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A COMPARATIVE STUDY ON CHEMICAL SYNTHESIS AND CHARACTERIZATION OF CERIUM OXIDE NANOPARTICLES.

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Abstract

The word nanoceria represents to nanoparticles of cerium oxide which is a rare earth metal oxide. It has variety of application in the diverse field of technology. This review is based on different chemical synthesis methods of nanoceria and their growth parameters. Method of preparation and their parameters are responsible for change in properties of material. It has been observed that, nanoceria with small size and controllable morphology can be prepared by co precipitation method. The synthesis parameter like precursors, pH value, stirring condition, bath temperature and annealing temperature may responsible for the change in properties of materials which directed toward variety of application.

***Key-words* :** Cerium Oxide; Chemical synthesis route.

Introduction:

Nanoparticles have a pivotal role in diverse applications owing to their astonishing physical and chemical properties, makes them different from bulk materials. Rare earth elements, known for their unique structures and qualities, have sparked significant interest in the realm of nanotechnology. Cerium with an atomic number of 58, belongs to a lanthanide series, and more abundant among all rare-earth metals. Along with all the available members of nanostructure lanthanide oxides, Cerium Oxide nanomaterial have been widely investigated as functional materials [1,2]. The rare earth metal oxide nanoparticles demonstrate amazing luminescence, electronic, and magnetic properties due to the presence of vacant 4f electronic structure as proven by various characterization techniques [3,4,5]. In stable state, cerium has a fluorite structure and has two (Ce^{3+} and Ce^{4+}) oxidation states. The material can store and release oxygen because it has capability to change their state between trivalent (+3) and tetravalent (+4). It has an optical band gap of 3.19 eV with transparency in the visible spectrum (400-800 nm), high refractive index and exceptional dielectric properties [6]. The striking properties of CeO_2 make this material important for applications, such as electrolytes [7,8] catalyst [9,10,11] solar cell [12] manufacturing of semiconductor [13] polishing material [14,15] fuel cell [16,17] absorbing material [18] and automotive exhaust purification [19,20]. It has also been used as a three-way catalyst (TWC) in vehicle gas engines. This material also shows tremendous biological application applications in treating cardiovascular diseases, sepsis and cancer etc [21].

The CeO_2 nanoparticles are synthesized by different methods such as combustion techniques, sol-gel techniques, hydrothermal techniques, solid-state reaction method, co-precipitation techniques, chemical bath deposition technique, spray pyrolysis techniques and so on. Among all the available methods chemical co-precipitation method is usually preferred because it is easy to perform, cost-effective, efficient, and also allows controlling the structural parameters through optimization of the synthesis conditions. This method gives precise control on shape and size-controlled synthesis of the nanomaterials. One can tailor the process to get micro and nano sized particle by adjusting the pH, stirring rate, reaction time, precipitating agent, temperature and solvent. In this review, a brief out-line about methods for preparing doped and undoped CeO_2 nanoparticles, their properties, and

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applications have been discussed.

Literature review:

Tumkar et al [22] has synthesized the CeO_2 nanoparticles by hydroxide mediate method and obtained particles were in yellowish white color. In this method the cerium nitrate hexahydrate is dissolved in deionized water to obtain a homogeneous solution, results the conversion of trivalent state to tetravalent state. Then it gets reacted with sodium hydroxide to produce the nanoparticles of cerium oxide. Size of the nanoparticle so obtained is around 10 to 30 nm having the cubic fluorite structure. They were performed biocompatibility studies using Beas-2B cells in MTT assay, Live/Dead viability assay, and ROS assay and found that the CeO_2 nanoparticles are compatible with the cells and there was no cell death even at higher concentrations of nanoparticles. Therefore, CeO_2 nanoparticles could propose for different biomedical applications like biosensors and in cancer therapy.

Mahsa Zarinkamar et al.[23] have synthesized CeO_2 nanoparticles by using wet chemical method. The cerium chloride, hydrochloric acid and ethanol were used as precursors. The cubic fluorite structure of CeO_2 with size 50 nm was obtained. It was observed that, the particle size is increases with the increase of the annealing temperature and the particle shape changes from sphere to cubic. The sharp peaks in FTIR spectrum shows the purity of CeO_2 nanoparticles and absorbance peak of UV-Vis spectrum shows the bandgap energy of 3.26eV.

Sr.No.	Name of the method	Precursor	Growth parameter	Compound and structure	Average Particle Size (nm)	Ref.
1	Hydroxide mediate method	Cerium(III) nitrate hexatydrate, Sodium hydroxide, Deionized water	15 min Centrifuged at a rate of 8000 rpm, dry it using hot air oven at 200 °C.	CeO_2 Cubic Fluorite structure	10-30	[22]
2	Wet chemical method	Cerium chloride, hydro-choleric acid, ethanol	pH=2, Precipitate heated at 70°C for 4 hours and then cooled down to room temperature. 4 hour calcinations is done at 500°C and1000 °C.	CeO_2 Cubic Fluorite structure	30-80	[23]
4	Chemical Bath	$Ce(NO_3)_2$, KOH, NH_4NO_3, $SC(NH_2)_2$	Temperature was Maintained at 23±2° and stirred for 4h. Samples were annealed at1000°C for 2h in air atmosphere.	CeO_2 face-centered cubic phase structure	2.7 to 28.5	[24]
5	Co-precipitation method	Ce(III) Nitrate, Distilled water, K_2CO_3	pH=6 Precipitates were dried at 65°C for 2 hours.Then first the sample were calcined at 220°Cfor a 2.30 hours, then sample were calcined at 600°C for a duration of 3 hours.	CeO_2 Cubic fluorite structure	20	[25]

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6	Chemical method	Cerium chloride, Deionized water, Ammonia solution	pH=10. Stirred for2 h then dried overnight. Then, heated in a furnace at a temperature 400°C for a duration of 2h.	CeO_2 Cubic nano crystals	15.14	[26]
7	Chemical precipitation	Cerium (III) nitrate, ammonium acid carbonate, n-butyl alcohol, poly ethylene glycol, Doubly distilled water, absolute ethanol.	Reaction temperature 90°C for time 1 hour 15 min. The mixture was stirred continuously for 30min. Afterword's sample was heated at temperature 80°C for a duration of 5h, and then dried powders were again heated at temperature 800°C.	Ceo_2 Hexagonal fluorite structure	90- 150	[27]
8	Chemical precipitation	Cerium nitrate, Europium nitrate, and ammonium hydroxide, Deionized water	pH = 9 was maintained for sample which dried at 120 °C. The mole fractions of Eu was varied for samples from1% to 30%.	$Eu:CeO_2$ Cubic fluorite	5-7	[28]
9	Wet chemical precipitation	Cerium (III)nitrate hexahydrate, NaOH, Distilled water.	pH=13 was maintained and then sample were subjected to ultra-centrifuge technique at a speed of 10,000 rotation per min. for a duration of 10 min.and dried at temperature 353K for duration of 3h.	CeO_2 Cubic Fluorite structure	~5.2nm	[29]
10.	Precipitation method.	Ceric ammonium nitrate, ammonium carbonate	Stirring duration 30min. yellowish solution obtained. After 30 min, this yellowish solution was filtered and 2 mL were mixed with 2 mL of pure etanol and maintained at 55 °C or 70 °C for 16 h. Then centrifuged and dried in oven at 90 °C for 48 h.	CeO_2 Cubic Fluorite structure	3 to 6.	[30]

11	Bio synthesis route	Salvadora persica Distilled water, Cerium Nitrate, Nickel Nitrate	Aquous S. persica mixed with Cerium Nitrate. Doping is done by adding Nickel nitrate. Kept in water bath of 70°C for 3 h, then dried at 80°C and annealed at temperature 400°C for2h.	Ni:CeO_2, Fluorite cubicstructure	5-6	[31]
12	Sol-gel process	Sodium sulphite, metal salt, cerium (III) nitrate, distilled water.	Treated thermally at60 °C in the drying oven for at least 6 h duration	TM-doped CeO_2	3 to 5μm	[32]
13	Co-precipitation method	Zinc chloride and Cerium (III) nitrate hexa hydrate and oxalic acid, De-Ionized water	Reaction time 24 h at room temperature. Then centrifuge at10,000 rotation per min. for 10 min, and then annealed at temperature 450°C For two hours.	Zinc doped Cerium oxide	6–8nm	[6]
14.	Facile method	Cerium chloride and $NH_3{\cdot}H_2O$	Stirred 24 h, then kept at room temperature for10 days.	CeO_2 Cubic fluorite structure	5-6nm	[33]
15	Hydro-thermal synthesis	$(NH_4)_2Ce(NO_3)_6$ distilled water. Ethylen diamine hydrazine $N_2H_4.H_2O$	All precursors are mixed and then heated at120°Cfor5 h. Then dried at 60° C. Further heated at 500°C for duration of 1h.	Cerium oxide	10–20 nm	[34]
16	Co-precipitation	cerium nitrate hexa hydrate, CTAB, NaOH	CTAB mixed with Distilled water and added with of NaOH solution. Continuously stirred at normal temperature, resultant solution is again stirred at same temperature for 2hour and left over night at rest.Centrifuged at 1200 rpm for 10 min. then dried at 100°C for 2hour. After grinding samples were calcinated at 600°C for 2 hour.	Cerium oxide cubic fluorite structure	15.39nm	[35]
17	Solution Combustion Method	Ceric ammonium nitrate, EDTA	$(NH_4)_2Ce(NO_3)_6$ and EDTA disodium salt was treated with HCl. The obtained precipitate was	CeO_2 Cubic phase	42nm	[36]

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			washed several times with water and dried at 80°C for 1h. The dried EDTA and Ceric ammonium nitrate, were mixed with distilled water, and then stirred for 10 min. The solution was preheated and dehydrated at 150°C. After dehydration, a gel formed, and this was introduced into a preheated muffle furnace maintained at 450°C.			
18.	Chemical precipitation	Aqueous ammonia, cerium nitrate hexa hydrate, deionized water	Precursor was strongly stirred for half an hour after adding ammonia, again stirred for a duration of 10 h at room temperature. And then dried in oven at temperature of 60°C for duration of 3 h. then grinded for 15min and then annealed to a range of temperature between450°C and 900°C for2h.	CeO_2 Cubic Fluorite structure	50nm	[37]

Table1:Various chemical method, and precursor used for the synthesis of Cerium Oxide nanoparticles.

The synthesis of CeO_2 powder by using chemical bath as green method is presented by O.Portillo Moreno et al [24]. He has developed an economically reasonable precipitation method which allows large-scale production at room temperature and studied its pre and post thermal annealing (TA) effect. The absorbance of as-grown spectra shows three bands, while the thermal annealed sample shows only two bands confirms its better crystallinity compared to the as-grown sample.

Farahmandjou et al [25] have synthesized cerium oxide nanoparticles using cerium nitrate and potassium carbonate using co-precipitation method. The constant pH=6 was maintained during the process. They have observed the change in morphology to the spherical shape and particles are less agglomerated by increasing temperature. These CeO_2 nanoparticles showing strong UV-Vis absorption around 500nm with a sharp absorption peak at 380nm and gives the direct band gap about 3.26 eV.

Muthuvel et al [26] has synthesized the nanoparticles of CeO_2 by sol-gel method using $CeCl_3$ and ammonia solution. He has reported that the particle size and shape of nanoparticle can control the absorption position, thus responsible for band gap energy.

Nanoparticles of Cerium oxide having sphere-like shape were synthesized by precipitation method, using cerium nitrate as raw material and ammonium acid carbonate as precipitation agent. The yellow coloured precipitate was obtained. The average particle size was 90 nm and having hexagonal fluorite structure. Q. Zhang et al [27] found that, dispersant agent showed important roles on the size of resultant particles, and the presence of supersonic wave prohibited the precipitate from agglomerating partially.

Reza Zamiri et al [29] has synthesized CeO_2 nanoparticles by a wet chemical precipitation method. The sample were prepared by using $Ce(NO_3)_3.6H_2O$ and NaOH at room temperature with a constant

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pH about 13. The dielectric properties and ac-conductivity of the sample has studied. The structural in-homogeneity in sintered CeO_2 nanoparticles which was created due of large surface-to-volume ratio may results the high values of dielectric constant in low frequencies regions.
Cerium Oxide nanoparticles with high surface area were synthesized by the precipitation method with a narrow band gap (2.73 ± 0.03 eV) by Alission et al [30]. Ceric ammonium nitrate, and ammonium carbonate were used to synthesize the cerium oxide nanoparticles. On mixing immediately white precipitates were appeared and get easily dissolved on stirring.The excess ammonium carbonates are used to maintain pH value of the mixture around 9. After30 min, the orange yellowish solution composed of ceric ammonium carbonate was filtered and mixed with water and/or ethanol at different temperatures for 16 h. Nanoparticles precipitated after this time period were washed 3 times with water, centrifuged and kept in a drying oven at 90°C for 48 h. The optical study shows a red shift in the absorption spectrum from UV to visible region. It was observed that the high structural disorder and and Ce (III) fraction are responsible for the narrow band gap. The high Ce(III) fraction at the surface also improves the water adsorption.The presence of oxygen vacancies makes it more reactive sites in cerium oxides, and it becomes an important factor to improve the photocatalytic activity.

Prabaharana et al [39] used precipitation method using cerium sulphate and oxalic acid to produce cerium oxide nanoparticals having cubic structure. The average crystallite size of cerium oxide nanoparticles wasfound to be 11nm.They have found that optical absorption spectra show a strong red shift of the absorption threshold edge compared with bulk CeO_2 material. The obtained band gap was found 2.8 eV. The author has found the decrease in values of dielectric constant and the dielectric loss of the cerium oxide nanoparticles with increase in frequency. The AC electrical conductivity result shows that both frequency and temperature are responsible for the conduction.
Guofeng Wang et al.[33] studied the photoluminescence of CeO_2 nanoparticles by a facile method at room temperature using precursor like cationic surfactant (CTAB), cerium chloride and aqueous ammonia. They have synthesized the crystalline particles of 4–6 nm of size having cubic fluorite structure. The photoluminescence properties at room-temperature were investigated under the excitation of 325 nm, results the PL of the blue light at 452, 469, 483, and 493nm, respectively.
Suresh et al [37] have prepared the nanoparticles by chemical precipitation method using cerium nitrate and aqueous ammonia as precursor. They have studied various optical properties of the material. Photoluminescence spectra confirm the presence of blue emission in the visible region. The FTIR spectra reveal the existence of phonon band of cerium oxide network. Annealing temperature increases the particle size and change the shape of particle. It affects the surface, structure, electrical conductivity andoxidation states of cerium oxide nanoparticle.

The role of oxidizing agents on the properties of CeO_2 nanoparticles were also studied by suresh et al [38]. They have prepared the sample with different precipitating agent like NH_3, NaOH and KOH using cerium nitrate as a source material. As compared to others the sample prepared by using NH_3 gives better crystallinity and smooth morphology with small particle size. This may use to develop gas sensors, electro-chemical devices and optoelectronic devices.

Vidhi Pathak et al [35] have synthesized nanoparticle of CeO_2 using coprecipitation method. The obtained sample has cubic fluorite structure. The maximum optical transmittance around 80% was observed in UV region. The defect-induced decrement of bandgap (2.47 eV) makes it most prominent material for photocatalytic application. It shows a admirable photocatalytic effect (~76%degradation) under UV light source for a 0.6g/L catalyst dose.

The effect on luminescence properties of cerium oxide doped with Europium nanoparticles have been studied by Amit kumar et al [28]. They have used simple chemical precipitation technique to synthesize europium-doped cerium oxide nanostructures. The strong visible emission was obtained even from 1 mol % dopant concentration by retaining the trivalent oxidation state of Eu in

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nanocrystalline ceria. The PL intensity increases with dopant concentration and saturates at 15 mol% dopant. Quenching of the PL was observed on further increase in the dopant amount. Reducing the concentration of oxygen ion vacancies by annealing gives an increase in the PL intensity.

Zinc doped Cerium oxide nanoparticles were prepared using co-precipitation method by Aseena et al [6]. Doped nanoparticles show enhanced absorbance in the UV–Vis range as compared to the pure CeO_2 nanoparticles. The change in energy band gap as a function of doping increases from 3.09 eV to 3.12 eV. This may be due to the quantum confinement effect suggesting that decrease in particle size improves the energy band gap. On the basis of obtained result, it is clear that Zn doped cerium oxide nanomaterial could be capable for optoelectronic applications such as Dye Sensitized Solar Cells, Supercapacitor, sensors and UVshielding devices.

The effect of pure CeO_2 and Cu doped nanoparticles prepared using co-precipitation method has studied by Govindarasu et al [40]. They have studied the effect of small doping up to 5%. The change in band gap energy has observed from UV-Visible absorption spectrum of Cu doped cerium oxide nanoparticle. Degradation of the methylene blue through photocatalysis has been observed for pure and Cu doped CeO_2 nanoparticles under solar spectrum.

Using co-precipitation method Fe and Ni-doped CeO_2 nanoparticles were synthesized by Reza Zamiri et al [41] The PL properties of the samples showed quenching effect of Fe and Ni ions on emission properties of the pure CeO_2 sample. The doping of Fe and Ni deeply decreases the dielectric constant value of the pure CeO_2.

Properties and applications:
CeO_2 is widely used as a catalyst in various industrial processes, such as automotive catalytic converters due to catalytic properties. It helps in promoting the oxidation of pollutants in exhaust gases. As cerium oxide has ability to undergo reversible oxidation and reduction reactions. This redox property is utilized in fuel cells, oxygen sensors, and as an oxygen storage material in catalytic converters.

Due to its high ionic conductivity at elevated temperatures makes it useful for the manufacturing of solid oxide fuel cells. It enables the efficient conversion of chemical energy into electrical energy. Oxygen storage capacity of this material enhances its application to three-way catalytic converters in automobiles to store and release oxygen as needed during combustion reactions. More efficient and cleaner combustion of fuel can be achieved by using this material.

The fluorite crystal structure of CeO_2 makes it suitable for use as a stabilizer in certain ceramic materials. Thus the mechanical strength and thermal stability of material get enhanced, and it is used in the production of high-temperature-resistant coatings and refractory materials. Nanoparticles of cerium oxide are also used in sunscreens and cosmetic products for their ability to absorb and scatter ultraviolet (UV) radiation, providing protection against skin damage caused by the sun. Due to its antioxidant properties it may have medical applications, such as in the treatment of oxidative stress-related diseases.

CeO_2 nanoparticles also have been investigated for their photocatalytic activity, which can find applications in environmental remediation, such as the degradation of organic pollutants in water and air. The inclusion of certain transition metal ions, such as Fe, Mn, or Co, can induce magnetic behavior. This magnetic behavioru of the material leads its application to magnetic storage device, MRI, drug delivery system etc.

The dielectric properties of a material refer to its ability to store and transmit electrical energy in response to an electric field. It also possesses dielectric properties that make it useful in various

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applications like dielectric ceramic, insulating layer in electronics, gate dielectric in transistors, energy storage device, for reducing power consumption as high K-dielectric device, dielectric resonator for microwave communication systems etc. The band gap of CeO_2 makes it suitable for use in optoelectronic devices. CeO_2 can be integrated into devices such as light-emitting diodes (LEDs) and photodetectors.

CeO_2 nanoparticles are explored for in vitro biomedical applications, including drug delivery systems and imaging contrast agents. In controlled laboratory conditions, researchers investigate the interactions of CeO_2 with biological systems on a cellular and molecular level, while CeO_2 nanoparticles can be explored for in vivo biomedical imaging applications, where they may act as contrast agents for imaging modalities such as magnetic resonance imaging (MRI) or computed tomography (CT) scans with can be used for targeted drug delivery in vivo. These nanoparticles are investigated for potential applications in neurological studies, including neuro protective effects.

Conclusion:

In this review article, synthesis of CeO_2 by various chemical method has been discussed. The structural and morphological features of cerium oxide depend on many factors such as precursors, solvent, pH value, growth temperature, annealing temperature etc. The chemical routes such as co-precipitation, solgel, combustion and green synthesis route resulted in nano sized particles. The co precipitation technique is found most appropriate, as it is cost effective and allows homogenous doping. This method enables the production of nano-powders with a small and uniform crystallite size.

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