

Green Synthesis and Characterization of Cerium Oxide Nanoparticles using *Euphorbia hirta* Leaf extract

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(Received November 07, 2021)

Abstract: In the present era, synthesis of cerium oxide nanoparticles (CeO_2NPs) employing natural resources as reducing and stabilizing agents has been widely used and advocated by the researchers because of their eco-friendly, simple, economical and viable features. Fabrication of non-toxic, cost-effective CeO_2 NPs was carried out by eco-friendly approach using *Euphorbia hirta* leaf extract. The optical properties of green mediated cerium oxide nanoparticles revealed through UV-visible spectroscopy. The active biomolecules and the functional groups responsible for biological reduction of metal ions into nano-scale CeO_2 particles were analysed using Fourier transform infrared spectroscopy (FTIR). The structure, phase and crystalline planes of nanoparticles were investigated using X-ray diffraction studies. The experimental results reveal that *Euphorbia hirta* leaf can be employed as an economical and beneficial potential bio-resource for the synthesis of metal oxide nanoparticles. These GNPs have potential applications in many fields, viz., medical, industrial, environmental, etc.

Keywords: *Euphorbia hirta*, Cerium oxide nanoparticles, UV-visible spectroscopy, XRD-EDX

Graphical Representation:

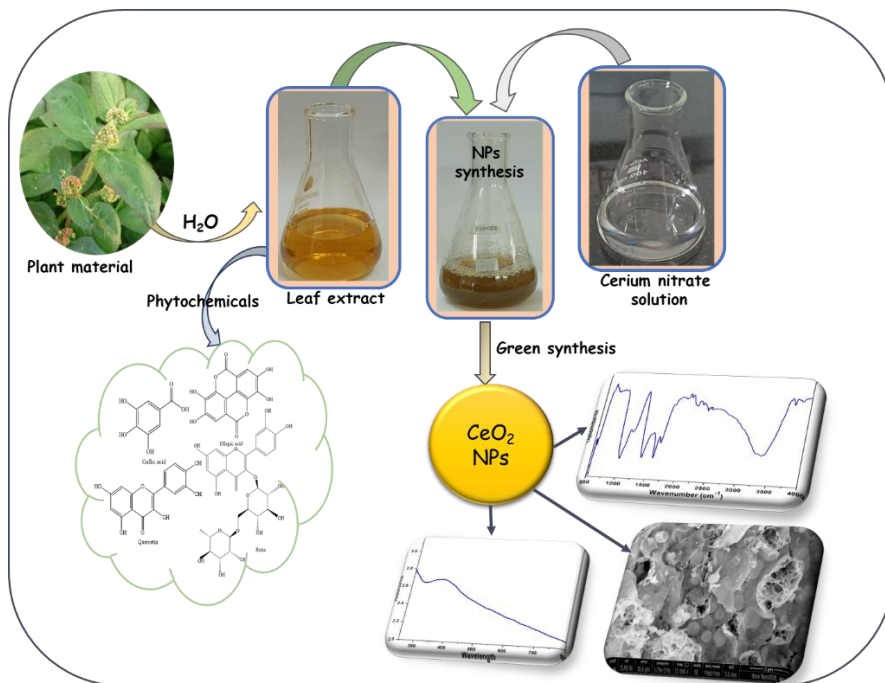


Figure 1. Schematic representation of the green synthesis of cerium oxide nanoparticles using *Euphorbia hirta* extract.

1. Introduction

Nanotechnology is the creation, characterization, expedition, and application of nano-sized materials (1-100 nm) which provides the aptness to modify the properties of materials by controlling their size. Nanotechnology navigates the research towards a lot of potential uses in different fields like electronics, industry, imaging and medicine¹⁻⁵. The nanoparticles have manifested significant attention owing to their high surface area, high reactivity, unique particle morphology. Recent years, metal oxide nanoparticles are in focused due to its remarkable properties and various applications. In particular, synthesis of nanomaterials had drawn much attention in nanotechnology research, as there is increasing demand in industries and medical sectors such as fillers, disinfectants, optics, antimicrobial agents, drug delivery and catalytic products⁶. Various metal oxide nanoparticles such as CuO, TiO₂, ZnO and Fe₂O₃ are investigated for their environmental and biomedical applications. Cerium

oxide nanoparticles (CeO₂-NPs) have gained extensive attention of researchers, due to their high stability and biocompatibility and its application in catalysis, water purification by photocatalysis, cells, batteries, sensors fabrication, therapeutics agents and drug delivery carriers⁷⁻¹⁰. Numerous methods are commonly used for the synthesis of NPs by chemical and physical routes such as Co-precipitation methods¹¹, solvothermal¹², template assisted precipitation¹³, spray drying system¹⁴, microwave¹⁵, hydrothermal¹⁶ and sol-gel method¹⁷ etc. However, many of these techniques are complex, time-consuming, expensive and utilize noxious reducing solvents causing several threats to the ecosystem. Thus, recently a simple, safe, less toxic and eco-friendly method has been used by researchers which is known as green synthesis. Nanoparticles produced through green route represent an innovative methodology in era of nanotechnology that is achieved using extracts of plant¹⁸⁻¹⁹, microorganisms²⁰⁻²¹, vitamins, sugars and other derivatives²²⁻²⁴. These biological extracts have a rich source of phytochemicals such as carbohydrates, saponins, amino acids, flavonoids, terpenoids and proteins, which are believed to be responsible for the reduction and stabilization of bulk salts into respective nanoparticles size. Literature survey reveal biosynthesis of CeO₂NPs was reported by using leaf extracts including Aloe vera²⁵, *Origanum majorana* L²⁶, *Gloriosa superba*²⁷, *Moringa oleifera* L²⁸, *Hibiscus sabdariffa*²⁹, *Prosopis juliflora*³⁰, *Azadirachta indica*³¹, *Jatropha curcus*³², *Calotropis procera*³³ have emerged as a simple and viable alternative to chemical synthetic and physical methods.

In this paper, the green synthesis of CeO₂ NPs using *Euphorbia hirta* (*E. hirta*) plant leaf extract has been reported and investigated their characterizations. Though, there is no literature on green synthesis of CeO₂-NPs using *Euphorbia hirta* leaf extract. *Euphorbia hirta*, a medicinal herb, belongs to genus *Euphorbia*, family *Euphorbiaceae*. Its leaves are usually greenish or reddish underneath and darker on the upper surface, oppositely arranged and lanceolate. The stem is slender, covered with yellowish bristly hairs and often reddish in colour. When cut or on injured aerial parts produce a milky juice³⁴⁻³⁵. The plant has been widely acknowledged for treatment of cough, coryza, asthma, bowel complaints, worm infestations, kidney stones in traditional medicine³⁶⁻³⁷. *E. hirta* contains various bioactive components such as alkaloids, flavonoids, saponins, carbohydrate, terpenoids, amino acids and poly phenols etc. and mostly of them used as inhibition of corrosion³⁸⁻⁴¹. The *E. hirta* elicits good antimicrobial, antioxidant, antifungal and anti-inflammatory properties. The point of this work is to synthesize CeO₂NPs using the cost-effective,

simple, time-saving and eco-friendly method using leaf extract of *Euphorbia hirta*. The synthesized CeO_2 -NPs were characterized by using standard tools viz., XRD, UV-Vis, FTIR and FE-SEM.

2. Experimental

2.1 Materials: Cerium nitrate hexahydrate [$\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$] of AR grade was used as precursor for nano-scale metal oxide preparations. Fresh leaves of *Euphorbia hirta* were collected from the University campus, University of Rajasthan, Jaipur, India. All solutions were prepared using triply distilled water.

2.2 Preparation of *Euphorbia hirta* leaf extract: The collected leaves of the selected weed plant were washed thoroughly to remove the dust particles and dry in shade for a stipulated time period at room temperature. Dried leaves were squashed and grinded into fine powder. The grinded leaf powder of the *Euphorbia hirta* was used for extract preparation by using Soxhlet extractor with triply distilled water as solvent. The aqueous colloidal solution obtained after extraction is centrifuged and filtered through Whatman paper to remove the plant residues and stored in a refrigerator at 4°C for further use.

2.3 Synthesis of Cerium Oxide Nanoparticles (CeO_2 NPs): Cerium oxide nanoparticles (CeO_2 NPs) were synthesized via greener route. For this 0.01M of $\text{Ce}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ solution was prepared in triply distilled water. To this, the *Euphorbia hirta* leaf extract was added slowly in a set ratio with continuous stirring by magnetic stirrer until a brown colloidal solution was obtained. The colloidal solution thus obtained was centrifuged at 10,000 rpm for stipulated time period. The centrifuged particles were washed 2-3 times with distilled water. The centrifuged particles were separated and finally dried in hot air oven at 60°C . The CeO_2 NPs so obtained were stored in a plastic tube vial at room temperature.

2.4 Characterization of Synthesized Nanoparticles: The synthesized nanoparticles were characterized by using certain standard analytical techniques.

2.1.1 UV visible spectrum: UV visible absorption spectra were recorded by using Shimadzu UV-2450, Japan, UV-spectrometer.

2.1.2 FT-IR spectroscopy: IR Perkin Elmer instrument was used to record the FT-IR spectra of fabricated metal oxide nanoparticles. The

spectrum was recorded by scanning the sample in the range of 400-4100 cm^{-1} .

2.1.3 X-ray diffraction spectra: The powdered raw material was used to carry out the X-ray diffraction spectra. X-PERT PRO diffractometer was used to record the X-ray diffraction spectra of synthesized nanoparticles. XRD was performed at 2θ range of 10-90° using Cu $K\alpha$ radiation of wavelength 1.5406 Å. Operating voltage of 40 kV with 40 mA current was provided at room temperature.

2.1.4 FE-SEM and EDS analysis: The SEM images of synthesized nanoparticles were captured by using Nova Nano 450FE-SEM instrument. The elemental composition of produced nanoparticle was calculated by Energy Dispersive X-ray Spectroscopy which is equipped with FE-SEM instruments.

3. Result and Discussion

The synthesis product, CeO_2NPs , via greener approach was characterized and analysed employing standard tools and techniques. The preliminary observation was change in colour of the reaction mixture depicting formation of metal oxide nanoparticles. The fabrication of metal oxide nanoparticles was endorsed by analyzing the spectral data of UV-Vis and FTIR.

3.1 UV-visible spectroscopy: UV-visible spectroscopy technique was carried out to validate the formation of nanoparticles. The UV-Vis absorption spectra of extract and CeO_2 NPs are shown in figure-2. CeO_2NPs exhibited an absorption maximum at 345 nm as presented in spectra (Fig-2). The direct band gap energy of synthesized cerium oxide nanoparticles is calculated by fitting the absorption data in following equation

$$\alpha h\nu = K (h\nu - E_g)^2.$$

where α is the optical absorption coefficient, $h\nu$ is the photon energy, E_g is the direct bandgap, and K is a constant. Plotting of $(\alpha h\nu)^2$ versus photon energy ($h\nu$) and extrapolating linear portion to the curve to absorption equal to zero, gives the value of direct band gap energy. The direct band gap energy for the synthesized CeO_2NPs was obtained 3.8 eV as shown in figure. This value of band gap is within the range of that reported in literature.

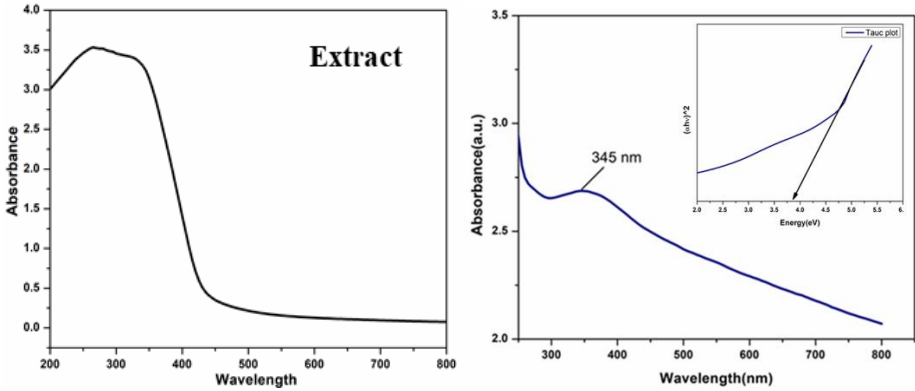


Figure 2. UV-Vis spectra and Tauc plot of *Euphorbia hirta* mediated CeO₂NPs.

3.2 FTIR analysis: Infrared spectroscopy is useful tool for the detection of chemical functional group present in the molecule and to assess the possible role of phyto-constituents responsible for the capping and stabilization of nanoparticles. The FTIR spectra of *Euphorbia hirta* mediated cerium oxide nanoparticles is shown in figure-3. Many prominent peaks were observed in the spectra (Fig.-3). A broad band near 3340 cm⁻¹ is assigned to the stretching vibration of (O–H) of hydroxyl molecules. The peaks at 1633 cm⁻¹ is corresponding to the bending vibrations of the adsorbed –OH groups. In our study, the peak at 992cm⁻¹ and 546 cm⁻¹ in the spectra assigned to the vibration mode of O–Ce–O and Ce–O respectively. The presence of the absorption bands characteristic to Ce–O vibrations confirms the synthesis of nanoparticles as CeO₂.

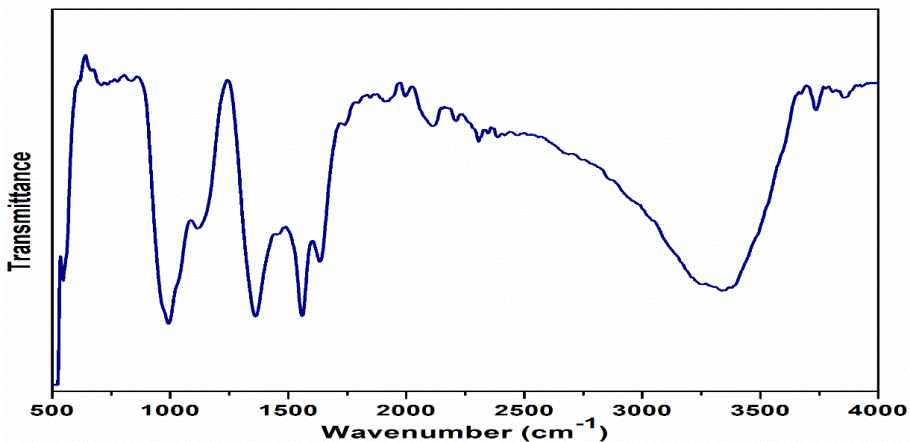


Figure3. FTIR spectra of *Euphorbia hirta* mediated CeO₂ nanoparticles.

3.3 XRD Analyses: The XRD pattern of synthesized cerium oxide nanoparticles was recorded in the range from 20° to 80° of 2θ values which is depicted in the figure-4. The diffraction peak found at various 2θ values 28.5° , 47.39° , 56.25° and 76.9° corresponding to the planes (111), (022), (113) and (133) respectively⁴²⁻⁴³. The indexes of these planes correspond to the FCC cubic fluorite type structure of CeO_2 . Rietveld refinement of the XRD patterns was performed using PANalytical high score software and it was matched with JCPDS reference code 98-062-1704. The phase structure of CeO_2 was cubic with Fm-3m as a space group with the lattice constant values (a) is $5.411 \text{ \AA}$. The unit volume for green synthesized CeO_2 mediated with *Euphorbia hirta* is calculated to be $158.43 \text{ \AA}^3$. The crystallite size of prepared nanoparticles was estimated by using Debye-Scherrer's formula:

$$\text{Average crystallite size (D)} = 0.9\lambda/\beta\cos\theta.$$

In the formula, λ is the wavelength of the X-ray source used for analysis, β is angular peak width at half maxima in radians and θ represents Bragg's angle of diffraction. The average crystallite size was using the above Scherrer formula was found to be 14 nm .

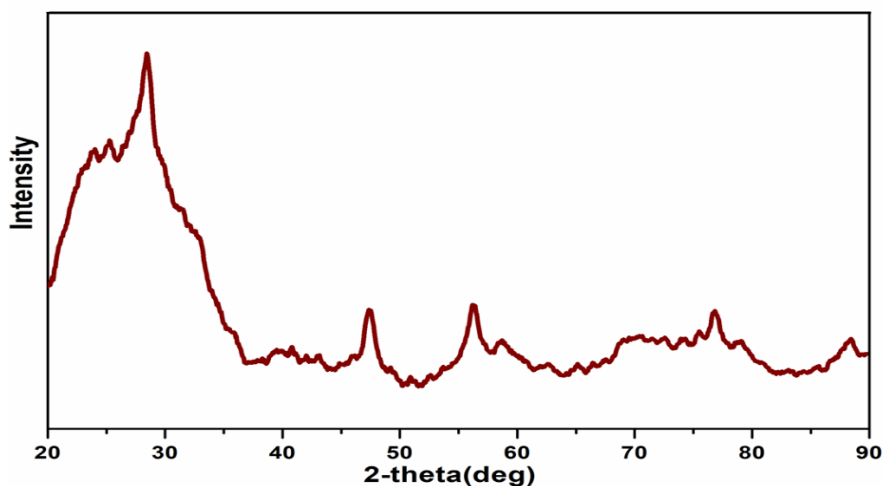


Figure 4. XRD pattern of *Euphorbia hirta* mediated CeO_2 NPs.

3.4 FE-SEM and EDS analysis: The morphology of the synthesized cerium oxide was examined by FE-SEM and the images are presented in Figure-5. From the images that non-uniform morphology of the cerium oxide nanoparticles with some spherical particles are clearly seen. The

CeO₂ nanoparticles were viewed at different magnification like 25,000X and 10,000X. The elemental composition of prepared cerium oxide nanoparticles was characterized by energy dispersive X- ray analysis (EDAX), which is attachment of FE-SEM. The sample for EDAX was prepared by depositing a very thin layer of cerium oxide nanoparticles on glass slide. The EDS spectrum of cerium oxide nanoparticles synthesized using *Euphorbia hirta* leaf extract is shown in figure-6, the composition of sample shows the presence of cerium and oxygen elements. The spectrum peak reveals the presence of Ce and O at 4.837 and 0.525 keV respectively.

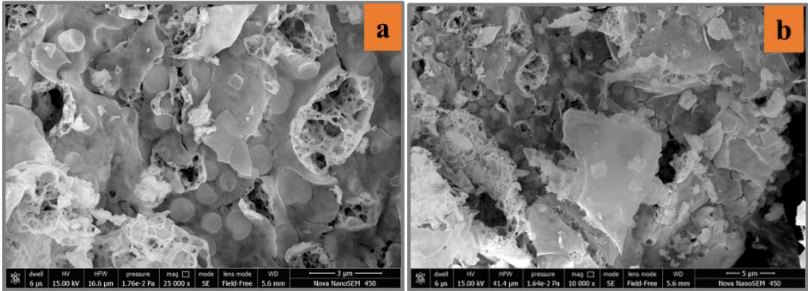


Figure 5. FE-SEM images of *Euphorbia hirta* mediated CeO₂NPs with a magnification (25,000X and 10,000X).

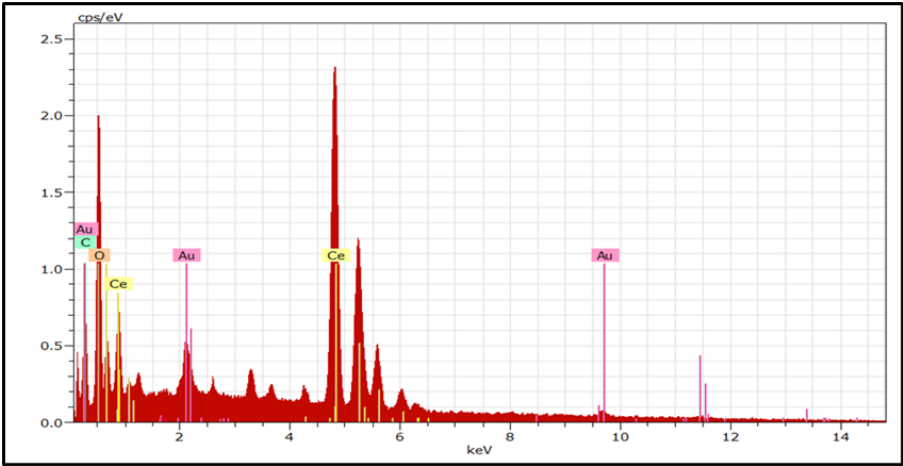


Figure 6. EDAX spectrum for *Euphorbia hirta* mediated CeO₂NPs.

4. Conclusions

Fabrication of Cerium oxide nanoparticles (CeO₂) were successfully carried out using *Euphorbia hirta* leaf extracts by a greener approach with

very minimal or almost negligible use of chemicals. The preliminary observations to justify the formation of CeO₂NPs were made through FTIR spectra. The peaks observed in FTIR spectra confirms the presence of Ce-O bond and signifies the role of *E. hirta* leaf extract in the synthesis of nanoparticles. The morphological, compositional and optical properties of prepared CeO₂ nanoparticles were studied by FE-SEM, EDAX and UV-visible spectroscopy. The XRD result confirms the formation of face centred cubic phase of CeO₂NPs with average crystalline size 14 nm. The band gap of CeO₂ nanoparticles was calculated by Tauc plot and it comes out to be 3.8eV. This fast, simple and economically green synthetic approach is very attractive and effective to fabricate nano-scale metal oxide particles having significant utility in many industrial sectors.

Acknowledgment: The author would like to acknowledge the CSIR for providing financial support to carry out the present work. Author is also thankful to the Head, Department of Chemistry, University of Rajasthan, Jaipur and Director, MNIT, Jaipur for providing access to necessary characterization facilities.

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