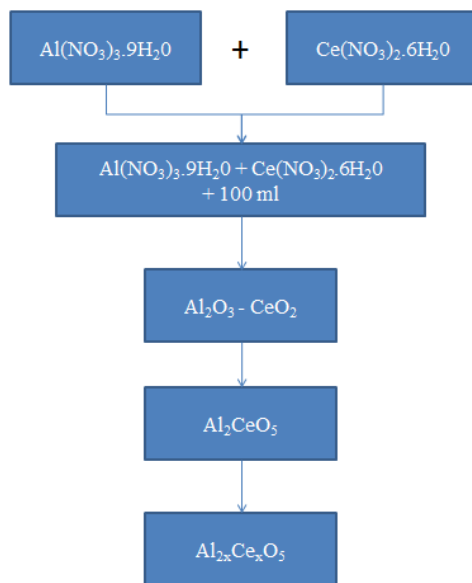


Fig 2.1Flow chart for the preparation of (Al - Ce) doped Nanostructured

To prepare Al doped with CeO by co-precipitation method, Firstly took 1M Al₂(NO₃)₃ nearly about 21.2996g aluminum nitrate in three proportion. Then took 0.1 M, 0.2 M, 0.3 M Ce (NO₃)₃. 6 H₂O nearly weight about 4.3422 g, 8.6844 g, and 13.0266 g respectively. Which is done by weighing balance? Then took one proportion of aluminum nitrate and 0.1 M cerium nitrate in 100 ml containing distilled water and dissolved with glass rod with constantly for few minutes. In other beaker took weight nearly about 21.2996 g of aluminum nitrate and 0.2 M cerium nitrate in 100 ml water again dissolved it constantly shaking for few time. Again in third beaker took aluminum nitrate with same quantity which mention above then added 0.3 M cerium nitrate in 100 ml water with constant shaking with glass rod. After this process, put one magnet in each sample and this sample put on the magnetic stir for 15-20 min until clear solution is obtained. Then after this step provided slightly basic medium for the process of precipitation, the level of conc. Of hydrogen ion must be 8-10. Then formation of precipitate takes place. Then filter it by Whitman eISSN1303-5150

filter paper. Then abandon this reacting sample for 24 hours for resting. After this process of filtration the reaction mixture washed with distilled water to remove the extra impurities and then placed on watch glass container then put into oven for drying nearly about 200-230 degree Celsius then formation of dry cake take place. The resulted samples were calcined at different temperature and thereafter, crushed in agate mortar and then put into sample tube similarly these process are carried out at different temperature for same hours.

3. Characterization Results

3.1 XRD characterization:

The grain size and lattice structure of the related sample investigation of Nano metal oxide of Al₂O₃.CeO₂ specimens were detected with the instrument powered X-ray diffraction pathway which is depicted in Fig 3.1 In the P-XRD graphs, the diffractions alps were majorly attributed to the Al atom incorporated at the position of Ceria in form of CeO₂ in the FCC structure according to the JCPDS card no. JCPDS – (00-004-0787) and JCPDS – (00 – 004 – 0787).



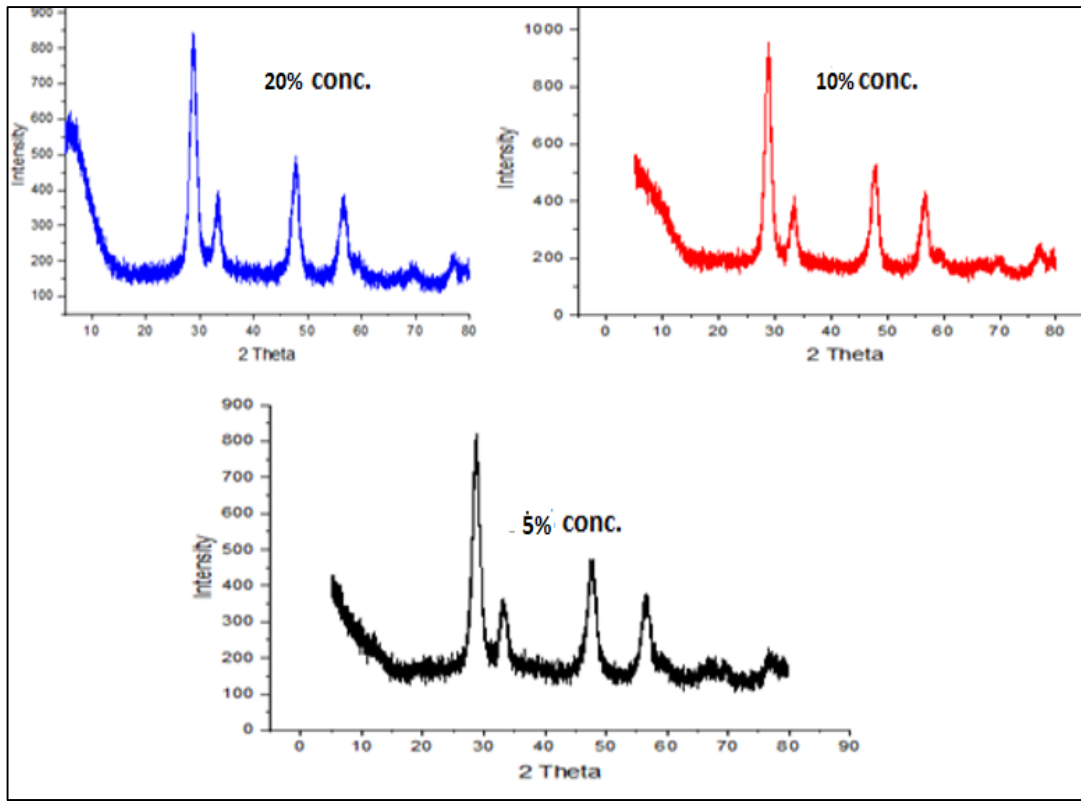


Figure 3.1 XRD of Ce doped Al₂O₃Nano stuffs

Table 3.1 – XRD -1 given the value of grain size of Aluminium oxide with Ce at various dopant concentration levels

S.No	Dopant conc. %	Position (2θ)	FWHM (β)	Size (nm) D(spacing)
1	5%	28.698	0.90	31.0816
2	10%	28.821	0.09	30.950
3	20%	28.944	0.09	29.883

The crystallite size was estimated using the Scherer formula

$$d = \frac{k \lambda}{\beta \cos \theta}$$

Where k = shape factor = 0.89

λ = wavelength = 1.54 Å

β = FWHM = given in the above table

cosθ = Angle (position) = given in the table

In X-ray diffraction, the spectra reveals that as we increase the dopant concentration of Cerium in Aluminum oxide, the grain size keeps on decreasing continuously as shown in the table above. Transformation of the Ce²⁺ to



Ce³⁺ ions is the main reason for this phenomenon due to which the bond length between Cerium atom and oxygen atom goes on decreasing. This is possibly due to more availability of Ce ions with varying concentration of dopants.

Table 3.2 XRD-2 pattern peak concentration at constant temperature

S.No	Sample	Dopant conc.%	Temperature for 2 hours	Contract peak 1	Contract peak 2
1	X1	5%	600°C	-	-
2	X2	10%	600°C	47.814	59.578
3	X3	20%	600°C	47.937	59.455

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It is clearly visible from the above pattern that an additional peak is seen as raising the dopant concentration as depicted in above table 3.2 where two additional peaks are shown. These peaks show a contracting behavior due to transformation of the Ce²⁺ to Ce³⁺ ions. Higher availability of Ce³⁺ ions under varying dopant concentration is another major cause. One peak is showing the left shift (59.578 to 59.455) due to merging Alps of Transformation Ce²⁺& Ce³⁺ ions.

3.2Transmission electron microscopy (TEM) analysis technique:

Transmission electrons analysis technique reveals that the surface (behavior) investigation of the cerium doped Al₂O₃ Nano stuff in figure 3.3 indicate the transmission graphs of spectroscopy of without doped and with doping Cerium into Al₂O₃ Nano particles. The TEM analysis of formed samples indicated a spherical shape of Nano moiety. It was further clearly observed that these have no substantial difference in the surface TEM of Nano stuff of Ce doped Al₂O₃ and has maintained a spherical shape but also growth of Al₂O₃Nano stuffs by doped Cerium.



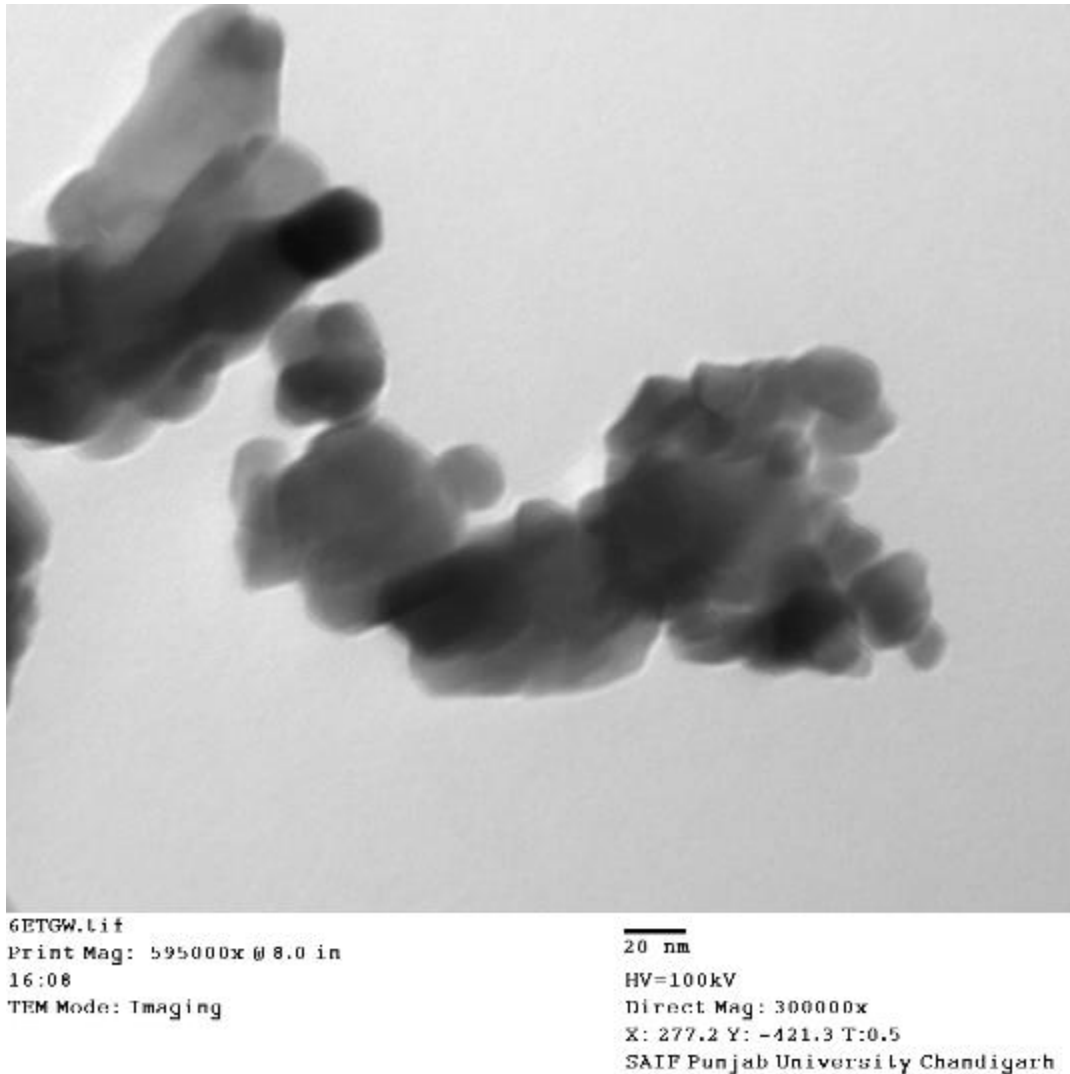


Fig 3.2: TEM-1 spectroscopy Ce (10% doped Al₂O₃)

3.3UV-VIS absorption spectrum analysis:

The spectroscopic result of UV–VIS adsorption of electromagnetic (behavior) radiation of the metal oxide doped Nano stuff had been examined in the scale range between 200nm to 1200nm. To procure the computation of UV-VIS adsorption, the ignited (calcined) an erect (as assembled) Al₂O₃.CeO₂ specimen has been disseminated ultra-visibly in the absolutist form of ethanol to prevail a homogeneous

suspension mixture form solution and was placed in the cubic shaped sample holder and examined relatively with ethanol in purest form. The UV visible graph plots depend in Nano sample data that is λ (wavelength) VIS adsorption rate for Ce doped)Al₂O₃specimen Nano stuff calcined at temperature range of 600°C for a time period of 2 hours as shown in the table 3.3

S.No	Concentration at 600°C	Wavelength in nm	E = 1240/ λ (Energy)
SU1	20%	238	5.210

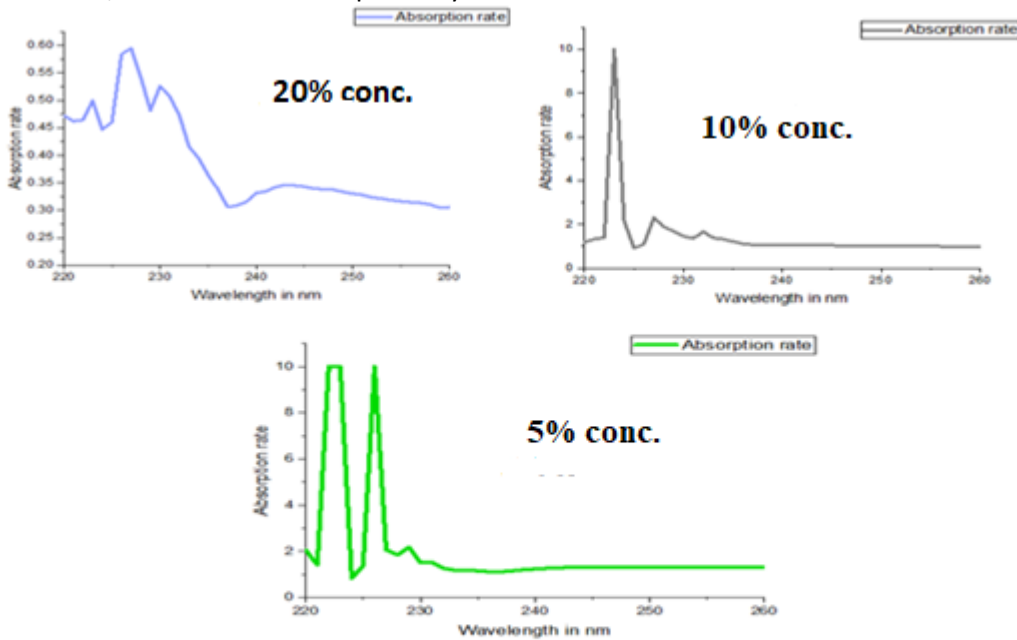


SU2	10%	233	5.321
SU3	5%	228	5.438

Table 3.3 UV visible spectra data of wavelength with varying concentration

It has been examined that the band edges of metal oxide nanoparticle is 238nm (UV range between 200 to 400nm). The above table clearly depicts the UV visible spectrum data of λ (wavelength) with varying concentration of dopant material. In the UV VIS spectrum data of Nano doped stuff at constant temperature mentioned in the table with increasing dopant concentration i.e. 10%, 20% and 30% respectively, its wavelength is derived by UV-VIS spectrum are 238, 233and 228 nm respectively. This

pattern shows a bathochromic shift also known as Red shift with a reducing energy band gap, i.e. 5.438, 5.321 and 5.210 respectively. This shows a radical shift in reduced insulating behavior but when the dopant concentration is gradually increased, the insulating behavior is lost and the formation of metal oxide Nano stuff goes towards exhibiting a dielectric behavior with increasing dopant concentration by Cerium.



Ce doped Al₂O₃ at 600°C for 2 hours at various concentration

3.4 Fourier transform infrared spectroscopy (FTIR)

FTIR is an elementary tool to ascertain and substantiate the functional compositional scrutiny of as-synthesized calcined Nano sample followed by dopant concentration of Ce in Al₂O₃ at 10%, 20% and 30% concentration level respectively. The spectroscopic mensuration of FTIR for the varying dopant concentration of Ce in Aluminum oxide

nanoparticle heated at 600°C for a time period of 6hours was completed using PerkinElmer instrumentation (SAIF, PU, Chandigarh facility). In fig-3.4 Given the FourierAlps of Ce doped Al₂O₃ at different conc. of dopant.

For 30% dopant concentration, it was observed from the analysis of FTIR spectra at 600°C for 2 hours' time period and the FTIR alps are



observed at 3860.5, 3394.6, 1627.8, 1383.4, 1070, 895.7 and 533.7 cm⁻¹. The broad Alps are observed at 3860.5 cm⁻¹ due to the -O-H vibration of water molecules present in the atmosphere and another broad Alps at 3394.6 cm⁻¹ showed the presence of amine group. The synthesized samples were prepared in the basic medium as some washing impurities were left in the sample. A strong alp is also observed at 627.8 cm⁻¹ due to the formation of -O-Al-O-OH bond. A sharp IR Alps at 1383.4 cm⁻¹ exists due to the presence of Al=O bond vibration that represents Al₂O₃ formation. One broader peak is formed at 1070 cm⁻¹. The FTIR peak at 895.7 cm⁻¹ represents γ -Al₂O₃ formation (in γ -phase out of vib) and CeO bonding at 533.7 cm⁻¹ respectively.

For 20% dopant concentration, the FTIR alps are observed at 3365.2, 1629.5, 1383.5, 916.8, 538.5 and 476.5 cm⁻¹. The broad FTIR peak at 3365.2 cm⁻¹ represents the NH₂ vibration due to the sample being prepared in the basic medium and some washing impurities were left. A sharp strong alp is also observed at 1629.5 cm⁻¹ due to the formation of Al (OH)₃ as an intermediate bond. A strongly sharp peak is observed at 1383.4 cm⁻¹ exists due to the presence of Al=O bond vibration that represents Al₂O₃ Nano stuff. A broad alp is found at the position of 916.8 cm⁻¹ and a sharp peak at 538.5 cm⁻¹ represents formation of γ -Al₂O₃. A clearly intense band appears at 476.5 cm⁻¹ indicating a Ce-O bonding which exhibits formation of CeO₂ Nano moiety.

For 10% dopant concentration, the FTIR alps are observed at 3890.2, 3429.8, 2098.2, 1632.8, 1384.3, 1068.9, 762.1 and 621.4 cm⁻¹. The broad FTIR peak at 3890.2 cm⁻¹ represents the NH₂ vibration due to the sample being prepared in the basic medium and 3429.8 cm⁻¹ represents O-H vibration due to water molecules present in atmosphere added during the sample investigation. The FTIR alps at 2098.2 cm⁻¹ and 1632.8 cm⁻¹ represent Al-O-O-H intermediate during the production prepared. 1384.3 cm⁻¹ arising due to Al=O bond to constitute Al₂O₃ genesis and 1068.9 cm⁻¹ recommended formation during the reaction mixture composed. Two more broad peaks had been

found at FTIR spectrum 762.1 cm⁻¹ of CeO₂ which is ascribable to Ce₂O₃ formation and 621.4 cm⁻¹ caused by Al-O bonding, it might be a layout of Al₂O₃ Nano stuff (Metal oxide particles). It was also observed that the FTIR peak at position 762.1 cm⁻¹ was omitted in 20% and 30% dopant sample which indicates the formation of CeO in the Nano sample. The peak at this point indicates formation of Ce₂O₃ whereas the peak at 621.4 cm⁻¹ indicates the formation of γ -Al₂O₃ in the sample. The peak in Nano sample at 10%, 20% and 30% is 1068.9, 916 and 1070 respectively was attributed to the formation of Al(OH)₃ as an intermediate bond and the peak at 1383 cm⁻¹ indicates Al=O trenching models.

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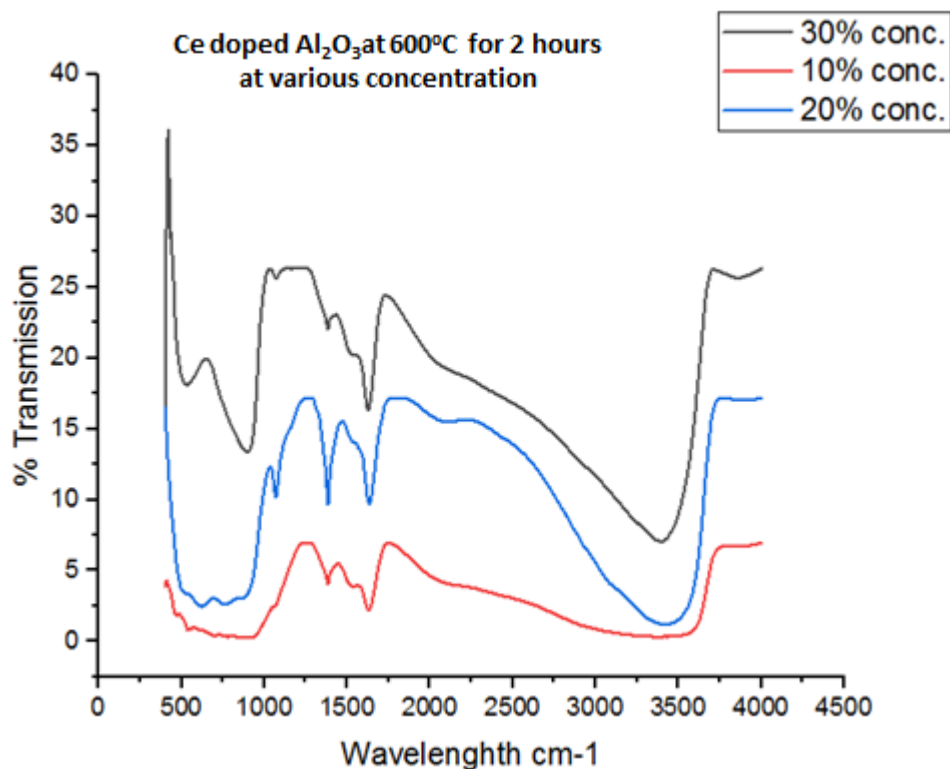


Fig 3.4

Conclusion:

The present research work has explained the preparation method of Al₂O₃modified CeO₂ Nano structured moiety by using the chemical co-precipitation method. The P-XRD and TEM (spectroscopy) results have clearly indicated that the Aluminum oxide Nano crystals are crystalline in nature and exhibit aFCC (Face centered cubic) structured geometry. The mean size of Nano-crystal goes on decreasing with an increase in Ce doping concentration. In terms of the UV visible spectroscopy, it was clearly revealed that the optical band gap or energy gap observed with the Ce doping concentration, a value above 5.0 eV shows an insulating behavior but when the dopant concentrate on is relatively increased, the insulating characteristics are overshadowed because the Nano stuff formation tends towards a dielectric behavior. The appearance of Ce in Al₂O₃sample is entrenched by FT spectra attributed to alps at 476 cm⁻¹ and 533 cm⁻¹. The TEM spectrum reveals that Nano moiety possesses spherical in shape and size aspect is

very much similar to that observed in XRD results.

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