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Article

Microwave Assisted Synthesis of Lanthanum Oxide

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ABSTRACT

Lanthanum oxides have attained significant interest for their diverse applications in hydrogen storage, electrodes, sorbent materials, gate insulators, and superconductors because of their multifunctional properties. In this present work, lanthanum oxide was successfully synthesized through the facile microwave-assisted chemical coprecipitation method and followed by calcination at 700°C. The crystal structure of the synthesized material was confirmed through X-ray diffraction (XRD), and average crystallite size was calculated from the XRD data based on the Debye–Scherrer equation. Fourier transform infrared spectroscopy (FT-IR) analysis confirms the presence of functional groups. XPS analysis was performed to examine the chemical composition. The surface morphology has been investigated using a field emission scanning electron microscopy (FE-SEM) technique.

Key words: Microwave, Coprecipitation, Characterization, Lanthanum oxide, Synthesis.

1. INTRODUCTION

Nanomaterials have been extensively investigated due to their potential applications in many scientific and technology-enhanced fields. The properties of nanomaterials are distinct from bulk materials and depend on their size and shape. Rare-earth elements are widely recognized for their high density, high melting point, and exceptional thermal conductivity. The unique physical and chemical properties of rare-earth elements arise from the presence of 4f orbital electrons, and have versatile applications in electronics, medical, and biomedical fields [1]. These materials find further application in microelectronic circuit manufacturing, sensor technology, piezoelectric devices, fuel cells, coatings for corrosion resistance, and catalysis. Rare earth metal oxides (MOs) are widely utilized as catalysts or sorbents in the chemical and petrochemical industries to eliminate CO, NOx, and Sox compounds produced during fossil fuel combustion [2-4]. Metal oxides play a key role in the semiconductor industry, where they are used to produce a variety of electronic components, such as computer chips [5]. La_2O_3 is regarded as one of the most attractive oxide semiconductors because of its exceptional chemical and physical properties. It features a wide band gap of 5.5 eV, is thermally stable, non-toxic, and has a high relative dielectric constant [6]. As a result, lanthanum oxide nanoparticles show promise for applications in rechargeable batteries, fuel cells, catalysts, optical devices, magnetic storage, MRI, dielectric layers, gas sensors, biosensors, biomedicine, optical coatings, photoelectric conversion, and catalysis [7-11]. La_2O_3 nanostructures such as nanoneedles, nanobundles, nanorods, nanowires, nanosheets, and nanopowders have been synthesized using several methods, including the hydrothermal method [12], chemical precipitation method [13], microwave method [14], sol–gel method [15], sonochemical method [16], solvothermal method [17], arc discharge method [18], and laser deposition method [19].

In this work, La_2O_3 was synthesized by a microwave-assisted chemical coprecipitation method, with lanthanum (III) chloride and sodium hydroxide as the starting materials. The structural and chemical properties of the synthesized material are characterized by Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), and field emission scanning electron microscopy (FE-SEM).

2. EXPERIMENTAL DETAILS

2.1. Materials

Lanthanum (III) chloride heptahydrate ($LaCl_3 \cdot 7H_2O$), and sodium hydroxide (NaOH), all chemicals were purchased from Merck India Pvt. Ltd.

2.2. Preparation of Lanthanum oxides (La_2O_3) by Microwave-Assisted Coprecipitation Method

Lanthanum oxides (La_2O_3) were synthesized through the microwave coprecipitation method. 0.5 molar solutions of lanthanum (II) chloride were stirred in a magnetic stirrer for 15 min. Then NaOH was subsequently added to the metal chloride solution. After the complete addition of NaOH, it is then stirred for about 15 min and heated in a microwave oven for about 5 min. The metal hydroxide formed is cooled, filtered, and dried in an air oven at 80°C for 24 h. The metal hydroxide is converted into metal oxide, lanthanum oxides (La_2O_3), by heating it in a muffle furnace at 700°C for 7 h at a heating rate of 2°C/min.

The chemical equation for the formation of La_2O_3 is as follows:

$$LaCl_3 + 3NaOH \rightarrow La(OH)_3 + 3NaCl$$

$$2La(OH)_3 \rightarrow La_2O_3 + 3H_2O$$

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2.3. Characterizations

The functional groups present in the sample are investigated using FT-IR spectroscopy, carried out in the Perkin-Elmer "Spectrum Two" FT-IR spectrometer. X-ray diffraction (XRD) studies of the La_2O_3 performed by Rigaku Miniflex-600 benchtop diffractometer with a Cu Kα radiation source (λ = 1.542 Å) in the range 10−90°. The chemical composition is examined by XPS analysis performed in Thermo Fisher Scientific ESCA – Laboratory, X-ray photoelectron spectrometer. The surface morphology of the sample is examined using FE-SEM (CARL ZEISS USA, resolution 1.5 nm).

3. RESULTS AND DISCUSSIONS

3.1. FT-IR Analysis

The FT-IR spectra of the La_2O_3 measured in the wavenumber range 4000–400 cm^{-1} is shown in Figure 1. The vibrational peak appears at 3611 cm^{-1}, confirming the presence of O–H stretching vibrations associated with absorbed moisture on the surface of the prepared compound [20]. The absorption peaks at 1440 and 1360 cm^{-1} are ascribed to the O–H vibration in absorbed water on the sample surface [21]. The absorption peak appears at 643 cm^{-1} due to the La–O stretching vibration [22]. The presence of these bands, therefore, confirmed the presence of the La_2O_3 in the synthesized nanoparticles.

3.2. XRD Analysis

The XRD patterns (Figure 2) of the synthesized material exhibit the characteristic diffraction peaks corresponding to the hexagonal phase structure of La_2O_3 (JCPDS card no. 73–2141). The diffraction peaks observed at 26.99°, 29.74°, 44.21°, 47.96°, and 54.93° correspond to the (100), (011), (003), (110), and (103) planes, respectively. The narrow and strong diffraction peaks confirm the highly crystalline nature of the synthesized material.

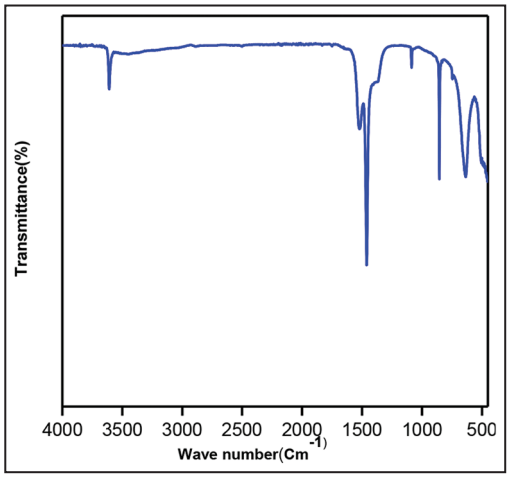

Figure 1: FT-IR Spectrum of La_2O_3.

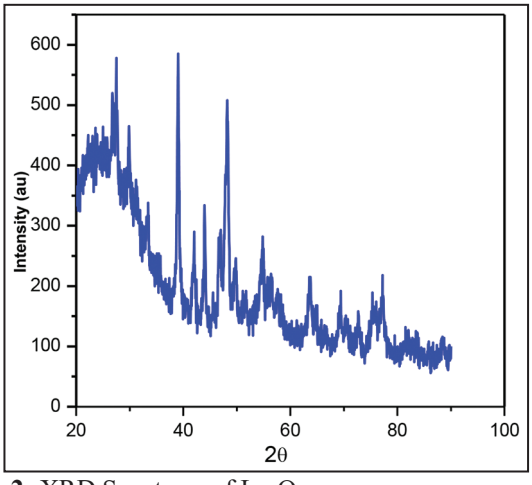

Figure 2: XRD Spectrum of La_2O_3.

The average crystallite size of La_2O_3 is calculated using the Debye–Scherrer Equation (1)

$$D = \frac{0.9\lambda}{\beta \cos\theta} \quad \rightarrow \quad (1)$$

where λ (1.5418 A°) is the wavelength of X-ray used, β represents the full width at half maximum of the peak under consideration, and θ is the Bragg's angle of diffraction (in radians). Thus, the average crystallite size of synthesized material is calculated to be 14 nm.

3.3. XPS Analysis

XPS analysis was performed and is shown in Figure 3. The XPS survey scan spectrum of the La_2O_3 is shown in Figure 3a, confirming the existence of La and O elements. The energy spectrum of La 3d is shown in Figure 3b; exhibited peaks with binding energies at 834.8 and 838.7 eV correspond to the La 3d 5/2 spin-orbital peaks. The additional peaks with binding energies situated at 851.8 and 855.5 eV were assigned to the $La3d_{3/2}$ spin-orbit peaks. These results indicate that lanthanum ions are present in the trivalent state (La^{3+}) [23]. The binding energy at 530.68 eV is related to the O^{-2} of oxygen in La_2O_3 [24].

3.4. Surface Morphology Analysis

The surface morphologies of the La_2O_3 were investigated using the FE-SEM analysis. La_2O_3 nanoparticles at different magnifications are shown in Figure 4. The micrograph demonstrates the nanoflake-like morphology with polyhedral aggregates of fine particles having enhanced available surface area, which improves the performance of the compound. This accumulation of the nanoparticles was caused by high surface energy, due to their high surface-area-to-volume ratio [25]. The energy-dispersive X-ray analysis (EDAX) spectrum of the synthesized compound is shown in Figure 4d and is used for the elemental

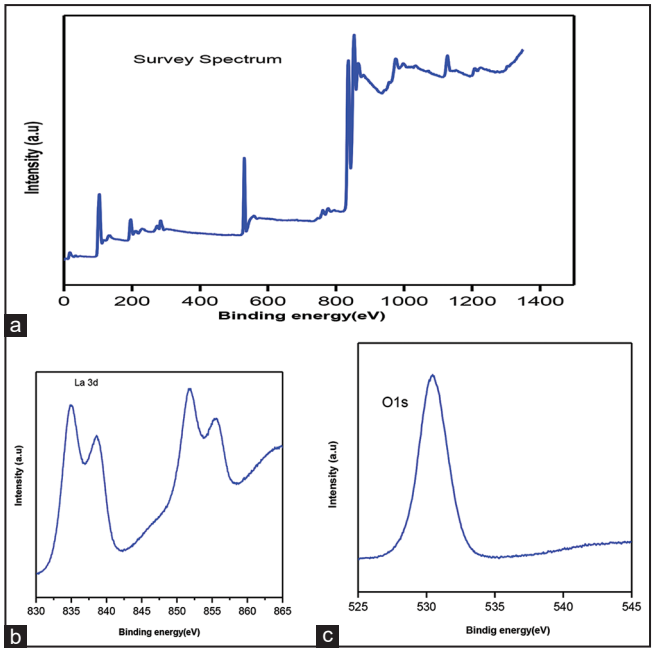

Figure 3: XPS spectra for the synthesized La_2O_3: (a) a total survey (b) La 3d (c) O 1s.

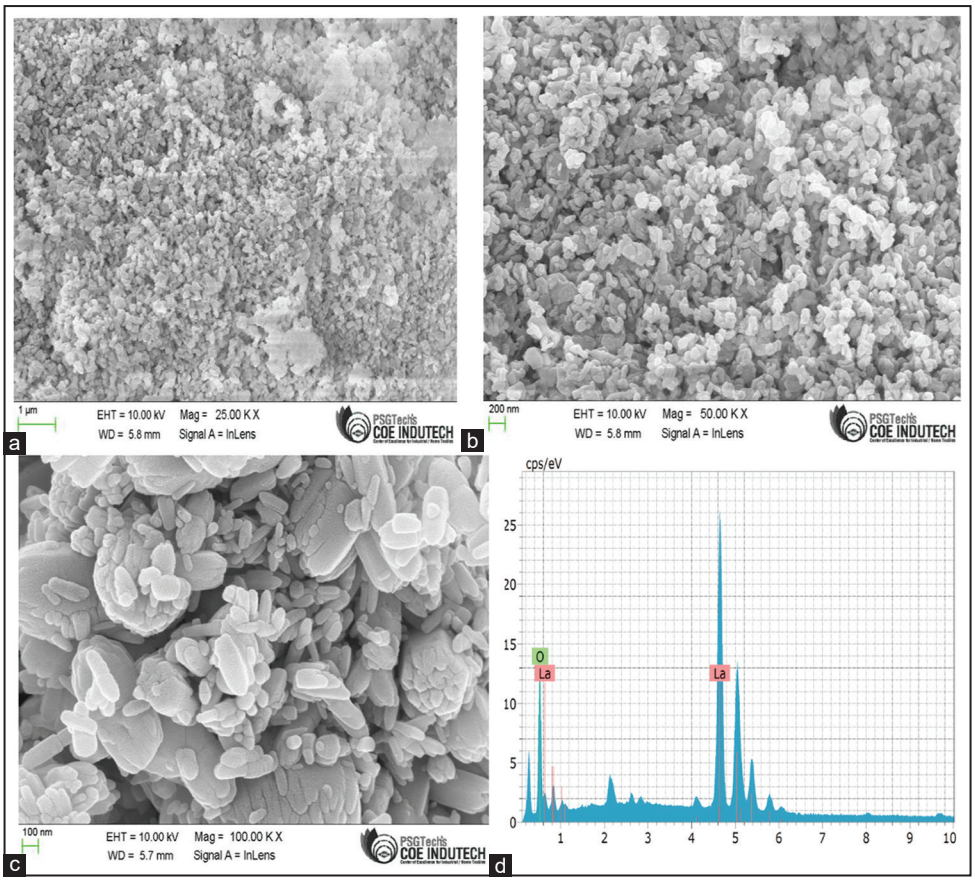

Figure 4: FE-SEM micrographs of La_2O_3 at different magnifications (a), (b), (c) and Edax spectrum (d).

composition study. It is examined that the highest weight percentage of lanthanum (La) is present in the sample along with a sufficient amount of oxygen (O). From the EDAX results, it is analyzed that the average weight percentage of lanthanum is 81% and oxygen is 18%, which is in good agreement with the theoretical weight percentage value.

4. CONCLUSIONS

La_2O_3 nanoparticles were successfully synthesized by microwave-assisted chemical coprecipitation. It is characterized by IR, XRD, XPS, and FE-SEM. FT-IR spectra reveal the presence of the characteristic vibrational peak of the La_2O_3. XRD diffraction peaks confirm the presence of the hexagonal phase structure of La_2O_3, and the average crystallite size of the synthesized material was calculated to be 14 nm. The morphology of the sample was studied by FE-SEM analysis, which demonstrates the nanoflake-like morphology with an accumulation of fine particles with enhanced available surface area. The Edax study confirmed the presence of oxygen (O) and lanthanum (La) in the synthesized nanoparticles. Thus, the synthesized lanthanum oxide may be used in photocatalysis, electrocatalysis, fuel cells, electrochemical sensing, and as electrode material for supercapacitor applications. Thus, the synthesized material has versatile applications in gas sensors, catalysis, fuel cells, dielectric layers, rechargeable batteries, photoelectric conversion, water treatment, and as electrode materials for energy storage systems.

5. ACKNOWLEDGMENTS

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6. CONFLICT OF INTEREST

The author declares that there is no conflict of interest regarding the publication of this article.

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***Bibliographical Sketch**

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