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# **Cerium oxide (CeO2) nanoparticles: synthesis, characteristics, and properties using the combustion technique**

**Yasmin A. Hosseny<sup>a</sup> , Mostafa Mahmoud<sup>a</sup> and Mostafa Y. Nassara,b\***

<sup>a</sup> Department of Chemistry, Faculty of Science, Benha University, Benha13518, Egypt <sup>b</sup> Department of Chemistry, College of Science, King Faisal University, Al-Ahsa 31982, Saudi Arabia \***Corresponding author: Mostafa\_y\_Nassar@yahoo.com**

### **Abstract**

 This paper reports a study on preparation of nanocrystalline ceria powder by solution combustion technique that is a simple and eco-friendly process, to take a glycine as a fuel and ammonium ceric nitrate as an oxidizer and followed by calcination at 500 ˚C for 2 h. The obtained product was characterized by various techniques and the results are presented and discussed. X-ray diffraction data confirms a cubic phase of cerium oxide with crystallite size was found to be 34.9 nm. The infrared spectrum shows the peak at ca.  $(413 \text{ cm}^{-1})$  is corresponding to the asymmetric (O-Ce-O) stretching vibration inside  $CeO<sub>2</sub>$  lattice.

**Keywords:** Cerium, Nanoparticles, Combustion, crystallite size

# **1. INTRODUCTION**

 Nanotechnology in science and engineering are really a recent revolutionary development that is growing at a very rapid rate [\[1\]](#page-4-0). Nanoparticles are particles between 1 and 100 nanometers in size. When the dimensions of a material are reduced below 100 nm, dramatic alterations may occur in their properties [\[2\]](#page-4-1). Materials therefore might be nanostructured in order to provide a specific performance or to provide new properties to a material in addition to changes linked specifically to size and structure [\[3\]](#page-5-0).

 The nanoparticles exhibit a unique physical, chemical and biological properties at the nanoscale may be quite different from those within a bulk material [\[4\]](#page-5-1) compared to their respective particles at higher scales and can be exploited for commercial applications and for novel performance that benefits society [\[5\]](#page-5-2). The integration of nanotechnology into larger systems has provided breakthrough solutions to many current environmental, medical [\[6\]](#page-5-3) and industrial problems, including smart materials, nanomanufacturing, electronics, drug delivery, energy and water, biotechnology, information technology, and

national security [\[7\]](#page-5-4). Nanotechnology will have a profound effect on our economy and society; it is a modern industrial revolution. There are different types of nano-sized structures have been developed to advance nanotechnology strategies including nanorods, nanowires, nanotubes, nanobelts, nanoribbons, nanofibers, nanoparticles, quantum dots and hollow spheres and compositions (organics, metals, oxides and semiconductors) [\[1\]](#page-4-0). Chemistry has played a key role in the development of nanoscience. Making and breaking bonds between atoms or groups of atoms is a fundamental component of chemistry.

 Metal oxides are an important class of inorganic nanoparticle due to their optical, magnetic, and conductance, electronic properties and chemical interactions. Among them, cerium oxide is one of the most important compounds [\[8\]](#page-5-5).

 Cerium oxide nanoparticles have gained a lot of attention as a potential future candidate for ending various kinds of problems by exhibiting redox activity, free radical scavenging property [\[9\]](#page-5-6), biofilm inhibition, high surface area, increased reactivity or stability in a chemical process, enhanced mechanical strength and high thermal resistance compared to bulk equivalents. etc [\[10\]](#page-5-7). Owing to their redox properties, switching between  $Ce^{+4}$  and  $Ce^{+3}$ , oxygen vacancies and high surface area, they exhibit excellent photocatalytic activity [\[11\]](#page-5-8). The enhanced photocatalytic activity of cerium oxide nanoparticles was ascribed to the significant suppression of the recombination rate of photogenerated electron-hole pairs due to charge transfer

and increasing oxygen vacancy [\[9\]](#page-5-6) and the smaller optical band gap  $(-3.1 \text{ eV})$  [\[12\]](#page-5-9). Cerium is one of the most reactive a lanthanide series rare earth metal oxides and exists as a free metal [\[13\]](#page-5-10). Bulk ceria has at least two stable stoichiometry forms, the cerium dioxide  $(CeO<sub>2</sub>)$  fluorite-type structure and dicerium trioxide  $(Ce<sub>2</sub>O<sub>3</sub>)$ hexagonal structure type having the cerium ions the respective oxidation states  $Ce^{+4}$  and  $Ce^{+3}$  [\[14\]](#page-5-11). At the nanoscale, the Cerium oxide lattice has a cubic fluorite structure and therefore exhibits antioxidant properties [\[15\]](#page-5-12). therefore, at nanoscale, cerium oxide nanoparticles contain intrinsic oxygen defects. These oxygen defects are actually 'hot spots' of catalytic reaction. The concentration of oxygen defects increases with a reduction in particle size. So, cerium oxide nanoparticles  $(CeO<sub>2</sub>)$  is widely used in various applications like act as catalyst [\[16\]](#page-5-13), optical materials, polishing agents [\[17\]](#page-5-14), sunscreens, solid electrolytes, photocatalysis, sensors, oxygen pumps [\[18\]](#page-5-15), solar cells, fuel cells, electrochromic thinfilm, phosphorescent/luminescent materials and biomedical applications [\[19\]](#page-6-0).

 Synthesis of cerium oxide nanoparticles have been the subject of numerous studies in the last decade. Synthesis of cerium oxide nanoparticles can be performed very easily by utilizing chemical, physical or biological methods [\[20\]](#page-6-1), different approaches including sol- gel [\[21\]](#page-6-2), ball milling, solid state reactions, chemical reduction, coprecipitation [\[22\]](#page-6-3), seeding microemulsion [\[23\]](#page-6-4), electrochemical synthesis, precursor thermal decomposition, hydrothermal synthesis [\[24\]](#page-6-5) and combustion method [\[12\]](#page-5-9).

 There is a relation between beginning materials, processing and prepared method, microstructure, structure and properties of the final product. As expected, different synthesis methods may lead to various structures, morphologies, properties and diverse potential applications [\[12\]](#page-5-9). The preparation and characterization of uniform oxide nanostructures has attracted much attention for their properties, which are much different from those of bulk materials.

 On the other hand, solution combustion method was involved in preparing cerium oxide nanoparticles as an easy, fast, short time, simplicity, the low energy input and inexpensive technique can produce nanosized powders due to the lack of time for grain (crystallite) growth [\[16\]](#page-5-13).

 The aim of the present work was the synthesis of cerium nanoparticles by solution combustion method. The effect of fuel type on chemical composition, structure, microstructure and band gap of synthesized particles are also investigated.

# **2. Materials and Methods**

### *2.1. Chemicals and reagents*

 Ammonium ceric nitrate 99% was provided from LOBA Chemie PVT.LTD. Glycine was purchased from EL-Nasr Pharmaceutical Chemicals Company, Egypt. All chemicals of analytical grade were utilized as received without further purification. Deionized water was used for the present work.

# **2.2. Synthesis of CeO2 nanoparticles using glycine fuel**

 Dissolving (9.998 mmol, 5.48 gm) of ammonium ceric nitrate in 25 ml distill water, then added (26.6 mmol, 2 gm) of glycine. The obtained product (Ce-G-500) was magnetically stirred at room temperature until a homogenous solution was formed. Afterwards, the solution was subjected to combustion at 300 ˚C on a hot plate for 15 min, washing through centrifugation at 2000 rpm for 5 min and dehydration at 120 ˚C for 3 h. The obtained powders were calcined at 500 ˚C for 2 h that enhances the formation of pure cerium oxide nanoparticles with crystalline nature.

### *2.3. Instrumental Methods*

 Cerium oxide nanoparticles (Ce-G-500) was characterized by using X-ray diffractometer (Bruker; model D8 Advance, 40 kv 40 mA) with monochromatic Cu / kα radiation ( $\lambda$ =1.54178 °A) in the angular rang of  $(3°-80°)$  with step size  $0.02°(2\Theta)$  and scan step time 0.4 (s). The functional groups were indicated by using Fourier–Transform infrared (FTIR) spectrometer (Thermo scientific; model iS10, Germany) from 4000 to  $400 \text{ cm}^{-1}$ .

# **3. Result and discussion**

# *3.1. X-ray diffraction study*

 Fig (1) shows the XRD pattern of cerium oxide nanoparticles synthesized using glycine fuel to study the phase composition. XRD pattern showed that the obtained samples were formed after the calcination at 500 ˚C for 2 h. XRD pattern showed the diffraction peaks at  $2\Theta$  values of 28.68˚, 33.19˚, 47.62˚, 56.45˚, 59.28˚,

69.39˚, 76.75˚ and 79.38˚ which correspond to the planes of (111), (200), (220), (311), (222), (400) and (331) in the form of cubic fluorite structure of  $CeO<sub>2</sub>$  nanoparticles (reference card No 00-043-1002). The average crystallite size (D, nm) of the synthesized cerium oxide nanoparticles can be evaluated by using the following equation (1).

$$
D = K \lambda / \beta \cos \Theta \tag{1}
$$

Where;  $\lambda$  (nm) is the wavelength of the X-ray radiation, β is the diffraction peak full wide at half maximum (FWHM) and  $\Theta$  is the Bragg diffraction angle. The average crystallite sizes  $(D, \text{nm})$  for  $CeO<sub>2</sub>$ nanoparticles are found to be 34.9 nm.



Fig (1) XRD patterns of CeO<sub>2</sub> nanoparticles; Ce-G-500 prepared by combustion method using glycine fuel and calcined at 500 ˚C for 2 h.

#### *3.2. FT-IR spectrum*

 According to Fig (2) the FT-IR spectra of cerium oxide nanoparticles after calcination at 500 ˚C for 2 h was recorded in the range  $400-4000$  cm<sup>-1</sup>. The bending and stretching vibrational bands of the adsorbed water molecules appeared at ca. (1624 and  $3420 \text{ cm}^{-1}$ ), respectively. Moreover, the peaks at ca.  $(2920 \text{ and } 2849 \text{ cm}^{-1})$ , may be attributed to (C-O) stretching vibration due to the formation of carbonate-like groups on the  $CeO<sub>2</sub>$  surface. In addition, the peak at ca.  $(1339 \text{ cm}^{-1})$  is due to (Ce-O-Ce) stretching vibration and the peak at ca.  $(1156 \text{ cm}^{-1})$  is responsible for the overtone band of the trace of Ce-OH stretching vibration. Absorption band at ca.  $(413 \text{ cm}^{-1})$  is corresponding to the symmetric (O-Ce-O) stretching vibration inside  $CeO<sub>2</sub>$  lattice.



Fig (2) FT-IR spectra of CeO<sub>2</sub> nanoparticles; Ce-G-500 prepared by combustion method using glycine fuel and calcined at 500 ˚C for 2 h.

### **4. conclusion**

 Nanoparticles of cerium dioxide have been synthesized by combustion solution method using ammonium ceric nitrate as the precursor and glycine as a fuel. Powder XRD studies reveal the crystal structure and nano-scale of the prepared cerium oxide nanoparticles with average crystallite size 34.9 nm. The functional groups of the sample have been identified from FT-IR studies.

#### **Data availability**

No data was used for the research described in the article.

#### **Declaration of competing interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### **Institutional Review Board Statement**

This study was conducted and approved according to the guidelines of the declaration of the ethical committee of the Faculty of Science, Benha University (BUFS-REC-2024-198 Chm)

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