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Synthesis and Characterization of Cerium Oxide Nanoparticles by Hydroxide Mediated Approach

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Abstract: Cerium oxide nanoparticles or nanoceria were synthesized by hydroxide mediated approach using cerium nitrate hexahydrate ($Ce(NO_3)_3.6H_2O$ and sodium hydroxide (NaOH) as precursors. Structural and morphological studies of the cerium oxide nanoparticles were carried out using X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM). XRD pattern confirmed the polycrystalline nature of the cerium nanoparticles with face centered cubic structure. Crystallite size was calculated using Debye Scherrer formula and the size was found to be in the range of 9-16 nm. SEM studies revealed the formation of nanosized spherical particles around 18-30.4 nm. The absorption band at 550.84 cm^{-1} (Ce-O stretch) in FTIR spectrum confirmed the formation of cerium oxide nanoparticles. Optical studies were carried out using UV-Visible absorbance spectrophotometry and a well defined absorbance peak was observed around 325 nm.

Key words: Cerium oxide, polycrystallinity, Debye Scherrer, Ce-O stretch

INTRODUCTION

Ceria or ceriuim oxide is an excellent semiconducting material with a wide band gap which is familiar for its catalytic properties (Trovarelli *et al*., 1997). Cerium, one of the elements in lanthanide series exhibit both oxidation states +3 and +4 and has the capacity to switch over these oxidation states very easily. Because of this excellent property cerium oxide is used in various applications such as catalytic converters, solid oxide fuel cells (Laberty-Robert *et al*., 1997) and oxygen buffers.

Nowadays nanocrystalline cerium oxide particles are studied due to its various applications. These cerium nanoparticles are used as UV absorbents and filters (Hu *et al*., 2006). Nanoceria is now extensively used in sensing applications especially in gas sensors (Jalilpour and Fathalilou, 2012) and electrochemical sensors. In electrochemical biosensors the cerium oxide nanoparticles are used as nanointerface to improve the basic characteristics of the sensors such as response time, sensitivity, selectivity, linear response. In these sensors the cerium oxide nanoparticles are used as the interface between the electrode and the biological element. The presence of these nanoceria as interface in these sensors increases the electron transfer rate between the electrode and the recognition element. Hence, the basic characteristics of the sensors improved.

Cerium oxide nanoparticles have wide range of biological and biomedical applications. The Reactive Oxygen Species (ROS) is responsible for number of diseases (e.g., vision loss). The specific redox properties of the cerium oxide nanoparticles make it interact with a large number of ROS and minimize their harmful effects. The introduction of the cerium oxide nanoparticles into the in-vitro cell culture increases the life span of the cells and decreases the cell damage caused by H_2O_2 (ROS).

A recent study revealed the effective role of cerium oxide nanoparticles in spinal cord damage and some diseases related to the Central Nervous System (CNS) due to oxidative stress. The presence of the cerium nanoparticles increased the life span of the neurons in the spinal cord and in the central nervous system. Because of the wide range of applications of the cerium oxide nanoparticles it was synthesized by hydroxide mediated approach and characterized.

MATERIALS AND METHODS

Cerium oxide nanoparticles are prepared by several methods such as hydrothermal, sol-gel, flame spray

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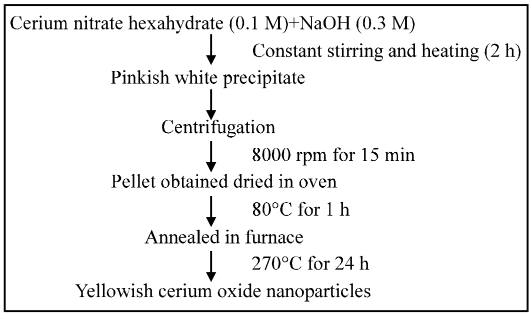

Fig. 1: Preparation of cerium oxide nanoparticles

pyrolysis, solvothermal methods etc. These methods depend on high temperature, pressure and the use of capping agents. We have developed a simple and novel method for the synthesis of cerium oxide nanoparticles. The precursor for the production of the cerium oxide nanoparticles by hydroxide mediated method was cerium nitrate and sodium hydroxide. 0.1 M cerium nitrate solution and the 0.3 M NaOH solution was prepared using double distilled water. NaOH solution was added dropwise to the precursor solution for about 2-3 h at room temperature under constant stirring. A pinkish white precipitate was obtained. The precipitate was centrifuged at 8000 rpm for 15 min and the pellet was collected by discarding the supernatant. The pellet was washed several times with distilled water and once with ethanol. The pellet was then dried at 80°C for 1 h in hot air oven and then annealed at 270°C (Fig. 1).

RESULTS AND DISCUSSION

Structural and morphological studies: The structural characterization, phase identification and the grain size of the cerium oxide nanoparticles were studied by X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).

Flow chart for the synthesis of cerium oxide nanoparticles

X-ray diffraction pattern of cerium oxide nanoparticles: The XRD pattern of CeO_2 nanoparticles is shown in Fig. 2. The XRD pattern was scanned from 10-80 degrees with the scan rate 2θ min^{-1}. The XRD profile confirmed the polycrystalline nature of the cerium oxide nanoparticles. The high intensity peaks were observed at 28.53, 33.09, 47.5, 56.26, respective to the 111, 200, 220, 311 crystal planes. The crystal planes were in well accordance with JCPDS No: 34-0394 of CeO_2 crystal. The diffraction peaks in these XRD spectra indicates the pure cubic

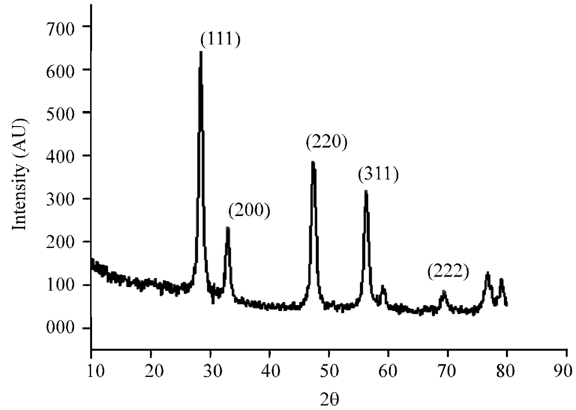

Fig. 2: XRD pattern of the CeO_2 nanoparticles

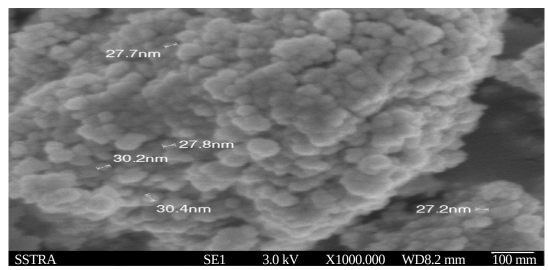

Fig. 3: SEM of cerium oxide nanoparticles

fluorite structure. Crystallite size was obtained by using the Debye's Scherrer equation:

$$D = \frac{K}{\beta \cos(\theta)}$$

where, K is the shape factor, λ is the X-ray wavelength, β is the line broadening at half the maximum intensity (FWHM) in radians and θ is the Bragg angle. The crystallite size was found to be in the range from 9-16 nm.

SEM of cerium oxide nanoparticles: SEM of cerium oxide nanoparticles is shown in Fig. 3. Surface and morphological characterization of cerium oxide nanoparticles were carried out using scanning electron microscopy. Nanosized spherical shaped CeO_2 particles obtained was confirmed. The mean size of the particles varies from 18- 30.4 nm.

Fourier transform infra-red spectroscopy of cerium oxide nanoparticles: The FTIR spectrum of cerium oxide nanoparticles is shown in Fig. 4. The spectrum was recorded in the wave number range of 400-4000 cm^{-1}. The

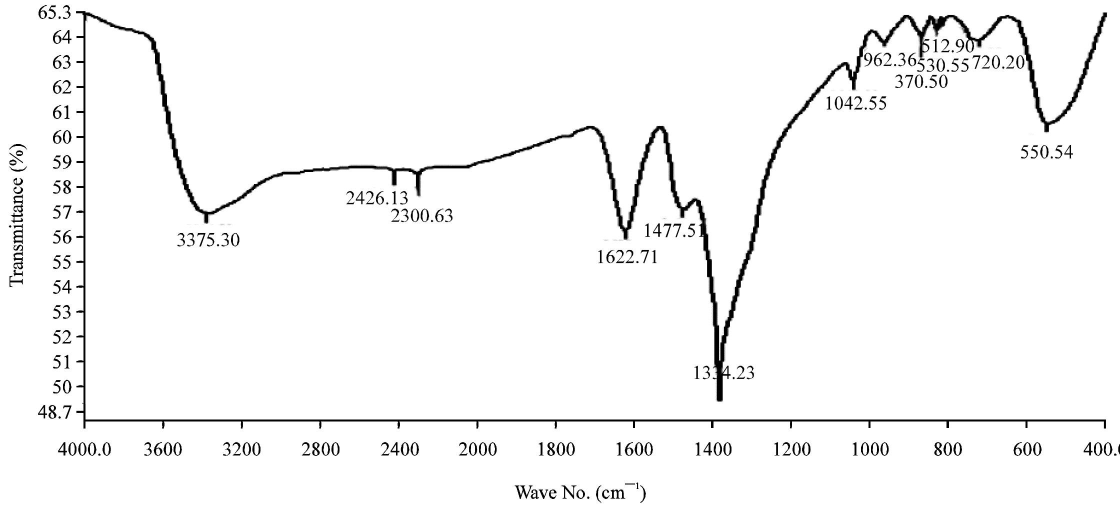

Fig. 4: FTIR spectrum of cerium oxide nanoparticles

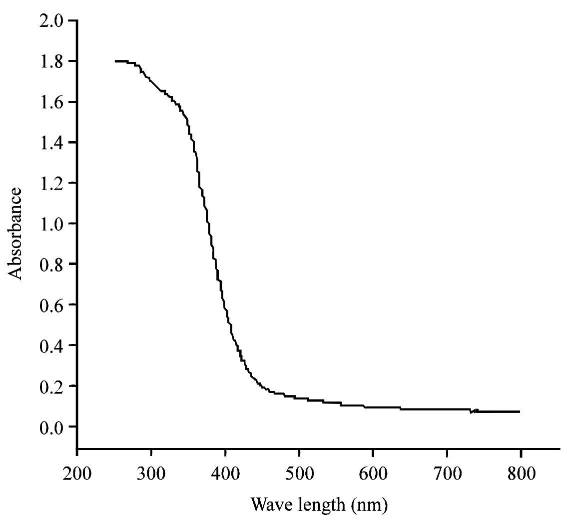

Fig. 5: UV-visible absorbance spectra of cerium oxide nanoparticles

bands at 1622.71 and 3375.80 cm^{-1} represents the water and the hydroxyl stretches, respectively. The intensive band at 1384.23 cm^{-1} represents N-O stretch due to the presence of nitrate. The absorption band at wavenumber 550.84 cm^{-1} represents the Ce-O stretch (Ansari *et al*., 2009). We confirmed the formation of nanoparticles using FTIR spectrum.

Optical studies: A UV-visible absorbance spectrum of cerium oxide nanoparticles is shown in Fig. 5. The absorbance spectra was recorded for the nanoparticles dispersed in water. Here, water is used as a blank. A strong absorption below 400 nm was observed. A very well defined absorbance peak was observed around 325 nm for the synthesized cerium oxide nanoparticles.

CONCLUSION

The cerium oxide nanoparticles were synthesized by hydroxide mediated method. XRD pattern of CeO_2 particles shows that the particles were in polycrystalline in nature. The SE micrograph shows the particles are in spherical shape with size range of 18-30.4 nm. The FTIR spectrum confirmed the formation of CeO_2 particles. The optical studies of CeO_2 particles were studied by UV-visible absorbance spectrophotometry. Thus the cerium oxide nanoparticles were synthesized and characterized.

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