

# Green synthesis and characterization of Ceria nanoparticles using Ricinus communis leaf extract

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**Abstract-** Nowadays, green synthesis of nanoparticles has great interest and achievement due to its eco-benign and low time consuming properties. In this work, Ceria nanoparticles is Synthesized using Ricinus communis (Castor oil) leaf extract. The Synthesized nanoparticles are characterized by UV-Vis spectroscopy, dispersive X-ray analysis (EDAX), which is attachment of scanning electron microscope (SEM). Crystalline nature and purity are revealed by X-ray diffraction (XRD) and their FTIR spectra are examined to identify the effective functional groups present in the synthesized nanoparticles. Raman studies show a characteristic peak at  $461\text{ cm}^{-1}$ .

**Index Terms-** Ceria nanoparticles, Green synthesis, Ricinus communis, Raman spectroscopy

## I. INTRODUCTION

Semiconductor oxide Nanoparticles play an important role in the recent research due to their interesting physical and chemical properties. Ceria is a semiconductor oxide with wide bandgap of  $3.19\text{ eV}$  at room temperature and high excitation binding energy. It is widely used in many applications like sensor, fuel cell, catalyst, cosmetic, antioxidant and bioimaging [1-6]. Ceria nanoparticles are less toxic compare to other semiconductor oxide nanoparticles. Generally Ceria nanoparticles were synthesized by physical and chemical methods. Phytosynthesis of ceria nanoparticles is more cost effective and ecofriendly. Cerium oxide has two different oxide forms,  $\text{CeO}_2$  and  $\text{Ce}_2\text{O}_3$ . On the nanoscale, the cerium oxide has cubic fluorite structure with the coexistence of  $\text{Ce}^{3+}$  and  $\text{Ce}^{4+}$  ions on its surface. The surface  $\text{Ce}^{3+} : \text{Ce}^{4+}$  ratio is influenced by the

microenvironment [7]. Recently, phytosynthesis of ceria NPs was reported using different plants. The plant extract acted as capping as well as stabilizing agent in ceria nanoparticles synthesis process[1,8,9].

Ricinus communis or the castor oil plant is a species of perennial flowering plant in the spurge family, Euphorbiaceae. Although it is indigenous to the southeastern and India today it is widespread throughout tropical region. Castor seed is the source of castor oil which has a wide variety of uses. Its leaf extract has many medicinal uses. Its seed contains a water-soluble toxin, ricin, which is also present in lower concentrations throughout the plant. In this paper, the synthesis of ceria nanoparticles using Ricinus communis leaf extract and its characterization studies have been reported for the first time.

## II. EXPERIMENTAL DETAILS

## 2.1 Materials and methods

Cerium chloride heptahydrate was purchased from the nice company. Leaves of Ricinus Communis) were collected in the local area of Madurai, Tamilnadu, India. The leaves were washed, dried and cut into fine pieces. Then the fine pieces were put in the mixer to get the leaf extract. The extract was filtered through Whatman No. 1 filter paper and it was collected.

## 2.2 Synthesis of ceria nanoparticle using Ricinus communis leaf extract

Aqueous solution of 1.8629g of cerium chloride heptahydrate [CeCl<sub>3</sub>.7H<sub>2</sub>O] was used for the synthesis of cerium oxide nanoparticles. 10ml of Ricinus Communis leaf extract was added to 50ml of CeCl<sub>3</sub>.7H<sub>2</sub>O in a 250ml Erlenmeyer flask and stirred at 80°C for two hours. Ricinus Communis leaf extract acts as a precipitating agent which is responsible for the synthesis of the nanoparticle. The particle formed after adequate time of stirring was collected by centrifugation at 10000 rpm for ten minutes. The centrifuged particles were washed with water and again subjected to centrifugation at 5000 rpm for ten minutes. The centrifuged sample was dried in an air oven, was powdered using mortar and pestle. This powdered sample was calcined in muffle furnace at 400°C to get ceria nanoparticles.

## 2.3 Characterization of Ceria NPs

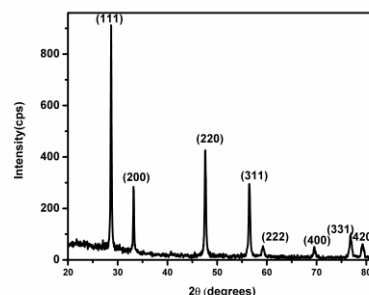
The phytosynthesized Ceria NPs were subjected to XRD analysis. The XRD pattern was recorded using Cu K $\alpha$  radiation ( $\lambda= 1.54 \text{ \AA}$ ) in the range of  $2\theta$  from  $20^\circ$  to  $80^\circ$ . Fourier transform infra red spectroscopy (FT-IR) analysis was carried out in the range of  $400\text{-}4000\text{cm}^{-1}$ . The micro Raman analysis of the synthesized ceria NPs was also carried out. UV-Visible absorption spectra of the synthesized ceria NPs was recorded in the range of  $190\text{-}800\text{nm}$ .

The energy dispersive spectroscopy (EDAX) associated with Scanning electron microscopy (SEM) of the synthesized NPs were also recorded.

## III. RESULTS AND DISCUSSION

### 3.1 X-ray diffraction analysis

The X-ray diffraction peaks of ceria NPs synthesized using Ricinus communis leaf extract are shown in figure 3.1. The X-RD peaks are located at angles ( $2\theta$ ) of  $28.9^\circ$ ,  $33^\circ$  and  $47.36^\circ$  corresponding to (111), (200) and (220) planes of ceria NPs. Similarly other peaks found at angles  $56.56^\circ$ ,  $59.34^\circ$  and  $69.71^\circ$  are corresponding to (311), (222) and (400) planes of ceria NPs.



**Figure 3.1 X-RD Pattern of the ceria NPs**

The Observed peak for ceria NPs matches with the JCPDS values (03-065-5923). Hence, the XRD pattern clearly shows that the synthesized ceria nanoparticles is identical and could be indexed to the standard ceria with fluorite cubic structure. The lattice constant 'a' of ceria NPs is calculated using the relation

$$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

The calculated value of the lattice constant 'a' of the ceria NPs is  $5.402\text{\AA}$ .

The average particle size of cerium oxide can be calculated using the Debye- Scherrer's equation,

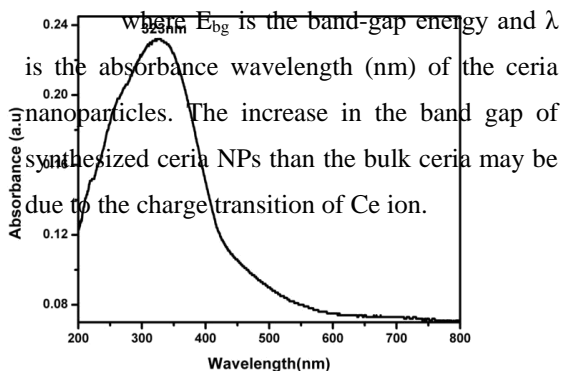
$$D = \frac{k\lambda}{\beta \cos\theta}$$

where k is the Scherrer's constant with the value from 0.9 to 1 (shape factor),  $\lambda$  is the X-ray wavelength (1.5418 Å),  $\beta$  is the width of the XRD peak at half-height and  $\theta$  is the Bragg angle and D the grain size. The average crystalline size of the synthesized ceria NPs is found as 34 nm.

### 3.2 UV-Visible Spectra

Figure 3.2 shows UV - Vis spectra of synthesized ceria nanoparticles using Ricinus Communis leaf extract. Ricinus Communis leaf extract consists of phytoconstituents which act as capping agent. The sample displays an optical absorption peak at about 323nm in the UV region which is typical of absorption for metallic ceria nanocluster. Usually peak around 300 nm corresponds to the fluorite cubic structure of ceria. Additionally, UV spectrum showed no other peak related with impurities and structural defects which confirms that the synthesized nanoparticles are pure ceria. Further band gap energy was calculated on the basis of the maximum absorption band of ceria nanoparticles and found to be 3.84 eV according to equation

$$E_{bg} = 1240/\lambda \text{ (eV)}$$



where  $E_{bg}$  is the band-gap energy and  $\lambda$  is the absorbance wavelength (nm) of the ceria nanoparticles. The increase in the band gap of synthesized ceria NPs than the bulk ceria may be due to the charge transition of Ce ion.

### Figure 3.2 Absorption peak of Ceria NPs synthesized from Ricinus Communis leaf extract

#### 3.3 FTIR Analysis

Each molecule absorbs only IR light of certain frequencies based on its own characteristics. Hence it is possible to identify the molecule type qualitatively and quantitatively by studying the absorption spectrum. Figure 3.3 shows the FTIR spectrum of the ceria nanoparticles which were acquired in the range of 400–4000  $\text{cm}^{-1}$ .

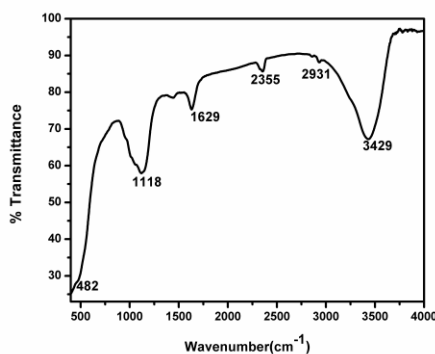
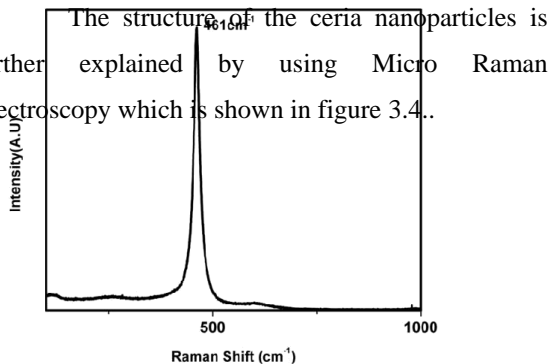


Figure 3.3 FTIR spectrum of Ceria NPs

The peak at  $3429\text{cm}^{-1}$  corresponds to stretching vibration of O-H bonds in water molecules and the band at  $1629\text{cm}^{-1}$  could be attributed to in-plane and out-of plane bending of O-H bonds present in the absorbed water molecule. The peak obtained at  $2931\text{cm}^{-1}$  is assigned to be C-H stretching vibration[10] The absorption peak at  $2355\text{cm}^{-1}$  responsible for some trapped  $\text{CO}_2$  from the environment. The peak at  $1118\text{cm}^{-1}$  is due to the overtone band of the trace of Ce-OH. The peak at  $482\text{cm}^{-1}$  is attributed to the O-Ce-O stretching mode of vibration[10-12].

#### 3.4 Raman studies

The structure of the ceria nanoparticles is further explained by using Micro Raman spectroscopy which is shown in figure 3.4..



**Figure 3.4 Raman spectrum of synthesized ceria nanoparticles**

Ceria nanoparticles exhibited a strong intense band at 461cm<sup>-1</sup> which generally corresponds to the F<sub>2g</sub> Raman active-mode due to symmetrical breathing mode of the O atoms around each cerium ions. Thus Raman spectrum confirms that the synthesized products have well crystalline fluorite cubic structure [13]. The particle size of the ceria NPs can also be calculated using Raman line broadening using the formula

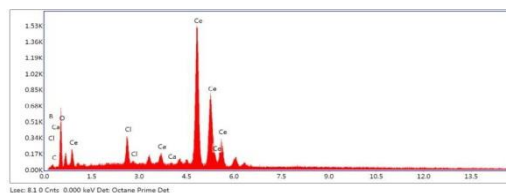
$$\Gamma (cm^{-1}) = 10 + \frac{124.7}{D_R}$$

where  $\Gamma$  is the full width half maximum of the Raman active mode peak and  $D_R$  is the particle size[ 14]. On substituting,  $\Gamma$  as 461cm<sup>-1</sup>, the calculated particle size is obtained around 3nm which is found to be lower than that of obtained from X-RD spectrum.

### 3.5 EDAX Analysis

Analysis through energy dispersive X-ray (EDX) spectrometer has verified the presence of elemental cerium and the oxygen in the synthesized

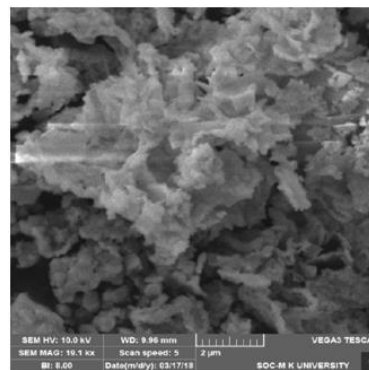
ceria nanoparticles (Fig. 3.5). The vertical axis displays the number of X-ray counts and the horizontal axis displays energy in KeV. The peaks in the spectrum marks the major emission energies of cerium and oxygen. It is found that the atomic percentage of Oxygen is more than that of cerium.



**Figure 4.4 EDAX spectrum of synthesized ceria nanoparticles**

### 3.6 SEM ANALYSIS

The surface morphology of the Ceria nanoparticles is examined by scanning electron microscopy (SEM) and is shown in figure 4.5.



**Figure 3.6 SEM image of ceria nanoparticles**

SEM images reveal that there are various sizes of particles in the as-prepared sample. It can be clearly observed that many agglomerates with an irregular morphologies. The particles are connected to each other to make long complex systems with irregular porous size and shapes. There are numerous micro pores are present on the surface of the particles. The pores are formed due to the liberation of large amount of CO<sub>2</sub> and H<sub>2</sub>O gases during

calcination process. These escaping gases are swelled the materials and resulted in the formation of pores in the materials.

#### IV. CONCLUSION

In summary, Ceria NPs have been successfully synthesized using Ricinus Communis leaf extract. UV studies indicated that the obtained absorption peak is of metallic ceria nanocluster. The XRD patterns, EDAX pattern and Micro Raman spectra suggest the formation ceria NPs with cubic fluorite structure. The functional groups in the ceria nanoparticles observed in FTIR spectrum confirms the presence of ceria. The SEM image clearly showed many agglomerates with an irregular morphologies. It is expected that the green synthesized ceria NPs can find many potential applications as bulk metal ceria in many fields.

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