Section A-Research paper

Study of Pure Cerium Oxide Nanocrystalline Particles using Co-precipitation Method : Optical and Photo-catalytic Characteristics

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Abstarct:

The characterization of materials is important for understanding their properties and applications. Characterization procedures provide a thorough insight of nanoparticles by evaluating their physical attributes such as crystal structure, microstructural details, film composition, surface morphology, and so on. The structural information of the particles is critical in particle research, process development, and reliability analysis. There are different characterization techniques used on pure CeO_2 material are Ultraviolet–Visible Diffuse Reflectance Spectroscopy (UV-DRS), X-ray Diffraction (XRD), and Field Emission Scanning Electron Microscopy (FESEM). The pure CeO_2 material is successfully synthesised for various concentrations by using the co-precipitation method. The optical and photo-catalytic characteristics of pure CeO_2 nanoparticles were investigated. It shows the phase change and colour change in the material with change in the temperature of material. It confirms that the pure CeO_2 material enhanced it properties with different concentration.

Keywords: *Co-precipitation method, Cerium Nitrate, UV-DRS, XRD, FESEM etc.*

1. Introduction :

Metal oxides at the nanoscale are crucial in many fields of Physics, Chemistry, and Materials research. Metal elements may combine to generate a wide range of oxide compounds. These elements can have a wide range of structural geometries with an electrical structure that can be metallic, semiconductor, or insulator**[1]**. Metal oxides are utilised in the manufacture of microelectronic circuits**[2]**, sensors**[3]**, piezoelectric devices**[4]**, fuel cells**[5]**, corrosion-resistant coatings for surfaces**[6]**, and catalysts for technical applications**[7]**. An objective in the promising subject of nanotechnology is to create metal oxide nanoparticles with special properties with respect to those of bulk or single-particle species. Because of

their small size and high density of corner or edge surface sites, metal oxide nanoparticles can display unusual physical and chemical characteristics **[8]**. Rare earths are distinguished by a reduction in atomic and ionic radii, a phenomenon known as lanthanide contraction **[9].** Cerium has the chemical symbol Ce and the atomic number 58. Cerium is a silvery white, soft, ductile metal that tarnishes when exposed to air. Cerium is the second element in the lanthanide family, and while it frequently exhibits the series' distinctive $^{+}3$ oxidation state, it also possesses a stable $^{+}4$ state that does not oxidise water. It is also classified as a rare earth element. Cerium has no known biological purpose in humans, yet it is not extremely hazardous until exposed for an extended period of time **[10].** Despite usually existing in association with other rare-earth elements in minerals such as monazite and bastnäsite, cerium is simple to extract from its ores due to its unusual ability to be oxidised to the $^{+}4$ form in aqueous solution, which distinguishes it among the lanthanides. It is the most prevalent of the lanthanides, with neodymium, lanthanum, and praseodymium following. It is the 25th most prevalent element, accounting for 66 ppm of the Earth's crust, half that of chlorine and five times that of lead **[11].**Doped with Cadmium (Cd), CeO_2 nanoparticles are produced to compare the changes in the characteristics of pure CeO_2nanoparticles and establishing applications of resulted products**[12].**

2. Materials and Method :

In the Co-precipitation technique, Nucleation, Growth, and Agglomeration processes occur consecutively. This process entails precursor co-precipitation followed by thermal breakdown to the required composition. The chemical reactivity is produced by maintaining the pH using a precipitating agent. The molarities of the precursor and precipitating agent solutions, as well as the pH are critical factors in the co-precipitation process. The stoichiometric quantities of the base and dopant ingredients are dissolved in double-distilled water.

a) Preparing 0.3M Cerium Nitrate solution[$CeN_3O_9.6H_2O$] [AR Grade] by adding 6.52 gm of Cerium Nitrate in 50 ml distilled water with 10% ethanol [C_2H_5OH] in it.b) Preparing 0.9M NaOH[AR Grade] by adding 1.80 gm of NaOH in 50 ml distilled water with 10% ethanol. Stirring all threesolution side by side until whole solute is dissolved, adding NaOH by burette drop by drop inside stirring Cerium Nitrate solution.After above reaction is completed keep it for more than 12 hours to stabilize and get precipitate of insoluble $Ce(OH)_3$.Due to NaOH which acts as a precipitating agent for the solution, supernatant liquid is formed the reaction follows:

$$\mathbf{Ce(NO_3)_3{\bullet}6H_2O + 3NaOH \rightarrow Ce(OH)_3 + 6H_2O + 3NaNO_3}$$

Using a dropper, remove as much of the solution of $6H_2O + 3NaNO_3$ floating above the precipitate of Pure $Ce(OH)_3$ as feasible, after that remaining solution is kept in hot air oven between 100^oC. That removes almost all moisture and remaining product obtained is dried CeO_2 particles.Crushing obtained product in mortar and pestle for 2hrs until they feel soft, and heating it in muffle furnace about 400^oC to remove remaining impurities (Calcination Process) final product of Pure CeO_2 nanoparticles (with different concentration)are obtained.

3. Results and Discussions:

Characterization procedures provide a thorough insight of nanoparticles by evaluating their physical attributes such as crystal structure, microstructural details, film composition, surface morphology, etc. The structural information of the particles is critical in particle research, process development, and reliability analysis. There are different characterization techniques used on Cd-doped CeO_2 material such as Ultraviolet–Visible Diffuse Reflectance Spectroscopy (UV-DRS), X-ray Diffraction (XRD), and Field Emission Scanning Electron Microscopy (FESEM).

3.1 Ultraviolet–Diffuse Reflectance Spectroscopy Analysis :

UV-Diffuse reflectance spectroscopy (DRS) is a surface analytical technique. It uses ultraviolet (UV) light as a probing medium. The interaction of light with "strongly absorbing materials", such as metals, alloys, semiconductors, etc. occurs in the first 10-20 nm. In this report the UV-DRS of the nanomaterials were performed by using Jasco V-770. Data taken within range 200 - 1000 nm with data interval of 0.5nm, scan speed was at 400nm/min with continuous scan mode.

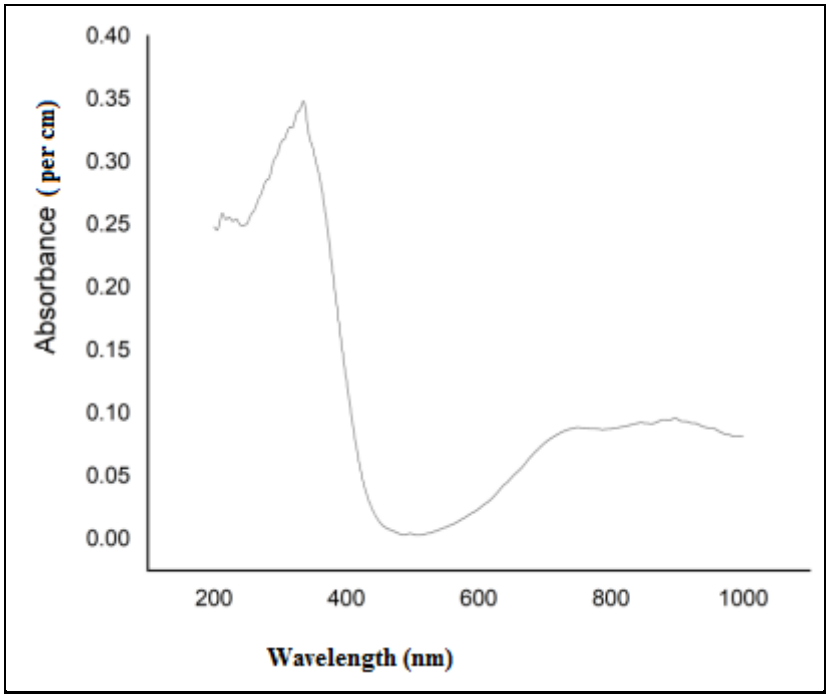

Fig-1: Absorbance of pure 0.3M CeO_2 Nanoparticles using UV-DRS

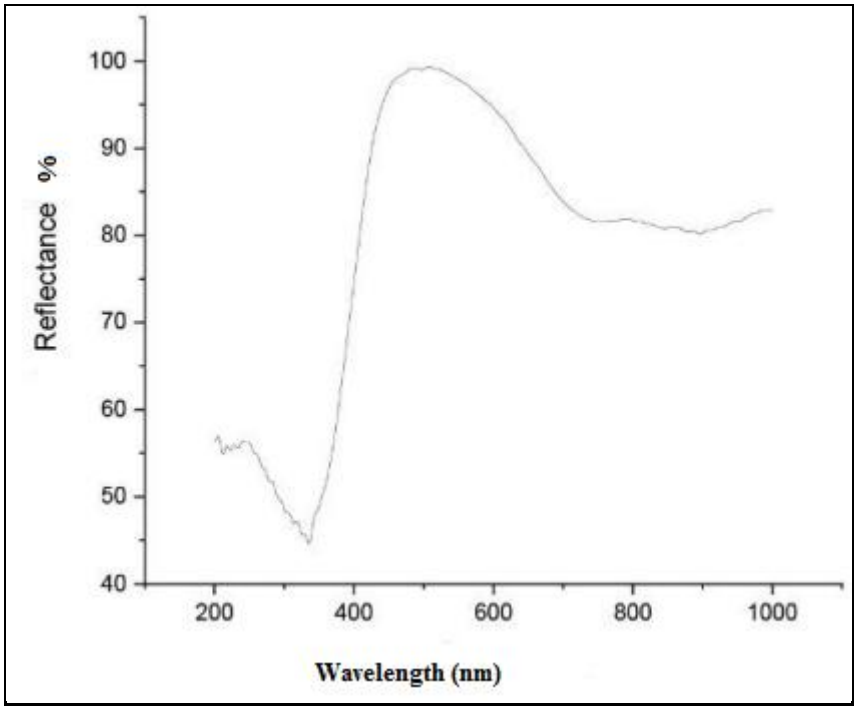

Fig-2: Reflectance of pure 0.3M CeO_2 Nanoparticles using UV-DRS

3.2 X-ray Diffraction Analysis :

Powder X-ray diffraction patterns of nanomaterials were recorded using an X-ray diffractometer employing CuKα radiation. The CuKα source was energized to 40 kV and 15 mA variable ant scatter and divergence slits were used to optimize the beam optics as a

function of θ. Diffraction data were recorded in a 2θ range from 20° to 80° in 0.02° steps for 0.6 s.

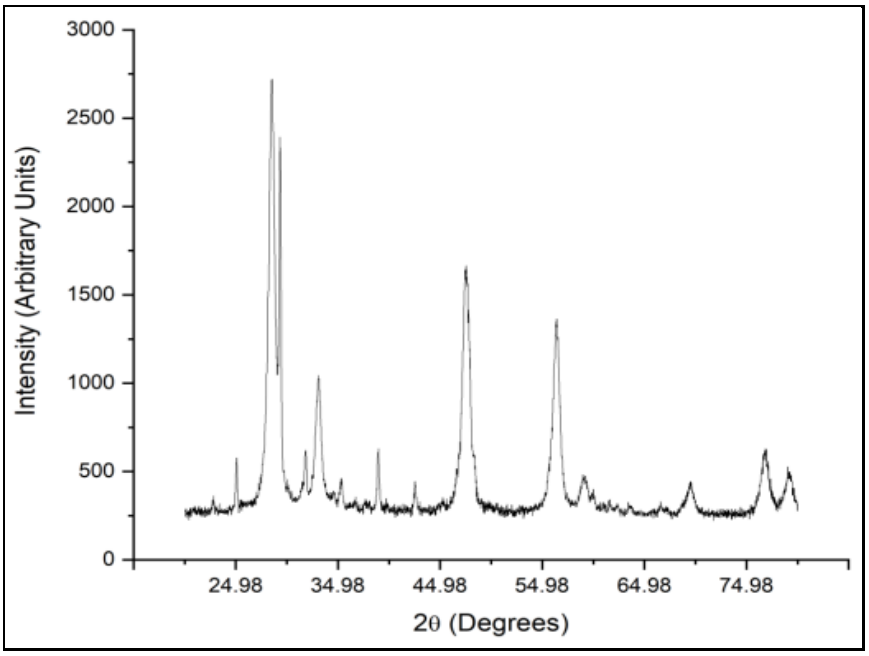

Fig-3: X-RD Pattern of Pure 0.3M CeO_2 Nanoparticles

3.3 Field Emission Scanning Electron Microscopy Analysis :

The Field Emission Scanning Electron Microscopyof the nanomaterials were performed by using Nova NanoSEM NPEP450. This instrument offers high resolution and excellent contrast at high, low and ultra-low voltage imaging, extended accelerating voltage ranging from 50 eV to 30 kV wide magnification range. The imaging use various accelerating voltages, beam currents, working distances and aperture settings to obtain the highest resolution possible.

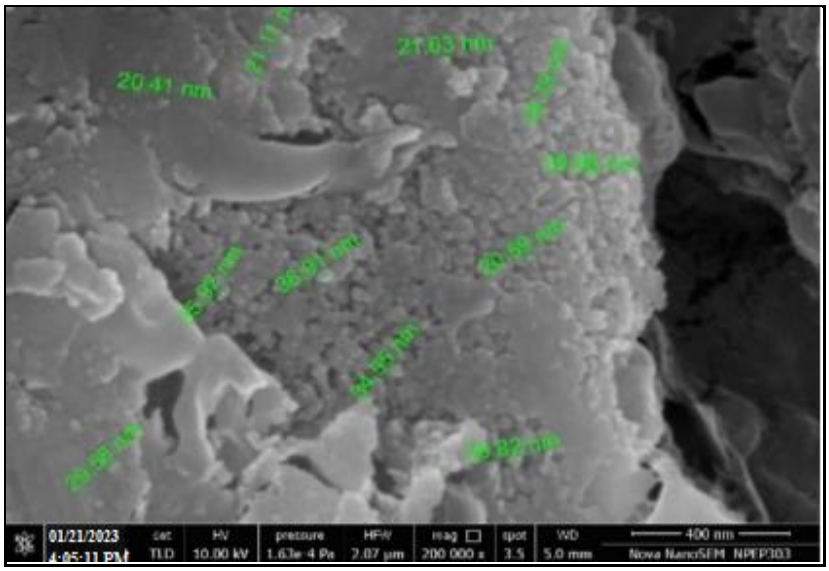

Fig-4 (a): Image of Pure 0.3MCeO_2Nanoparticles using FESEM

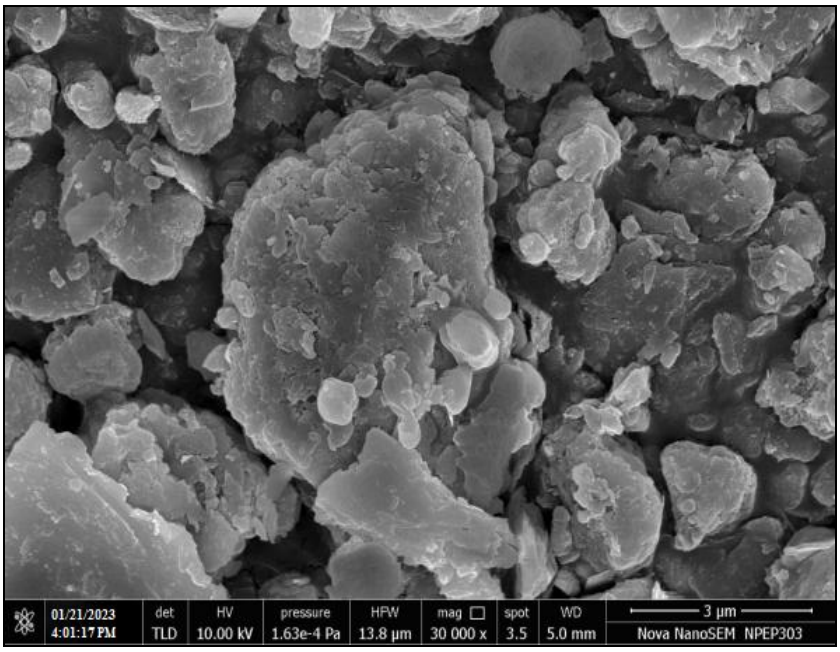

Fig-4 (b): Image of Pure 0.3MCeO_2Nanoparticles using FESEM

4. Conclusion :

The co-precipitation process is used to create the pure CeO_2 material. The synthesis material is made from a $CeN_3O_9.6H_2O$ solution for various concentrations by using the Co-precipitation method. The optical and photo-catalytic characteristics of pure CeO_2 nanoparticles were investigated. It shows the phase change and colour change in the material with change in the temperature of material. It confirms that the pure CeO_2 material enhanced it properties with different concentration.

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