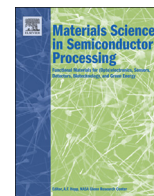




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Synthesis and characterization of ceria quantum dots using effective surfactants



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ABSTRACT

The stable and crystalline phase of different surfactants (CTAB, PEG and SDS) capped CeO₂ nanoparticles were directly synthesized by chemical precipitation method at room temperature. The effects of surfactants on the structural and optical properties of nanoparticles are characterized. The optical properties of the nanoparticles were investigated by UV-visible and PL spectroscopy. The effects of surfactants with observed band shifts are due to quantum confinement effect. The optical band gap values are determined by simple energy wave equation and Tauc plot method. The observed particle sizes are very closer to the Bohr excitonic radius. The emission bands such as violet, blue, green and orange are observed in PL spectra. The PL integrated intensity ratio of the UV emission to the deep-level green emission (I_{UV}/I_{DLE}) for CTAB, PEG and SDS capped CeO₂ nanoparticles are observed. The XRD measurement shows that CeO₂ has cubic fluorite structure having the particle size 6–10 nm. The lattice strains were detected by Williamson–Hall plot method. The surface morphology of the nanoparticles is studied by SEM and FESEM analysis. TEM images show that the particles are nearly spherical in shape with diameter of 5–10 nm. Using FTIR spectra, the functional groups of the ceria are identified.

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1. Introduction

Ceria (CeO₂) is one of the most reactive rare earth metal oxides. It possesses excellent physical and chemical properties. It is having important applications in the fields of electrochemistry, catalysis, photochemistry, and materials science [1]. Numerous techniques have been proposed by several workers to synthesize CeO₂ materials including decomposition of oxalate precursors [2], hydrothermal synthesis [3], sol-gel [4], microemulsion [5] and coprecipitation [6] methods, in order to improve performance. So far lot of efforts have been taken by various researchers towards the morphology controllable synthesis of ceria nanostructures. This provides new opportunities for designing CeO₂ nanomaterials with desired properties. The various CeO₂ nanostructures such as nanoparticles, nanotubes, nanorods, nanocubes, nanospheres, and nanoplates have been developed by several workers [7–11]. These nanomaterials exhibit superior properties than to their bulk [1]. The spherical and one-dimensional nanostructure of ceria exhibits excellent physicochemical properties, resulting in its important applications as three-way catalysts in electrolyte materials for ultraviolet-blocking materials, vehicle emission control systems, solid oxide fuel cells, conversion catalysts, sunscreen cosmetics,

solar cell gates for metal-oxide semiconductor devices, polishing materials, etc. Motivated through various applications and the excellent properties, the development of effective methods to synthesize nanostructures with size/shape control is one of the key trends in materials science [12]. In the past years, the synthesis of size/shape controlled CeO₂ nanoparticles accompanied by the investigation of their size/shape dependent properties have been carried out by several research groups [13–17].

However, these methods have defects in the complex process and expensive raw materials, or require very stringent control of various processing parameters, together with a low production yield, complicated manufacture and high energy consumption. These methods are not environmentally friendly behavior. The cost of the production is very high and it is difficult to be realized at an industrial scale. To overcome these problems, the simple and low cost route to synthesize of nanostructures has been achieved via surfactant-templated synthesis. Cationic, anionic, and nonionic surfactants can play an important role in synthesizing the nano-material in different interesting morphologies. It can be used to control the size, shape, and agglomeration among the particles [18]. Tunusoglu et al., [19] have reported the synthesized cationic surfactant (CTAB) assisted nanocrystalline CeO₂ using organophilic method. Ketzial et al., [20] have reported the formation of non-ionic surfactant (PEG) assisted CeO₂ nanostructures using chemical precipitation method. Das et al., [21] reported the details of the preparation of anionic surfactant (SDS) assisted gold nano particles

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in aqueous medium.

The high efficiency luminescence quantum dots (QDs) with narrow size distribution have attracted much attention of researchers, since these are used in wide applications in the areas of biological fluorescence imaging and labeling [22]. A key property of ceria NPs is their ability to switch effectively between +3 and +4 oxidation states. In particular, the oxidation state observed for NPs is influenced by the processing conditions and synthesis, environment storage and time (aging). These influence physical (and chemical) properties of CeO₂ NPs including optical absorption and lattice parameter [23,24]. Kuchibhatla et al. [25] and Masui et al. [6] reported the blue shift of CeO₂ optical absorption with decreasing particle size. These are consistent with the exciton confinement (quantum size effect). This leads to blue shift in optical transmission spectra, widely observed in the semiconductors and some oxide NPs. The literature on quantum confinement effects in ceria has been inconsistent and stimulated a lively debate.

In the present work, different surfactants (CTAB, PEG and SDS) capped CeO₂ nanoparticles were synthesized by precipitation process. The optical, structural and morphological analysis of CeO₂ nanoparticles were investigated using UV–visible and PL, XRD, SEM, TEM and FTIR techniques.

2. Experimental

2.1. Materials

To synthesize of CeO₂ and surfactant capped CeO₂, the following materials were used such as cerium nitrate hexa hydrate Ce(NO₃)₃·6H₂O, sodium hydroxide (NaOH), polyethylene glycol (PEG-5000-7000), acetone, obtained from Nice chemical company. Pvt. Ltd and Sodium dodecyl sulfate (SDS) and Cetyl trimethyl ammonium bromide (CTAB) obtained from s-d fine chem. Pvt Ltd were used. All the glasswares used in this experimental work were acid washed. The chemical reagents used were analytical reagent grade without further purification. Ultrapure water was used for all dilution and sample preparation. All the chemicals are above 99% purity.

2.2. Methods

A flow chart of the overall synthesis procedure is shown in Fig. 1. Initially 0.1 M of cerium nitrate. Hexa hydrate (Ce(NO₃)₃·6H₂O) in 50 ml of water. The mixture was stirred magnetically at room temperature until a homogeneous solution was

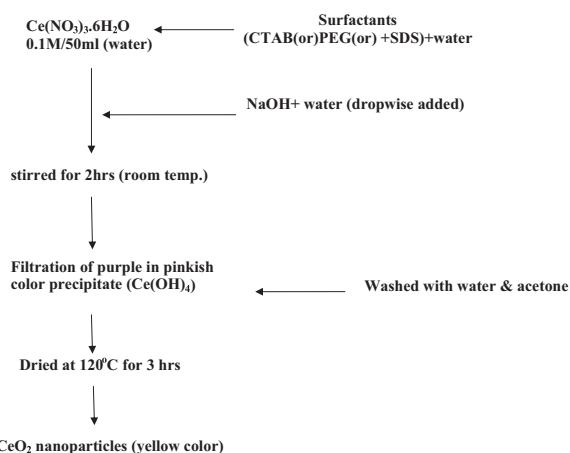
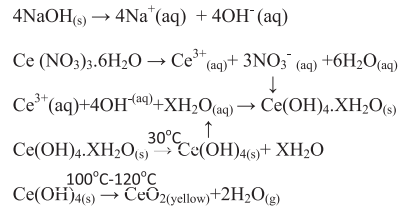


Fig. 1. shows the overall synthesis procedure for CTAB, PEG and SDS capped CeO₂ nanoparticles.

obtained. When the mixing solution became homogeneous, 0.5 g of CTAB in 50 ml of water was added slowly to the above solution and mixed for 5 min. After that, 0.2 M of NaOH in 50 ml of water was added drop wise in to the above mixture solution. The pinkish color precipitate was appeared during the addition of NaOH solution. The appeared precipitate was stirred at room temperature for 2 h. The resultant precipitant (Ce(OH)₄) was filtered, and then washed with deionized water and acetone for several times. Then the precipitant was dried at 120 °C for 2–3 h. During the period of 2–3 h, the pinkish color is changed in to pure yellow CeO₂. Then the sample was powdered in agate mortar. The same procedure was followed for synthesis of PEG and SDS capped CeO₂ nanoparticles. The main reaction occurring during the experimental procedure can be written as follows [26].



2.3. Characterization techniques

The optical absorption spectra of the samples were recorded by UV-1650 PC SHIMADZU spectrometer. Fluorescence measurements were performed on RF-5301 PC spectrophotometer. Using X pert PRO diffractometer with CuK_α radiation ($K_{\alpha}=1.5406 \text{ \AA}$), the X-ray diffraction (XRD) patterns of the powdered samples were recorded. During the recording of the diffractogram, a narrow slit of 0.1 mm was used with a scanning speed of 0.02 /s. The sample was firmly pressed into an aluminium holder, with an area of 10 mm. The sample was scanned from 20° to 80° at a scan step time of 10.3356 (s). Scherer's equation ($D=k\lambda/\beta\cos\theta$) is used to calculate the crystallite size [27]. The morphology of the nanoparticles is analysed using scanning electron microscopy (SEM; JEOL-JSM-5610 LV). Specimen for SEM was prepared with required quantity of powder sample in ethanol through ultrasonication and keeping suspension dispersion. The FTIR spectra groups were recorded by SHIMADZU-8400 Fourier transform infrared (FTIR) spectrometer.

3. Results and discussion

3.1. Optical study

3.1.1. UV–visible spectroscopy

In general, the UV absorption is related to the electronic transition from filled valence states to empty conduction states. In this study, the absorbance of samples was measured as a function of wavelength in the range of 230–600 nm and all samples showed a highly transparent mode in a visible region.

The UV–visible absorption spectra of cerium oxide nanoparticles capped with different surfactants (CTAB, PEG and SDS) are shown in Fig. 2. The absorption maxima of CTAB capped CeO₂ a nanoparticle is observed at 265 nm in the UV-region. This is due to the charge–transfer transition from O²⁻(2p) to Ce⁴⁺(4f) orbitals in CeO₂ (shown inset in Fig. 2) and indicate that this material is suitable for UV blocking and shielding material [28]. This band is shifted to higher wavelength side for PEG and SDS capped CeO₂ nanoparticles (290 and 288 nm respectively) compared to CTAB. However the absorption bands of all the capped CeO₂ nanoparticles are blue shifted compared to bulk 388 nm [29].

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