

Structural and Morphological Study of Cerium Oxide (CeO) Nanoparticles by Combustion Method

G. Vinodhini^a, K. Jenifer Asuntha^a, S. Priya^a, V. Sabari^{b*}

^aPG & Research Department of Physics, Kamban College of Arts and Science for Women, Thenmathur, Tiruvannamalai – 606 603, Tamilnadu, India.

^{b*}PG & Research Department of Physics, Marudhar Kesari Jain College for Women, Vaniyambadi, Tirupattur – 635 751, Tamilnadu, India.

ABSTRACT

The cerium oxide (CeO) nanoparticles have been successfully prepared by combustion method. The crystallite size and morphology of cerium oxide have been investigated by X-Ray diffraction (XRD), fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), energy dispersive spectrum (EDS), and transmission electron microscopy (TEM) techniques. The calculated average crystallite size was cerium oxide in 19 nm. Furthermore, cerium oxide nanoparticles have the crystallite size in the range ~14-31 nm, as confirmed by TEM. Results obtained indicate that the co-precipitation method is a promising low temperature, cheap, and fast method for the production of cerium oxide nanostructures.

Keywords: XRD; combustion method; SEM; EDS; TEM.

INTRODUCTION

In the present study was report the synthesis of cerium oxide nanoparticles using co-precipitation method and the characterization of cerium oxide nanoparticles using X-ray diffraction (XRD), transmission electron microscopy (TEM), selected area electron diffraction (SAED), scanning electron microscopy (SEM), fourier transform infrared spectroscopy (FTIR) energy dispersive spectrum (EDS) are discussed. Cerium oxide (CeO) is a white solid inorganic powder. It is non flammable, stable and insoluble in water, II-VI semiconductor with wide band gap energy that is 3.3ev and high excitation energy that is 60ev [1]. This semiconductor has several favorable properties including good transparency, high electron mobility, strong room temperature, low toxicity, luminescence and photo chemical stability and higher breakdown field strength.

General, cerium oxide crystallizes in two main form, hexagonal wurtzite and cubic zinc blende but the (B4 type) wurtzite structure is obtained only at optimum pressure and temperature [2-3]. cerium oxide crystallizes in the typical wurtzite hexagonal structure where oxygen and cerium oxide atoms are spatially arranged in a way that O atoms are arranged in a closed hexagonal structure, while the Zn atoms occupy the centre of the distorted tetrahedron structure [4]. The variety of structures of Nano metric zinc oxide means that cerium oxide can be classified among new materials with potential applications in many fields of nano technology. cerium oxide can occur in one - (1D), two - (2D), and three-dimensional (3D) structures. One dimensional structure make up the largest group, including nano rod -needles, -helixes, - springs and -rings, - ribbons, - tubes - belts - wires and -combs. Cerium oxide can be obtained in 2D structures, such as Nano plate/Nano sheet and Nano pellets. Examples of 3D structures of zinc oxide include flower, dandelion, snowflakes, coniferous urchin-like, cerium oxide provides one of the greatest assortments of varied particle structures among all known materials [5-6]. The large specific surface area high pore volume, nano structured properties, low cost and low toxicity of nano cerium oxide [7] make it a promising candidate, particularly in catalysts [8], photo catalysis, electrostatic dissipative coating, transparent UV protection films, and chemical sensors [9-12], gas sensor, solar cells. Moreover, cerium oxide nanoparticles have a tremendous potential in biological applications like biological sensing, biological labeling, gene delivery, drug delivery and nano-medicine [13-22]. In recent years, noble metal oxide nanoparticles have been the subject of focused research due to their unique electronic optical, mechanical, magnetic and chemical properties. Cerium oxide is the topic of interest in these days due to its presence much unique and important morphology likes nanorods, nanoflowers, nanowires, nano dendrites and nanoparticles.

EXPERIMENTAL PROCEDURE

Materials

Cerium nitrate ($\text{Ce}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$) and ammonia solution of analytical grade (SD fine chemicals, 98.5%) were used as such without further purification for synthesis process. Double distilled water was used through the experiments.

Synthesis of Cerium Oxide Nanopowder

CeO samples have been prepared by using a starting solution of zinc nitrate with 0.1 M concentration diluted in deionized water, Cerium nitrate ($\text{Ce}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$) salt used as dopant source is added with a small amount in the starting solution. The doping level is varied by changing the weight ratio [Ce], in the solution. Then, NH_3 was added, under constant stirring conditions, up to at the pH level of 8. The stirred mixture was irradiated by the microwave radiation of frequency 2.45GHZ, for 5 minutes continuously. The precipitates were collected and, washed with distilled water for several times until the extracts turns into a white product. The final product was annealed at 300 °C for 4 hours.

Characterization

The resulting powders were analyzed by X-ray diffraction (XRD) using a Bruker AXS D8 Advance instrument diffractometer with monochromatic $\text{CuK}\alpha 1$ wavelength of 1.5406 Å. The samples morphology was observed by scanning electron microscopy (SEM), using a JEOL 5600LV microscope at an accelerating voltage of 10 kV. The microstructure was studied by transmission electron microscopy (TEM) and selected-area electron diffraction (SAED) in a Tecnai G20-stwin operated at 200 kV. The Fourier transform infrared spectra (FT-IR) of the samples were recorded by using a Nicolet 5DX FTIR spectrometer.

RESULTS AND DISCUSSION

X-Ray Diffraction (XRD)

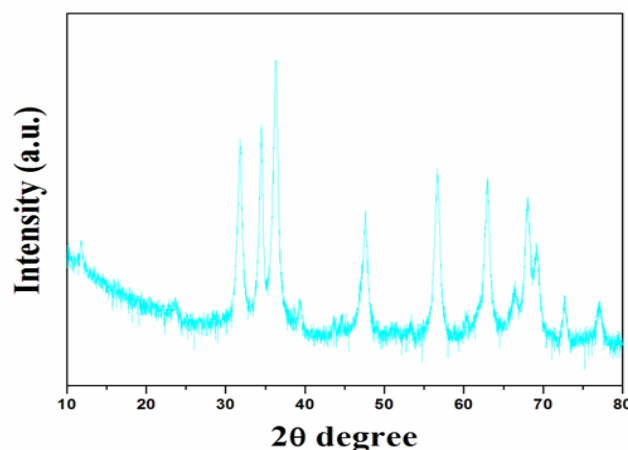


Fig.1: X-ray diffraction patterns of CeO were annealed temperature at 300°C.

The synthesized CeO is particles were subjected to valuable studies to understand their structure, morphology size and other properties. The results obtained from those studies are discussed in this chapter. X-ray diffraction pattern was studied to determine the structure of the as synthesized nanomaterials cerium oxide. Fig. 1 depicts on the XRD pattern to determine the phase composition of the sample annealed temperature at 300°C. The sample shows the diffraction peaks of crystalline CeO in its characteristic cubic structure (JCPDS, No. 89-9066). The average crystalline size of the crystallites was evaluated using the scherrer's formula,

$$D = \frac{K\lambda}{\beta \cos \theta}$$

Where D is the crystalline size (nm), K is a grain shape dependent constant (0.9), λ is the wavelength (1.5406 nm) of the incident beam, θ is a Bragg reflection angle and β is the full width at half maximum (FWHM) of the main diffraction peak. As estimated from the width at half-maximum of the main diffraction peak according to the Scherer equation, are about 24 nm for the annealed temperature at 300°C. The calculated unit cell parameters are $a = 5.145 \text{ \AA}$, $b = 5.205 \text{ \AA}$, and $c = 5.312 \text{ \AA}$.

Fourier Transform Infra-Red Spectroscopy (FT-IR)

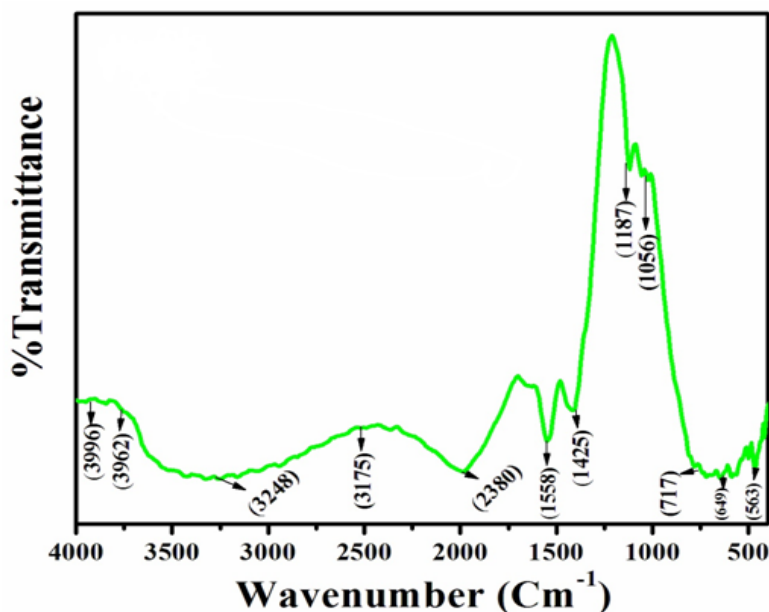


Fig.2 FTIR spectra of CeO₂ were annealed temperature at 300°C.

FT-IR spectrum was annealed temperature at 300°C. The IR spectroscopy was used to learn the surface interactions of the adsorbed water in dynamic symmetry with the gas phase on the CeO surface. Frequency shifts and absorbance values were carefully observed to interpret the surface structure of the CeO₂ phase. It is well-known that H₂O and CO₂ molecules are simply chemisorbed onto the CeO₂ surface as soon as exposed to the ambience.

CeO Nanoparticles Wave number	Assignment
563 cm ⁻¹ - 717 cm ⁻¹	CeO stretching frequency of Ce-O bond
3175 cm ⁻¹ - 3996 cm ⁻¹	water re-absorption during the storage of the sample
1425 cm ⁻¹ 1558 cm ⁻¹	C=O stretching vibrational modes C=C stretching vibrational modes
2380 cm ⁻¹	symmetric C-H bonds

Scanning electron microscopy (SEM)

The morphology of the synthesized nanocrystalline was analyzed by scanning electron microscope (SEM). The presence of the agglomerated with crystalline nature with composed by the agglomeration and non agglomeration of the smaller crystallites. Consequently the temperature distribution is homogeneous and is transferred to the materials interior, making an explosive reaction followed by vigorous evolution of the gases to form CeO with good polycrystalline nature.

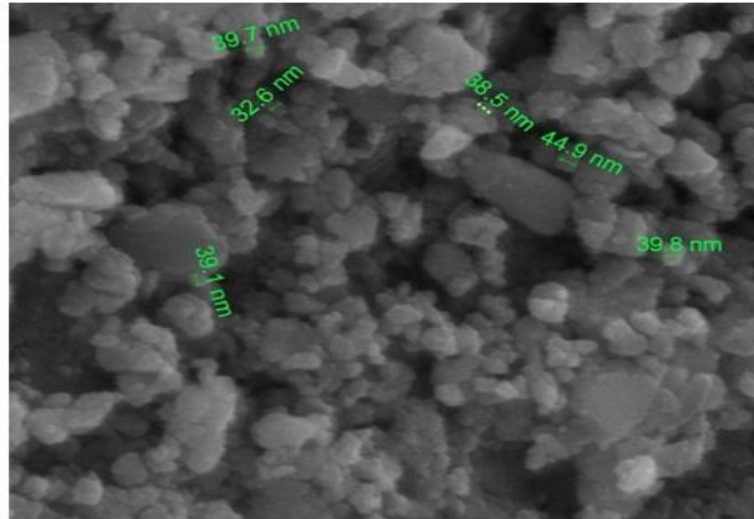


Fig.3. Scanning electron microscope of CeO annealed temperature at 300°C.

Energy dispersive spectrum (EDS)

The composition of the obtained CeO was analyzed by means of energy dispersive spectrum (EDS) as shown in Fig. 4. The EDS result showed the presence of Ce and O by the appearance of Ce and O peaks.

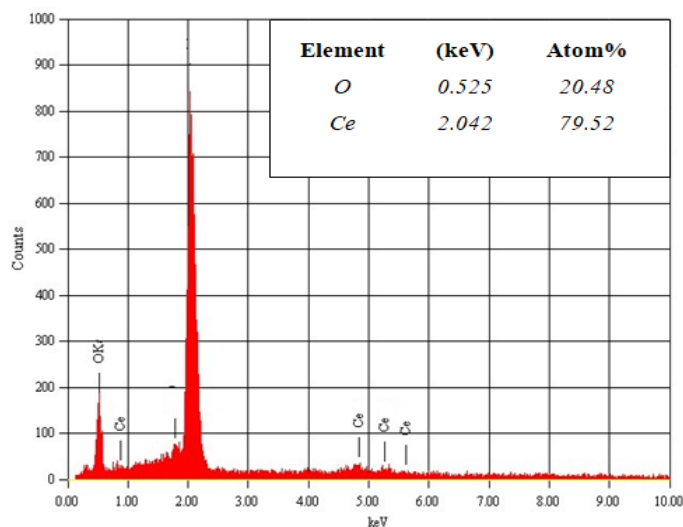


Fig.4. Energy dispersive spectrum of CeO annealed temperature at 300°C.

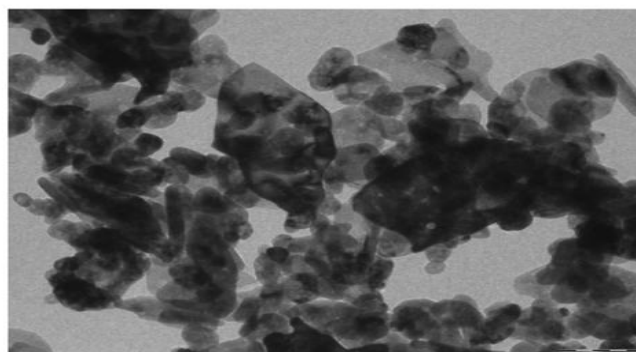


Fig.5. Transmission electron microscope of CeO annealed temperature at 300°C.

The TEM image presence of the agglomerated with crystalline nature with composed by the agglomeration and non agglomeration of the smaller crystallites. Consequently the temperature distribution is homogeneous. The Transmission electron microscope of CeO annealed temperature at 300°C and the average particle size is 21 – 36 nm.

CONCLUSION

The CeO Nanoparticles synthesized by combustion method have been investigated. The sample have been investigates to the annealed at 300°C temperature. The X-ray diffraction pattern of the nanocrystalline CeO possessed monoclinic structure with average crystallite size in the range of 24 nm for annealed temperature at 300°C. The FT-IR spectra of CeO are well-known that H₂O and CO₂ molecules are simply chemisorbed onto the CeO surface as soon as exposed to the ambience. SEM results are presence of the agglomerated with crystalline nature with composed by the agglomeration and non agglomeration of the smaller crystallites. The EDS result showed the presence of Ce and O by the appearance of Ce and O peaks. This is the simple synthesis method was using for optical and gas sensor applications.

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