

**INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH
TECHNOLOGY****EFFECT OF ANNEALING TEMPERATURE ON THE PROPERTIES OF
LANTHANUM OXIDE (La₂O₃) NANOPATES BY REFLUX ROUTES****S. Karthikeyan¹ & M. Selvapandiyan^{*2}**^{1&*2}PG and Research Department of Physics, Periyar University PG Extension Centre, Dharmapuri
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ABSTRACT

Two-dimensional (2D) crystals, possessing a nano scale dimension have been considered as important new materials due to their unique properties and potential applications in electronic and catalysis areas. In this work lanthanum oxide (La₂O₃) nanoplates were synthesized by a simple reflux method. The approximate band gap of the as-prepared and calcinated samples as calculated from the absorption spectrum are 5.7 and 5.8 nm respectively. Lanthanum oxide (La₂O₃) nanoparticles were obtained from as-prepared and calcinations of the La₂O₃ nanoparticles. The as-prepared and calcinations products were characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM), Fourier transform infrared (FT-IR), Band gap was calculated by UV-visible spectroscopy.

Keywords: La₂O₃ nanoplates, Reflux method, Monoclinic structure, Optical band gap.**I. INTRODUCTION**

The synthesis, production and manipulation of materials on the nanoscale is currently one of the favorable areas of research which also attracts the industrialists for designing and fabricating new functional materials with novel special properties [1-2]. Because of their unique electronic configuration [4f electron] lanthanides have been applied in various fields, also these lanthanide-based materials have attractive and interesting magnetic [3], optical [4-5] electrical and therapeutic [6] properties. Among the lanthanide, lanthanum oxide has been extensively examined for its unique properties [7-8]. Lanthanum has been synthesized in various compositions such as La(OH)₃ [11], LaF₃ [5], La₂(CO₃)₃, LaPO₄ [9-10]. In this work lanthanum oxide nanoparticles were successfully prepared from the reaction of lanthanum nitrate and urea by using reflux method. Then, lanthanum oxide nanoparticles were prepared from calcinations of resulted product 500°C. Also effects of weight ratio or molar ratio of lanthanum nitrate and urea on size and morphology of products were examined. The main advantage of these methods are increased homogeneity and high surface area of the resulting powders, which lead to relatively high reactivity and hence low sintering temperatures. There are not many reports on preparation of La₂O₃ nanoparticles [12]. Herein the preparation of lanthanum oxide nanoplates by reflux method has been reported and the effect of calcinations has been investigated and reported. As-prepared and Calcinated products were characterized by powder X-ray diffraction (XRD), Scanning Electron Microscopy (SEM), FT-IR and UV-Vis Spectroscopy.

II. EXPERIMENTAL PROCEDURE

All the materials were obtained from commercial suppliers and were used without further purification. Lanthanum oxide (La₂O₃) has been synthesized by a simple reflux method using Lanthanum nitrate (LaN₃O₉.6H₂O) and Urea (NH₂CONH₂) as starting materials. In a typical synthesis, 0.01 mole of Lanthanum nitrate and 0.01 M of Urea were dissolved in 500 ml double distilled water. The precursor solution was transferred into a round bottom flask and maintained at a constant temperature of 120°C for 24 hr. The final products were collected by sample washing the precipitate for several times with double distilled water. Finally, the as-prepared nanoparticles were calcinated at 500°C for 1 hr. The as-prepared and calcinated samples were characterized by XRD, SEM, FTIR, and UV-Visible analysis.

III. RESULTS AND DISCUSSION

1. Structural Analysis

The crystal structure of the lanthanum oxide was characterized by XRD. Fig.1a shows the XRD pattern of the lanthanum oxide sample prepared with 0.01 mole concentrations. The maximum orientation of the crystallites is along (1 0 1) plane. Fig.1a displays the crystalline structure and phase purity of the as-prepared La_2O_3 . One can conclude from the appearance of more than one prominent peak that the prepared lanthanum oxide samples are polycrystalline in nature. The position and relative intensities of all diffraction peaks match well with the pure La_2O_3 with monoclinic crystal structure according to the standard JCPDS card #50-0602. The exposure to higher temperature during calcinations has resulted in increased crystallinity as can be witnessed from the increased XRD peak intensities. The crystallite sizes as calculated from the XRD patterns using Debye Scherer formula were 5 nm and 24 nm for as-prepared and calcinated samples respectively.

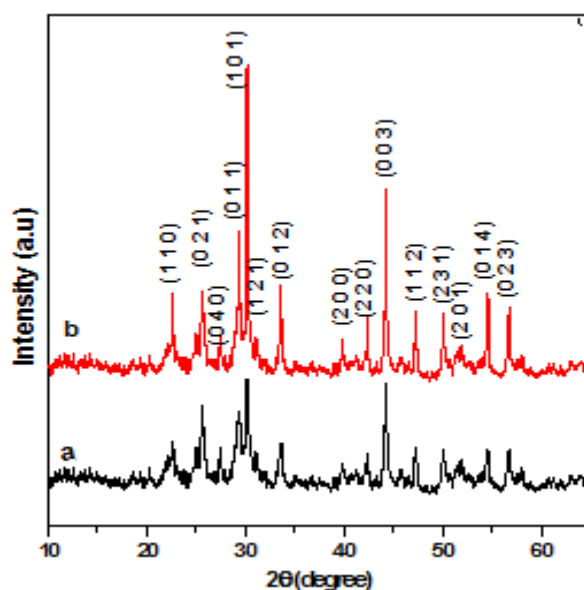


Fig.1 XRD pattern of La_2O_3 nanoparticles a) as-prepared and b) calcinated

2. Morphological Analysis

The morphology of the prepared Lanthanum Oxide (La_2O_3) nanoparticles were studied using scanning electron microscope model SEM Quanta 200. Fig.2a corresponds to the as-prepared La_2O_3 nanoparticles. Fig.2a shows uniform smooth surfaced nanoplates with thickness ranging 50-80 nm. Fig.2b corresponds to the SEM images of La_2O_3 nanoparticles calcinated at 500°C for 1 hour. The breaking up of plate like morphologies is witnessed and non-uniform structures appear on exposure to higher temperature. Thus on calcinating the as-prepared sample, the uniformity of the sample is lost and also there prevails a wide particle size distribution which is not quite encouraging.

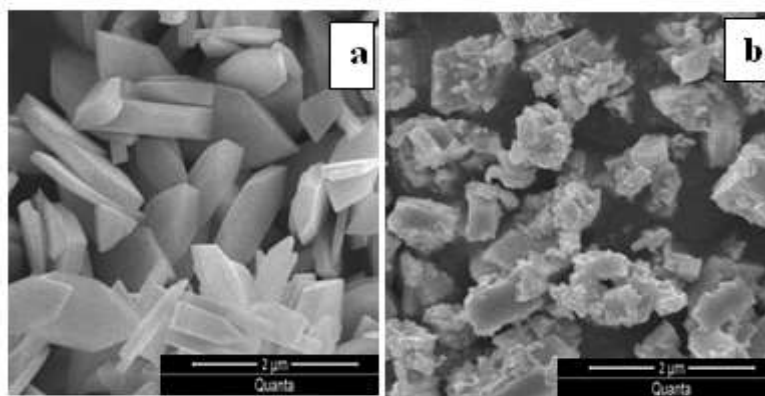


Fig.2 SEM images of La_2O_3 nanostructures a) as-prepared and b) calcinated

3. Vibrational Analysis

Fourier transform infrared spectrum has been recorded for the as-prepared and calcinated samples. The spectrum was recorded in the wavelength ranges 500 - 2000 cm^{-1} . From the FTIR spectrum, one can observe the absorption due to the molecular vibration. The frequency of various functional groups present in the prepared sample can be predicted with the position of the vibration peaks. A sharp peak was observed at about 682, 807, 887, 1077, 1467, and 1628 cm^{-1} which can be attributed to stretching vibration of the La-O bond. The obtained peaks are in match with the earlier reported values as shown in the table thus confirming the formation of La_2O_3 phase.

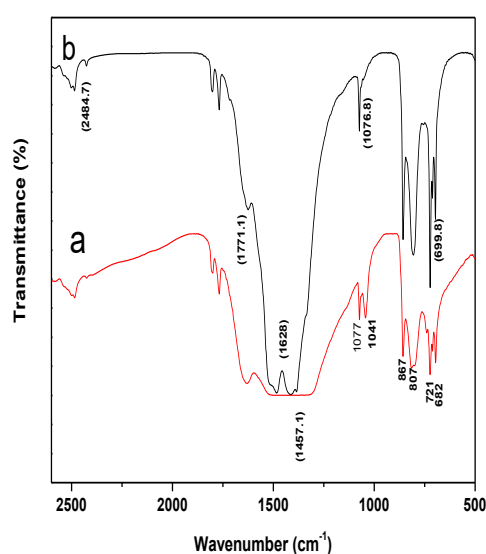


Fig.3 FTIR spectrum of the La_2O_3 a) as-prepared b) calcinated

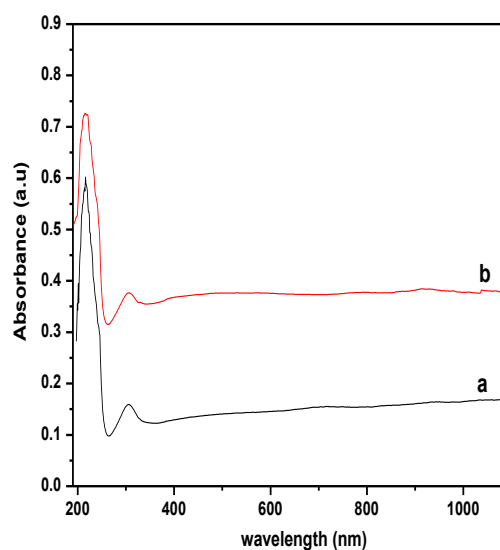


Fig.4 UV-Vis spectra of La_2O_3 a) as-prepared b) calcinated

4. Optical Analysis

The optical absorbance spectrum of La_2O_3 nanoparticles for the wavelength length range (200-1100 nm) was recorded using UV-visible spectrophotometer. Fig.3 (a and b) shows the UV absorption spectrum of as-prepared and calcinated La_2O_3 nanoparticles prepared by reflux method. It can be seen that there is a slight increase in the absorption and slight decrease in the transmittance when the sample is subjected to calcinations. This can be correlated with the reduced particle size in the SEM images, which should have increased the surface to volume ratio thus increasing the absorbing capability of the samples after calcinations. The approximate band gap of the as-prepared and calcinated samples as calculated from the absorption spectrum are 5.7 and 5.8 nm respectively.

IV. CONCLUSIONS

La_2O_3 nanoplates with the monoclinic structure were successfully synthesized by simple reflux method. This method brings forward a board idea to synthesize other rare-earth compounds with various morphologies and novel properties. Morphological studies revealed the thickness of the as-prepared La_2O_3 nanoplates particle size 50nm and on calcinating the plate like structure break into smaller La_2O_3 nanoparticles. The optical band gap being greater than 5eV for both the samples such as that the prepare La_2O_3 structure may be used for MOSFET application.

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