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INVESTIGATION ON THE PREPARATION AND PROPERTIES OF NANOSTRUCTURED CERIUM DI-OXIDE (CEO_2)

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ABSTRACT

Cerium di-oxide (CeO_2) nanoparticles were successfully synthesized by the microwave assisted method. The synthesized CeO_2 nanoparticles were characterized by Powder X-Ray diffraction (XRD), Scanning Electron Microscopy (SEM), UV-Vis spectroscopy and FTIR spectroscopy. The structure and morphology of the prepared samples were investigated using XRD and SEM. The XRD studies reveal that the ceria nanoparticles have face-centered cubic structure. The SEM images reveal that the prepared Ceria nanoparticles are an aggregated form of spherical nanoparticles .FTIR analysis confirms the presence of CeO2 in the prepared samples. UV-Vis spectral studies show that the UV cut off wavelength decreases and the optical band gap increases The results of synthesis and various studies of ceria nanoparticles are presented and then discussed.

I INTRODUCTION

In recent years, Cerium oxide (CeO2) has been extensively studied because it is a rare –earth oxide material and also its applications in high temperature ceramics, three way catalysts, solid oxide fuel cells, polishing agents, gate oxides in metal oxide-semi conductor devices, UV shields, organic –dye-free solar cell, etc.[1,2].In many applications, for example in solar cells, it is desirable to prepare CeO2 particles with large surface area and smaller particle size. Smaller particles in nano scale exhibit novel properties, otherwise not possible in higher dimensions, mainly because of the large ratio of number of atoms in the surface to the number in the bulk. This led to increasing research on CeO2 – based nano particles [2-8].It has a fluorite-like cubic structure in which each Cerium site is

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surrounded by eight oxygen sites in fcc arrangement and each oxygen site has a tetrahedron cerium site.Methods based on wet chemical routes like co-precipitation, hydrothermal, sol-gel method, microemulsion method etc have been employed to synthesis CeO2 nano particles by many researchers [9-16].But the above methods seem to be tedious and expensive to obtain CeO2 nanoparticles.

It is reported that nano particles can be prepared in an easy manner using a domestic microwave oven[17].This method has been successfully applied for the preparation of nanosized inorganic materials[18-20].Compared with conventional heating,microwave heating has an advantage of high efficiency and rapidformation of nanoparticles with a nano size distribution. In this work, we have prepared CeO2 nanoparticles using micro wave assisted solution method.

II EXPERIMENTAL

The entire chemicals were purchased from Merck and used as received without further purification. In a typical reaction process, Ammonium Ce (IV) Nitrate was dissolved in deionized water with a constant stirring at room temperature and then pH value was adjusted to 10 by drop wise addition of Sodium Hydroxide. The resultant solution was then transferred to a microwave oven and heated at 70°C for 30 minutes. After terminating the reaction, pale yellow precipitate was obtained and then washed with water and ethanol several times and dried at 95°C for 8 hours. Conventional thermal treatment of as prepared at 230°C and 800°C in air for 2h resulted in the formation of CeO2 nanoparticles.

The structure and phase purity of the powders were examined by powder X-ray diffraction (XRD) technique using an X-ray diffractometer (Model Bruker D-8). The phase purity and the presence of functional groups of the as-prepared ceria nanoparticles are analyzed using FT-IR spectroscopy(Shimadzu 8400S FT-IR spectrometer). To study the size-dependent quantum size effect and the optical quality of the ceria nanoparticles, optical absorption studies are carried out by UV-1800 series spectrophotometer. The morphologies of as-prepared samples were investigated through SEM (Hitachi).

III RESULT AND DISCUSSIONS

1. Structural analysis by XRD

XRD measurements indicate that the CeO2 powder is well crystallized with Bragg reflection peaks corresponding to a cubic fluorite structure,consistent with the reference data[JCPDS file (PCPDF 34-0394)].Fig 1 represents the XRD stack pattern of as-prepared, annealed at 230 ^{o}C and annealed at 800 ^{o}C samples . The exhibited XRD peaks correspond to the (1 1 1), (2 0 0), (2 2 0), (3 1 1), (2 2 2), (4 0 0), (3 3 1) and (4 2 0) planes respectively. The unit cell parameters of this sample were found using the software 'UNITCELL' and the observed values are a = b = c = 5.407 Å and $\alpha = \beta = \gamma = 90^{o}$. It clearly states that after annealing the nanoparticles at high temperature (800 ^{o}C), the values of particle size are observed to be nearly same and all are in nearly spherical in shape, which is consistent with the SEM image.

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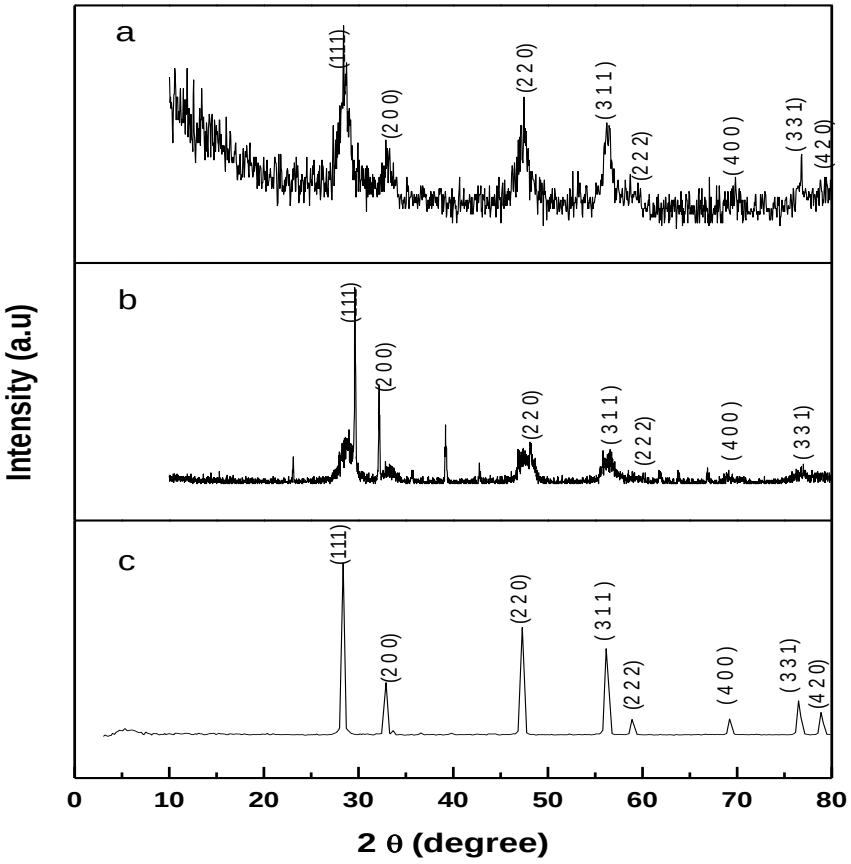

Fig 1. XRD stack view of as-prepared (a) and annealed (b& c) CeO_2 nanoparticles.

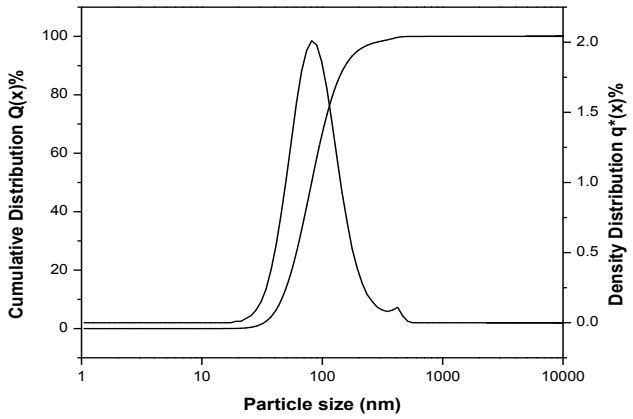

Fig. 2. Particle Size Analyzer Spectrum

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The particle size analysis for the sample annealed at 800 ^{o}C was carried out using the particle size analyzer (Nanophox, model: 0102 P). The particle size distribution is presented in the Fig. 2 and it is observed that the size of maximum number of particles is varied from 10 and 75 nm. It is in good agreement with the particle size observable on the TEM image.

2. FT-IR Spectroscopic Studies of Ceo_2 Nanoparticles

The FT-IR spectrum of the nanoparticles of CeO_2 annealed at 800 ^{o}C is shown in Fig. 3. The broad absorption band located around 3448 cm^{-1} corresponds to the O-H stretching vibration of residual water and hydroxyl groups, while the absorption band at 1647 cm^{-1} is due to the scissor bending mode of associated water. The existence of CH_2 vibrations at 2453.3 and 2939.3cm^{-1} indicates that the surfactant is not present in the as-synthesized sample. The bands at 3382.9 and 1647.1 cm^{-1} can be attributed to the O–H vibration in absorbed water on the sample surface. In addition to the bands in the 850-1600 and 2800-3000 cm^{-1}, the band due to the stretching frequency of Ce-O can be seen below 700 cm^{-1}. The FT-IR peaks at about 1515, 1265, 1130, 1064, 952 and 862 cm^{-1} are similar to those of commercial CeO_2 powders and CeO_2 nanoparticles. The band at 862 cm^{-1} and 819 cm^{-1} corresponds to (Ce – O) metal-oxygen bond. It is to be mentioned here that the FT-IR spectra of as-prepared and annealed at 230 ^{o}C samples of CeO_2 are observed to be similar as that of CeO_2 nanoparticles annealed at 800 ^{o}C. Hence FT-IR spectra of as-prepared and annealed at 230 ^{o}C samples of CeO_2 are not presented here.

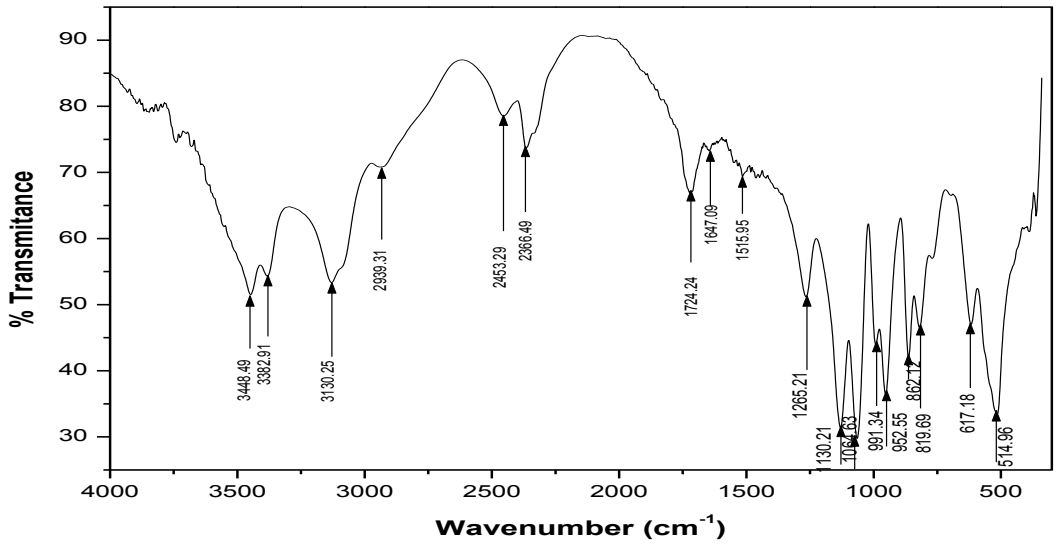

Fig. 3. FT-IR spectrum of CeO_2 nanoparticles

3. UV-visible spectroscopic study of CeO_2 nanoparticles:

The optical absorption spectrum of the ceria nanoparticles is depicted in Fig. 4 shows a well-defined absorption peak located at 321 nm which confirm that CeO_2 nanoparticles are optically active. It is found that the band gap of the

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CeO_2 nanoparticles in the present study is 3.22 eV and this value is found to be smaller than that of bulk CeO_2 nanoparticles. The bulk band gap of CeO_2 is 3.19 eV .

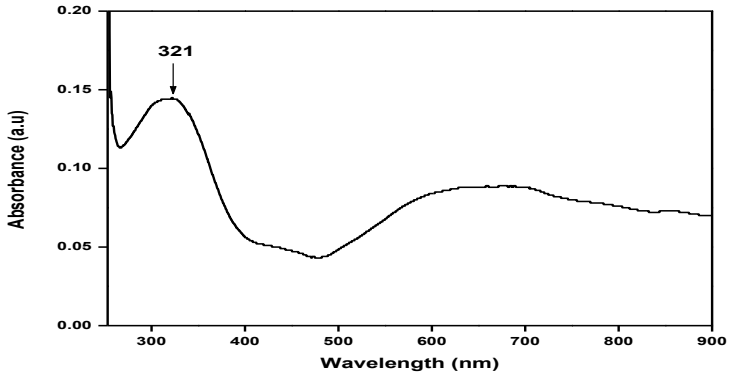

Fig. 4. UV-Vis absorption spectrum of CeO_2 nanoparticles

It implies that the band gap energy of nanocrystalline CeO_2 increases by 0.03 eV when compared to that of bulk CeO_2 and this increase in the band gap energy is due to qunantum confinement of charge carriers in the CeO_2 nanosystem. As a result, the absorption band is shifted to the range of higher energy and blue shift is observed.

4. SEM image of CeO_2 nanoparticles

Surface morphology of the deposited film was examined by scanning electron microscopy (SEM).

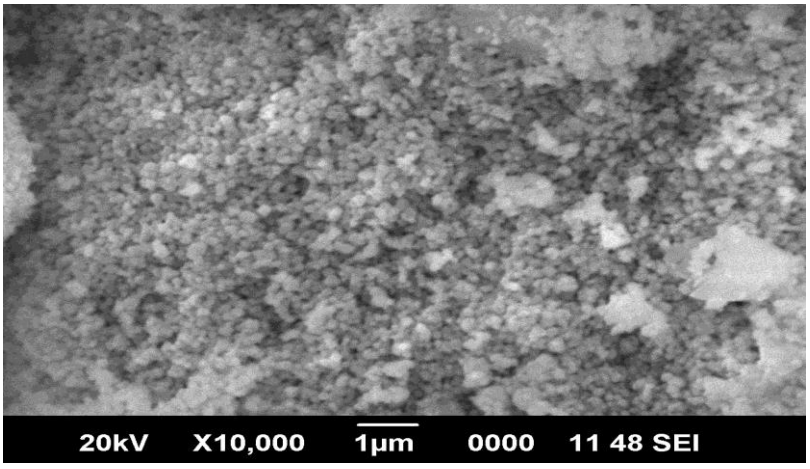

Fig 5. SEM Image of Ceo_2 Nanoparticles

The particles were spherical nature and seemed to be nanosized, typically in the range of < 75 nm. The SEM micrograph displays a spherical structure with high porosity and interconnectivity enhances the electrical conductivity of the sample.

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IV CONCLUSION

CeO_2nanoparticles were successfully synthesized by microwave assisted method and were analyzed by XRD, FTIR,SEM, and UV-Visible spectroscopy. The XRD studies revealed that they have face centered cubic structure and have the average grain size of 20 nm. From the SEM studies, the average particle size of CeO_2 nanoparticles was found to be 63 nm. The UV-visible spectroscopic studies reveal that the CeO_2 nanoparticles have a lower UV cut off wavelength, wider optical transmission range i.e. it has better optical properties.

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