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## Hydroxyl radical scavenging activity of La<sub>2</sub>O<sub>3</sub> nanoparticles

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### Abstract

In the present investigation has purely narrowed on the synthesis and characterization of lanthanum oxide nanoparticles by the method of Co-precipitation. The prepared lanthanum oxide powder was synthesized from the raw material of lanthanum nitrate and sodium hydroxide. This study mainly focused on the reducing property of prepared lanthanum oxide nanoparticles thereby reducing the hydroxyl radicals. It was experimentally demonstrated by the following techniques such as Fourier transform infrared spectroscopy and X-ray diffraction. The XRD analysis clearly revealed that the intensified hexagonal "Lanthania" is formed and the nanoparticles are poly-crystalline in nature. The morphological studies and the size of the prepared nanoparticles can be examined through the Field Emission Scanning Electron Microscopy and the percentage composition of metal present in the prepared lanthanum oxide nanoparticles can be exactly assured from the Energy Dispersive X-ray Analysis and elemental mapping.

**Keywords:** lanthanum oxide, hydroxyl radicals, lanthania, nanoparticles, EDAX etc.

### 1. Introduction

Now-a-days, there is lot of innovative invention is going on the field of preparation of metal oxide nanoparticles [1]. From that, we have focussed on the lanthanum metal oxide nanoparticles with specific size and its morphological studies. Due to their innumerable physical and chemical properties, it is used in various fields such as catalysis, solar cells, photo detectors, sensors, light emitting diodes and laser communication, made them attractive and more efficient materials [2-5]. Preparation of various nanomaterials have many different synthetic way of approach such as chemical vapour deposition (CVD), thermal evaporation, chemical bath deposition, laser ablation, hydrothermal, homogeneous precipitation in an organic matrix, sonochemical, Co-precipitation and sol-gel method.

Rare earth elements are used as an attractive materials in industry and play an important role in a number of current technologies as more active components. Among the rare earth elements, lanthanum metal show its own unique and prominent properties and it is prepared as lanthanum oxide nanoparticles. Lanthanum oxide (La<sub>2</sub>O<sub>3</sub>) powders have lot of industrial oriented applications which is very attractive are as follows: Nano-sized lanthanum oxide is very much useful in the synthesis of highly refractive optical fibers, Nano-lanthanum oxide can be used for the preparation of organic chemical products catalysts, Nanometer Lanthanum oxide can enhance the burning rate of propellant and also Nano-lanthanum oxide can be used in the field of electrochemistry as an electrode materials and it is also used in light-emitting material (blue powder), hydrogen storage materials, laser materials etc [6-8].

Sensitive oxygen species (ROS) which include the hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), superoxide radical anion (O<sub>2</sub><sup>•-</sup>), and hydroxyl radical (•OH). ROS have the tendency to react with a large variety of easily oxidizable cellular biological components. Among ROS, •OH radical exhibits the strongest oxidative activity towards cellular tissues. Non-specifically toxic hydroxyl radical can oxidize all functional sensitive groups of giant bio-molecules including fat, amino acids and ribose sugar at essentially diffusion-controlled reaction rates. Therefore, •OH radical ruptures the texture of viable cells and it leads to promote many diseases including Alzheimer's disease, rheumatic arthritis, atherosclerosis, breast cancer and ageing [9]. As a result of the investigation, hydroxyl radical scavenging assay delivers the lead role and it was a task which owing to the high potent activity, lower the strength of •OH and short life span. In this present study, we report on the preparation of lanthanum oxide (La<sub>2</sub>O<sub>3</sub>) nanoparticles by Co-precipitation method and also report the hydroxyl radical scavenging activity of the prepared La<sub>2</sub>O<sub>3</sub> NPs.

## 2.1 Materials

Fischer make, AR grade (99.9%) Lanthanum Nitrate and NaOH was used as precipitant in the La<sub>2</sub>O<sub>3</sub> NPs.

## 2.2 Methods

### 2.2.1 Preparation of lanthanum oxide nanoparticles

About 25 mL of 0.1 M La (NO<sub>3</sub>)<sub>3</sub> was added drop wise to an aqueous solution of Sodium Hydroxide (25 mL, 1.0 M), making a final volume of 100mL. The mixture was stirred well and refluxed at a moderate temperature for 2 hrs using magnetic stirrer. The sample was collected by Eltek high speed centrifugation machine, washed several times with double distilled water and dried over for 2-3 days at room temperature. Finally the obtained sample was grinded using mortar and pestle and finally it was allowed to calcinate at 300 °C for 2 hrs.

### 2.3 Hydroxyl radical scavenging assay (HRSA)

HRSA was one of the dynamic reactive oxygen species in the living cells. It binds with phospholipids which are polyunsaturated fatty acid moieties present in the cell membrane and it paved a lethal damage to the cell.

#### 2.3.1 Reagent preparation

Iron-EDTA solution was prepared by mixing 0.13% ferrous ammonium sulphate and 0.26% EDTA. 0.018% EDTA was prepared by dissolving 0.018g EDTA in 100ml distilled water. 0.22% ascorbic acid was prepared by dissolving 0.22g ascorbic acid in 100ml distilled water. 17.5% TCA was prepared by dissolving 17.5g TCA in 100ml of distilled water. Nash reagent was prepared by adding 7.5g ammonium acetate, 0.5ml of glacial acetic acid and 0.2ml of acetone to 100ml distilled water.

#### 2.3.2 Procedure

Various concentrations of extract (250, 500, 750, 1000µg) were taken and 1ml of iron EDTA solution, 0.5ml of EDTA solution, 1ml of DMSO and 0.5ml of ascorbic acid were added to it. The mixture was incubated in a boiling water bath at 80 to 90°C for 15 min. After incubation, 1ml of ice cold TCA and 3ml of Nash reagent were added and the reaction mixture was incubated at room temperature for 15 min. The

absorbance was read at 412 nm. The % hydroxyl radical scavenging activity is calculated by the following formula,

$$\% \text{ Inhibition} = \frac{\text{Absorbance (Control)} - \text{Absorbance (Test)}}{\text{Absorbance (Control)}} \times 100$$

## 2.4 Characterization

FT-IR spectra were taken using SCHIMADZU Fourier Transformer Infrared Spectrometer, model - IR Affinity - 1 at Department of chemistry, V.O. Chidambaram College, Tuticorin. XRD Analysis was done at Kalasalingam University using D8 Advance ECO XRCD Systems with SSD160 1D Detector (Bruker). FESEM analysis was performed on an MIRA3 TESCAN (Japan) model with maximum magnification of 600,000X and resolution of 1.5 nm at Avinasilinagam University, Coimbatore. EDX analysis was performed on Quanta 200 with x-Flash (England) at Avinasilinagam University, Coimbatore.

## 3. Results and Discussion

### 3.1 Fourier Transformer infrared spectroscopy

FT-IR spectra can illustrate the precise structural detail of the Lanthanum oxide NPs. Fourier transform infrared spectrum has been recorded for the as-prepared sample. The spectrum was measured in the wavelength ranges 400 - 4000 cm<sup>-1</sup>. From the FT-IR spectrum, one can confirm that the absorption is due to the molecular vibration. The frequency of various functional groups present in the prepared sample can be studied with the position of the vibration peaks present in the spectra. The absorption peak at 3,329 cm<sup>-1</sup> was corresponds to the stretching vibration of the O-H bond and the bending vibration of H-O-H from water molecules on the external surface, and the absorption bands at 1,463 and 1,384 cm<sup>-1</sup> were attributed to the asymmetric stretching mode of CO<sub>3</sub><sup>2-</sup> group. It was highly probable that all CO<sub>3</sub><sup>2-</sup> groups occupy more or less identical sites in the crystal lattice, so the lanthanum oxide precursor might be lanthanum carbonate [10]. A sharp peak was observed at about 467, 669, 854, 1020, and 1463 cm<sup>-1</sup> which can be ascertained to stretching vibration of the La-O bond. The obtained peaks are in match with the earlier reported values as shown in the fig.1 thus confirming the formation of La<sub>2</sub>O<sub>3</sub> phase [11].

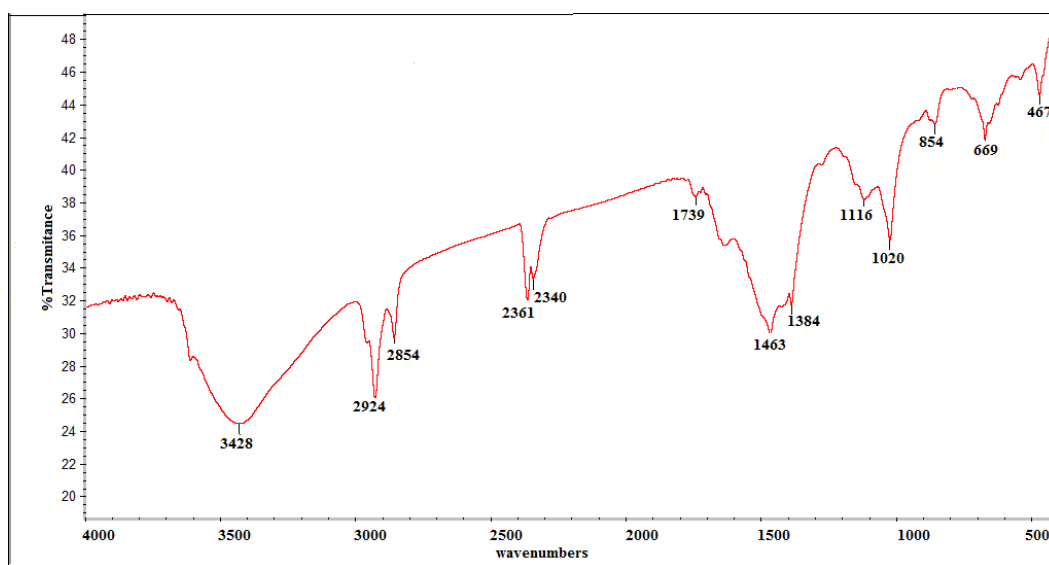


Fig 1: FTIR spectrum of lanthanum oxide NPs

### 3.2 X-ray diffraction pattern (XRD)

The X-ray diffraction studies insist the properties such as crystal size and its nature. The X-ray diffraction pattern was recorded at  $2\theta$  values between  $0^\circ$  and  $80^\circ$  are represented in fig.2. Lanthanum oxide has 3 prominent peaks at  $2\theta$  values 28.1268, 39.7072 and 48.7199 respectively. The Diffraction Peaks was observed at  $28^\circ$ ,  $39^\circ$  and  $48^\circ$  which corresponds to the (h k l) values of the peaks (4 0 0), (4 0 0) and (6 2 2) respectively [12]. The lattice parameters were in good agreement with JCPDS card number 04 - 0856 having lattice parameters  $a=b=c=11.420$ . In the XRD patterns, we can noticed the as-prepared NPs was exist in a hexagonal  $\text{La}_2\text{O}_3$  NPs. In this spectrum, the intensity hexagonal “Lanthania” is formed which is so small component which clearly indicates this phase is poly-crystalline into “Lanthania” phases [13]. The crystallite size was calculated by Debye-Scherrer’s formula,

$$D = K\lambda / \beta \cos\theta \quad \dots (1)$$

Where, D is the average crystallite size of the particle,  $\lambda$  is the wavelength of the radiation,  $\beta$  is the full width at half maximum (FWHM) of the peak,  $\theta$  is the Bragg’s angle. The average crystallite sizes of samples synthesized by wet chemical method are 8.66 nm.

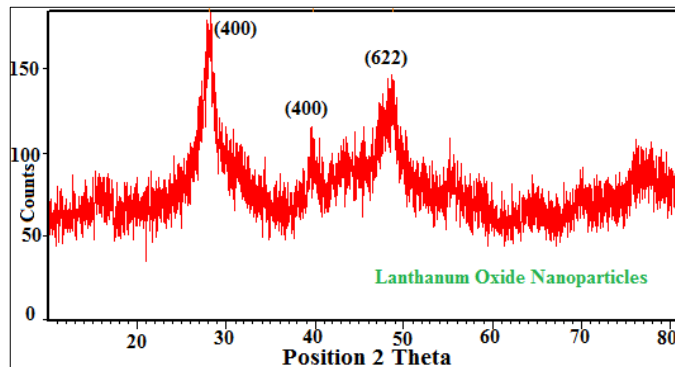


Fig 2: XRD spectrum of lanthanum oxide NPs

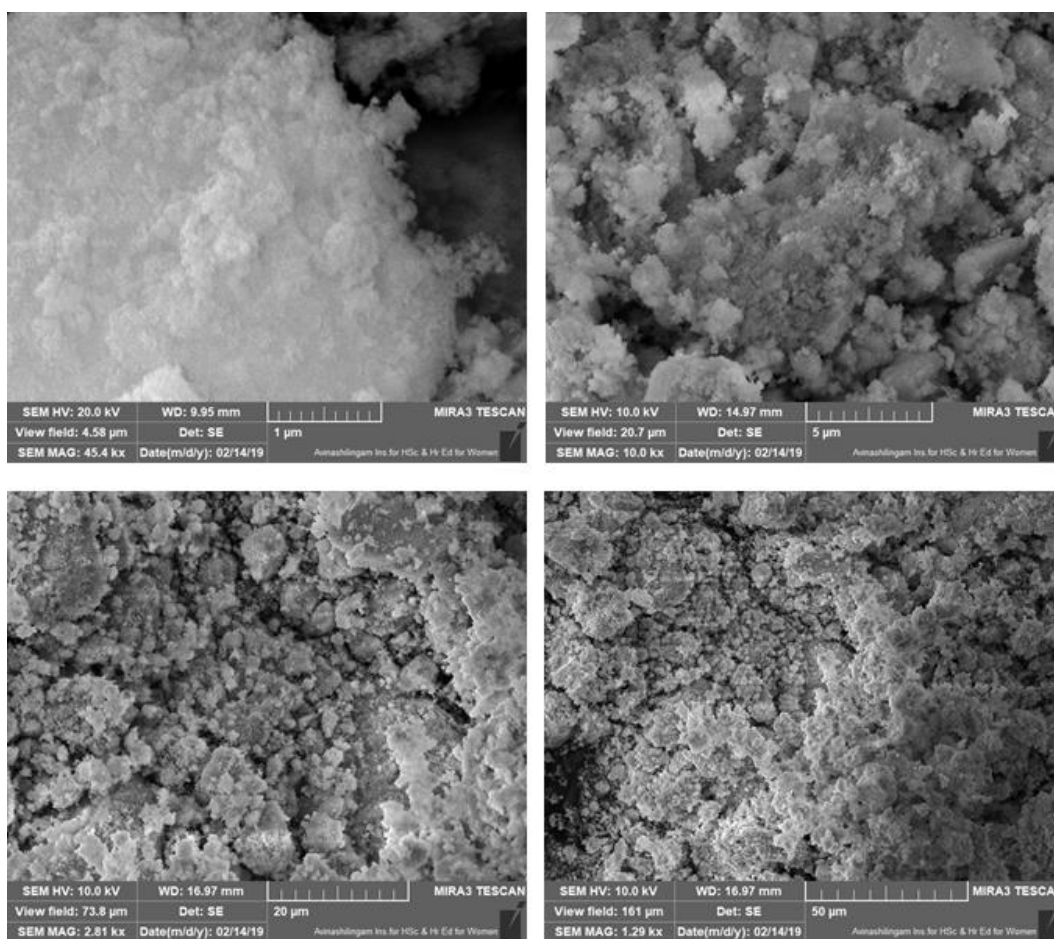


Fig 3: FESEM image of Lanthanum Oxide NPs

### 3.3 Field emission scanning electrom microscopy (FESEM)

The morphology and size of the prepared lanthanum oxide nanoparticles was studied by FESEM analysis. Fig.3. shows the FESEM images of lanthanum oxide at different magnification. The pictorial representation of FESEM clearly illustrate that the  $\text{La}_2\text{O}_3$  NPs formed are polydispersed in nature and almost spherical shape [14].

### 3.4 EDAX with dot mapping analysis

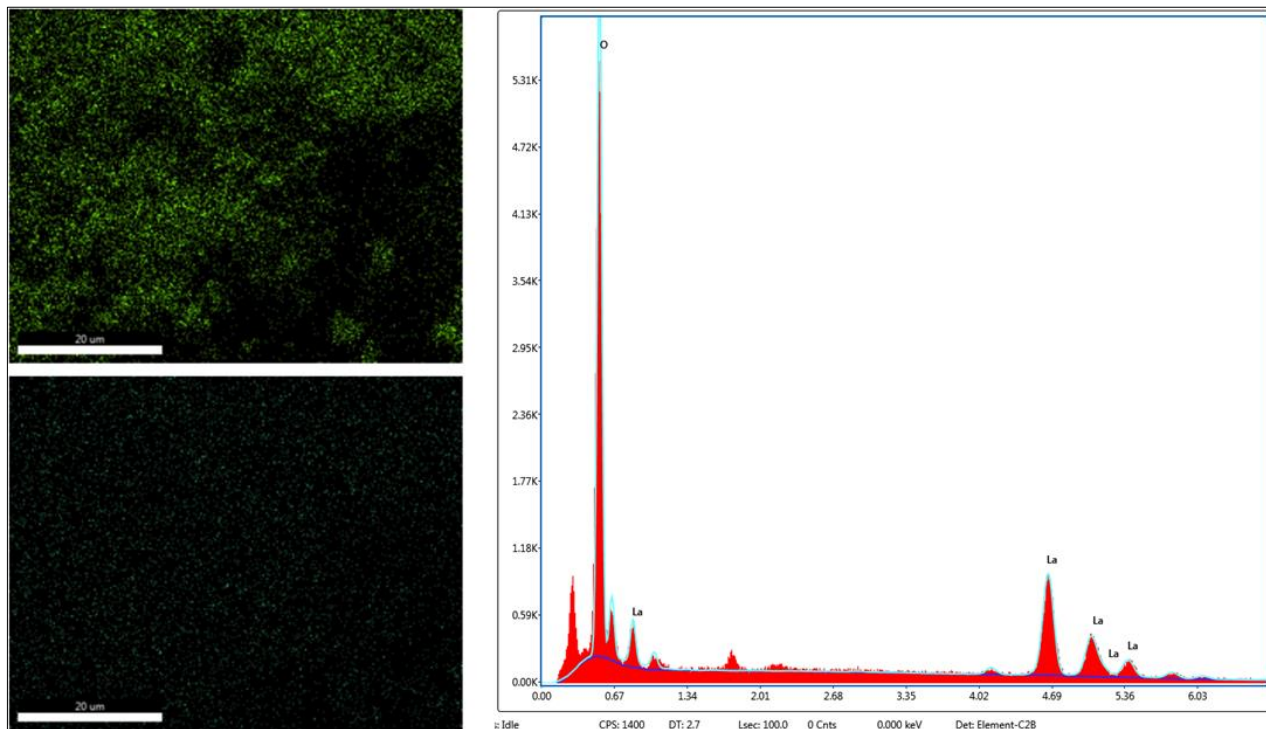
Chemical purity and stoichiometry of the  $\text{La}_2\text{O}_3$  sample was

investigated by EDAX analysis. The energy repulsive spectrum of precipitated  $\text{La}_2\text{O}_3$  is given in fig.4. Generally peak related to lanthanum was found at 4.650 KeV. In our case it is present at 4.69 KeV. EDAX study confirms the presence of La and O in the synthesized  $\text{La}_2\text{O}_3$ . X-Ray mapping results were also obtained which shows the distribution of all the elements present in the microstructure. Energy Dispersive Spectroscopy (EDS) results obtained shows the quantitative analysis of the microstructures present in the samples. Fig.4. shows Elemental X-Ray mapping of  $\text{La}_2\text{O}_3$  sample. X-ray elemental mapping reveals that



spherically porous microstructure image was observed in  $\text{La}_2\text{O}_3$ . Oxygen is rich in the matrix (72%) and lanthanum

(28%) are unequally distributed.



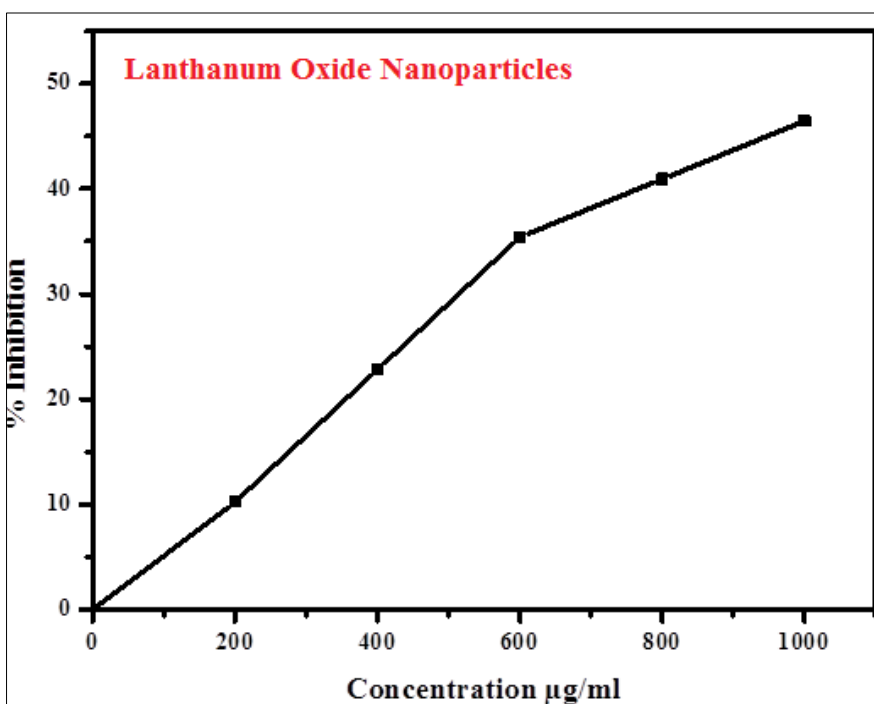
**Fig 4:** EDAX spectrum and element mapping of lanthanum oxide NPs [Green - La and Brown –Oxygen]

**3.5 Hydroxyl radicals scavenging activity [HRSA]**

The Lanthanum oxide nanoparticles has the ability to neutralize the hydroxyl radicals by reducing it. It was found that the Lanthanum oxide nanoparticles exhibited 46.46 % inhibition at concentration of 1000 µg/ml. The HRSA of lanthanum oxide NPs (Fig.5.) absorbance measured at 412 nm, while decreasing absorbance the inhibition activity [15] also increased it leads to increasing antioxidant activity as prepared NPs. The HRSA result are presented in table.1.

**Table 1:** HRSA of lanthanum oxide NPs

Sample	Concentration µg/ml	OD @ 412 nm	% Inhibition
Control	0	1.270	0
Lanthanum oxide	200	1.140	10.24
	400	0.980	22.83
	600	0.820	35.43
	800	0.750	40.94
	1000	0.680	46.46



**Fig 5:** HRSA of Lanthanum Oxide NPs

#### 4. Conclusions

Lanthanum oxide nanoparticles were synthesized by Co-Precipitation Method. FTIR spectra clearly reveals that the peak at  $476\text{ cm}^{-1}$  corresponding to La-O stretching vibration. The XRD pattern confirms the polycrystalline nature of the nanoparticles. The pictorial representation of FESEM images shows that the particles are spherical in shape. The distribution of lanthanum and oxygen in the matrix was précised from dot mapping analysis. From the information of HRSA, we conclude that the increasing the NPs concentration predominantly increasing the hydroxyl radical activity. So the dangerous disease like cancer, Alzheimer's disease, atherosclerosis and rheumatic arthritis can be prevented.

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