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XRD, FT-IR, SEM and TGA-DTA Characterization of Hydrated Cerium Oxide Nanoparticles

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Abstract
Hydrated CeO_2 nanoparticle (CNPs) was synthesized via chemical precipitation technique at room temperature in presence of ammonium acetate. Powder XRD study reveals single phase with cubic structure. Particle size is determined by Scherrer's equation & Scanning electron micrograph which exhibit in range of 50-60nm. Single stage dehydration at high temperature is confirmed from thermal analysis. FT-IR analysis qualitatively confirms the formation of the title compound.
Keywords: Synthesis, XRD, SEM, Particle size, TGA-DTA.

I. Introduction

In the recent years, much attention has been drawn on the development of new synthetic paths for preparing nanostructure cerium oxide as they finds applications in various fields such as in medicine, Abrasives, Catalysts, UV blocks, Ceramics, Cosmetic Applications and opto-electronics devices and so on [1].

Several methods are available for the synthesis of nanostructure material [2-5]. Among them, chemical precipitation technique has been used in the present study, as it is easy scale up, mild synthesis condition, simple and cheap. Zhang *et al.* synthesized CeO_2 nanoparticles by precipitation method using alcohol-water mixed solvent [6]. Although, several report was found in the literature on the synthesis of CeO_2 nanoparticles at more than room temperature [7-9]. However, synthesis of CeO_2 nanoparticles at room temperature has an advantage of no aggregation of particles and a very small size nanoparticles was obtained [10]. Zhang *et. al*. synthesized CeO_2 nanoparticle at room temperature by mixing HMT with CeO_2 nitrile solution [11]. Synthesis, formation, assembly and application of metal oxide nanoparticles were summarized in the book written by Markus Niederberger, Nicola Pinna [12].

Liu et al. synthesized cerium dioxide nanoparticles using ammonia-water solution. They were successful in obtaining particle in the range 100-200 nm [13]. So, in the same foot print, we tried to use ammonium acetate as solution. It is expected that, ammonium acetate can be used to control morphology, size & agglomeration of hydrated ceramic oxide nanoparticles.

In the present study, CNPs were successfully synthesized using chemical precipitation technique at room temperature in presence of ammonium acetate. The properties of the material were characterized by powder XRD, Scanning electron microscope (SEM), TGA-DTA.

II. Experimental

Material synthesis

The Starting materials were Cerium nitrate hexahydrate, ammonium acetate and conc. Aq. NH_4OH with an equivalent concentration of 1.1 mol L^{-1}. Ammonium solution was taken in excesss during precipitation to achieve pH 9.5-10. In each of experiments, cerium nitrate hexahydrate was dissolved in double distilled water and dropwise aqueous NH_3 was added slowly added with vigorous stirring at room temperature. Precipitation began immediately with characteristic gelation of the mixture. The gel was stirred for 20 min. to achieving a homogenous mixture. The gel resulting was divided into four partions. The gel solution was added into without and with different concentration of ammonium acetate solutions. Each solution was stirred for 5 min and then left to age 7 days at room temp. After 7 days, the resulting product was washed several times by centrifugation at 3000 rev min^{-1} with dil. NH_4OH maintaining pH 9.5-10 throughout the experiment and then once with double distilled water. The final product dried in oven at ~100 ^{0}C and grind into fine powder.

Characterization

The powder X-ray diffraction pattern was determined on Regaku Miniflex Diffractometer using CuKα radiation (λ=1.5405, nickel filter). The surface morphology study was performed using scanning electron microscope (SEM) (Model: Philips SEM, model 30 XL). TGA-DTA analysis was performed on the instrument DTG-60H simultaneous differential thermal analyzer (Shimadzu, Japan) within the temperature range 32-800^{o}C.

III. Results and discussion

Powder XRD

The powder X-ray diffraction (XRD) pattern of the synthesized hydrated CeO_2 nanoparticles using ammonia acetate is shown in Fig.1. The diffraction peaks of the nanoparticles are quite broad indicating the crystalline size of the sample is very small. No peak of any other phase has been observed indicating high purity of the sample. The hydrated CeO_2 displays cubic structure with lattice parameter of 0.554nm.

The crystalline size <D> of CeO_2 has been calculated using Debye- Scherrer formula;

$<D> = 0.94 \lambda / \beta Cos \Theta$

Where 'λ' is the wavelength, β is the full width half maxima,'Θ' is the Bragg's angle. The calculated crystallized size and lattice parameter of hydrate ceramic oxide nanoparticle material is tabulated in Table 1.

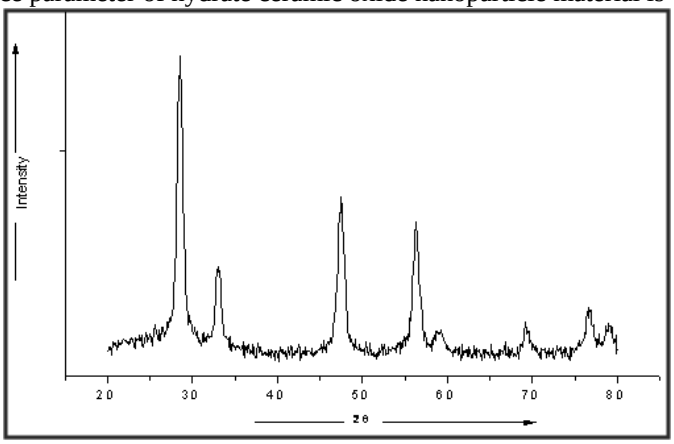

Fig.1. Powder XRD of CNPs.

Compound	Lattice parameter (nm)	Mean crystallite size (nm)	Average particle size in nm (From SEM)
CeO_2	a=b=c=0.554 α=β=γ=90^{o}	22.70	59.70

Table 1. Lattice parameter, crystallite size and particle size of CNPs.

As can be seen from this table, the particle size is quite small in the range 50-60nm.

FT-IR Morphology study

SEM image of CeO_2 nano-powder is as shown in Fig.

Fig.2. FT-IR spectrum of CeO_2 nanopowder.

Table 1. Band and corresponding peak assignment.

The FT-IR spectrum of hydrated CeO_2 is as shown in Fig

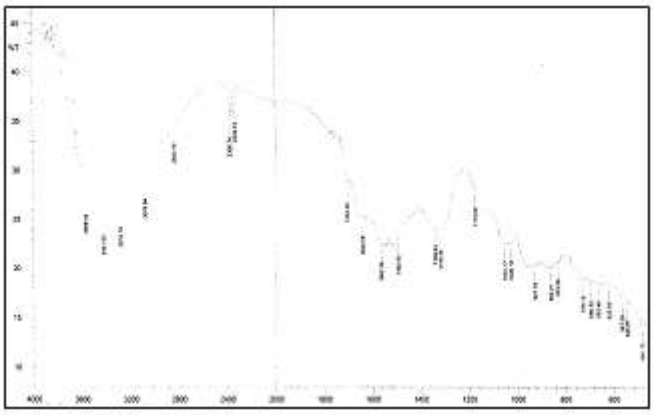

Fig.2. FT-IR spectrum of CNPs .

Band(cm^{-1})	Peak Assignments
3421	υ HOH
1562	υ bending –OH groups
567	υ Ce-O
465	υ Ce-O

Table 2. Bands and corresponding peak assignement of CNPs.

The bands at around 3400 cm^{-1} correspond to presence of lattice water. The band around 1570 cm^{-1} corresponds to –OH group present in the title compound. The bond originates from the absorption of H_2O. The stretching bands near 565 and 465 cm^{-1} assigned to bonding of Ce-O bonds [13]. The presence of bond at 3421 and below 700 cm^{-1} qualitatively confirms the formation of hydrated cerium oxide nanoparticls. The bond and corresponding peak assignment are tabulated in Table II.

SEM

The SEM image of CNPs is as shown in Fig.3. It shows that particles tend to agglomerate during centrifusing & drying or during synthesis process. The formation of agglomerate was most likely due to Vander-Wall's interaction between crystallites. Average particle size of ceramic oxides is given in Table 1.

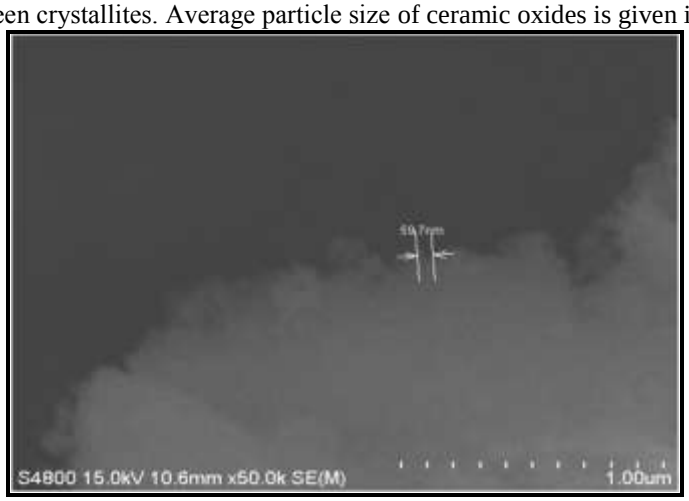

Fig.3. SEM image of hydrated CeO_2.

TGA-DTA

TGA-DTA curve of hydrated cerium oxide nano-powder under normal atmosphere condition is as shown in Fig.4. The study has been carried out in the range 32-800 ^oC .The peak in DTA curve is observed at 325 ^oC. TGA curve indicate 4.903 percentage mass losses while the calculated one is 4.969 percentages at 325 ^oC. It is

evident from the graph, the single state change behaviour. Thermal study confirms the formations of hydrated cerium oxide nanoparticles at room temperature. The hydrated group has been evolved at high temperature.

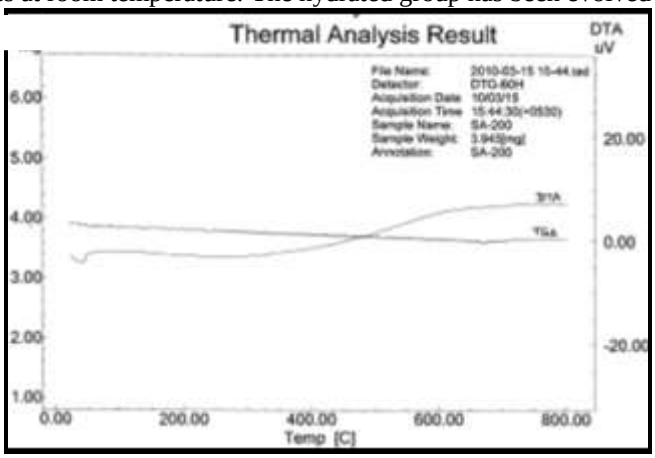

Fig.4. TGA-DTA spectrum of CNPs.

IV. Conclusions

Hydrated CeO_2 nanoparticles was successfully synthesized by chemical precipitation method at room temperature using ammonium acetate. FT-IR spectral analysis qualitatively confirms the formation of title compound. Thermal analysis indicates single stage dehydration to form hydrated cerium oxide. At high temperature high temperature, cerium oxide nanoparticles are formed. XRD study confirms the title compound synthesized into cubic structure with single phase. The calculated particle size of hydrated CeO_2 nanoparticles was in the range of 50-60nm confirms from XRD and SEM.

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