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Controlled Synthesis and Characterization of Lanthanum Nanorods

Jayadeep Tejani[1], Rahul Shah[2], Hiral Vaghela[3], Shailesh Vajapara[4] and Amanullakhan Pathan[4],*

[1]National Rubber Corporation, Canonsburg, PA 15317, USA
[2]Dr. Indu Dayal Meshri College of Science and Technology, Patan – 384265, Gujarat, India
[3]Government Science College, Gandhinagar-382016, Gujarat, India
[4]Department of Chemistry, Hemchandracharya North Gujarat University, Patan- 384265, India

Abstract: The present paper addresses the synthesis and characterization of La2O3Nano rods using Precipitation Method. We use Acetamide as fuel, CTAB as Surfactant and DMF as solvent in precipitation method. FTIR spectroscopy was done for observing the presence of La-O bond. The synthesized lanthanum oxide nanoparticles were characterized by X-ray diffraction (XRD), Raman Spectra, field emission gun Scanning Electron Microscopy (FEG-SEM) with EDS spectra and High resolution Transmission Electron Microscopy (HR-TEM) for morphological percentage of metal and particle size determination. In XRD analysis, the average nanorods sizes were near 38 nm. The lanthanum nano-rods prepared by Precipitation Method are thick and short with diameter of~30 nm and length of ~200 nm.

Keywords: La2O3nanorods, XRD, Raman Spectroscopy, FEG-SEM, EDS, HR-TEM, FTIR.

1 Introduction

Nanomaterials have distinctive chemical and physical properties compared to their corresponding bulk or isolated atoms and even molecules. Lanthanum oxide (La_2O_3) is a rare earth metal oxide, which shows a band gap of 4.3 eV and the lowest lattice energy with high dielectric constant, ε = 27 pF/m [1].La_2O_3 has been used as a p-type semiconductor. It also has several applications in areas of optics, magnetic, electronics, cells, fuel, ceramics, catalysis, biosensor, water treatment and biomedicine [2-4]. Lanthanum oxide has different applications, such as synthesis of ferroelectric and optical materials [5]. Lanthanum oxide is used to make optical glasses and increase their density, refractive index, and hardness. In combination with oxides of tungsten, tantalum, and thorium, La_2O_3 improves glass resistance against alkali compounds and is known as one of the ingredients for production of piezoelectric and thermoelectric

Materials [6,7].Owing to its excellent physical and chemical properties, $La(OH)_3$ has been extensively used as high-potential oxide ceramic, hydrogen, and materials, superconductive material [8]. Currently, various techniques available to synthesize metal oxide nanoparticles, such as Pechini method [9], hydrothermal synthesis[10], microwave hydrothermal synthesis [11,12] solution combustion method [13], sol-gel method [14], reverse micelle method[15], displacement method [16,17] and solution combustion method using different fuel as well as chelating agent like Propylene glycol and Glutaric acid [18].In this paper we have demonstrated synthesis of La_2O_3Nanoparticles using Precipitation method. Moreover, we have successfully synthesized La_2O_3 Nanoparticles and characterized size, structural morphology, elemental analysis, presence of metal-oxide bond and crystallinity using X-ray diffraction (XRD), scanning electron microscopy (FEG-SEM), energy dispersive X-ray spectroscopy (EDS), FT-IR spectroscopy and Transmission electron microscopy (HR-TEM) techniques.

*Corresponding author E-mail: amankhan255@gmail.com

2 Experimental Details

2.1 Materials

Analytical-grade Lanthanum chloride (Lacl₃), Acetamide, Cetyl trimethyl ammonium bromide (CTAB) and DMF reagents were used as received from the *s.d fine* chemicals (India). All reaction was performed using double distilled water.

2.2   Synthesis of La₂O₃ Nanoparticles Using Precipitation Method

In this method, all chemicals were used without further purification. In reaction assembly, 2.0 g Acetamide and 1.6 gm CTAB were dissolved in 40 ml of DMF and starring at 80 °C for 30 min. After that, 5 gm of lanthanum chloride was dissolved in 30 ml DMF. This solution was added to known volume of the Acetamide and CTAB solution. The resultant solution was stirred for 4 hrs at 80-90 °C. Then, the solution was filtered and washed four times with double distilled water and the ppts for Calcination was transferred in muffle furnace for 4 hrs at 600 °C.

3 Results and Discussion

3.1 XRD Spectra of La₂O₃ Nanoparticles

Figure-2 shows how to prepare the XRD spectra of the La₂O₃Nanorods using Precipitation method. This result indicates that the structure of the La₂O₃ Nanorods is in pure hexagonal phase when synthesized using Precipitation method. The extended peaks are represent the dimensions of the Nano range particles. Peaks are observed at 16.55º, 23.55⁰, 28.9⁰, 33.44⁰, 44.79⁰, 48⁰and 56.99⁰ corresponding to the (h k l) values of the peaks (1 0 0), (1 0 1), (2 2 0), (2 0 1), (2 1 0) and (0 02), respectively. The lattice parameters were in consistent with (JCPDS card number 73-2141) [19].
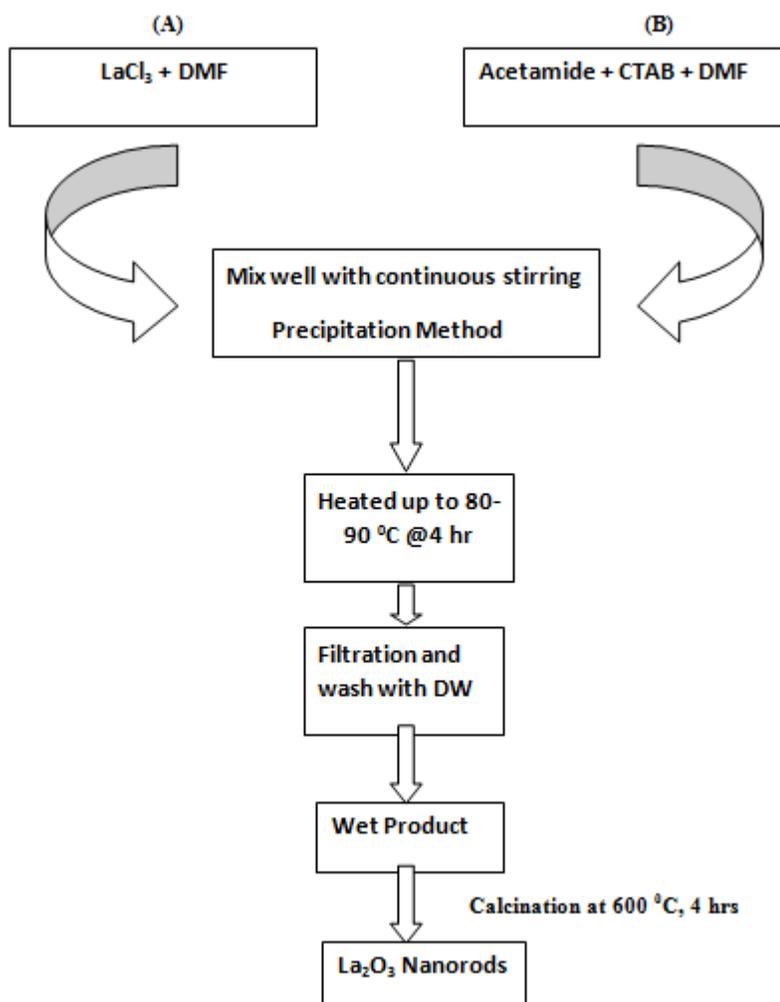

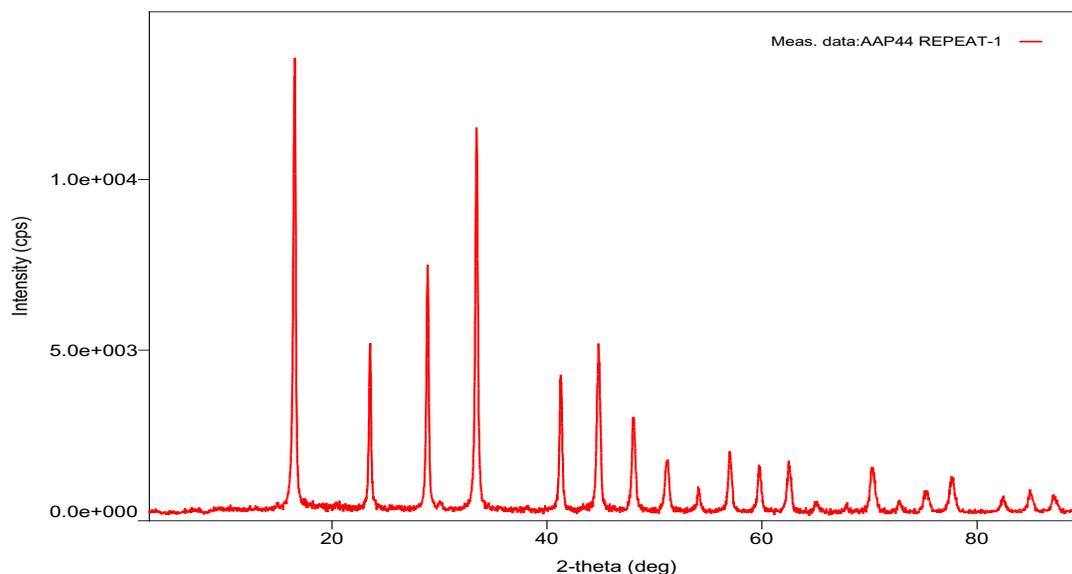**Fig. 1:** Schematic diagram of Precipitation method for La₂O₃ Nanorods.

Fig: 2: XRD Spectra of La_2O_3 Nano rods Synthesized by Precipitation Method.

The average particles size is calculated by Debye-Scherer's formula,

$$D = \frac{K\lambda}{\beta \, Cos\theta} \qquad (1)$$

Where D is the average crystallite size of the particle, λ is the wavelength of the radiation, β is the full width at half maximum (FWHM) of the peak, and θ is the Bragg's angle. The average crystallite size of samples synthesized by this method is 30 nm.

The average crystallite sizes were 30 nm and strain was 0.0028 for Nanorods synthesized by this method.

The lattice parameters of the hexagonal phase were measured using the below formula.

3.2 Fourier Transform Infrared Spectroscopy of La_2O_3 Nanoparticles

FTIR analysis has been conducted in the wave number ranging from 500 cm^{-1} to 4000 cm^{-1}. The samples have been admixed with KBr, and thoroughly mixed and pelletized by pressing under sufficient pressure, before FTIR analysis. La_2O_3 Nano particles were analysed with the BRUCKER (αT Model) FTIR spectrometer as shown in fig. 3.The very weak absorption bands at 2908 cm^{-1} are assigned to O-H stretching vibration of water molecules because of the presence of moisture in the sample. Very weak bending vibrations of water molecules appeared at 1575 cm^{-1}, C-C Stretching. Medium strong band positions

in the range of 1357 cm^{-1} to 1427 cm^{-1} are possible due to stretching vibrations of ions. The narrow absorption peak observed around at 1085 cm^{-1} can be ascribed to the C=O bonding. The medium to strong absorption bands at 777 cm^{-1} occurred because of La-O stretching [20].

Hence, the existence of the above-mentioned bands identify the presence of La_2O_3.

3.3 Raman Spectroscopy

Lanthanum Oxide (La_2O_3) band at 313.605 cm^{-1} was observed. These bands have been identified as Raman bands. The observed band in the present work has not been previously reported. The Raman shifts reported by White and Boldish for their La_2O_3 sample, 410 cm^{-1} and 195 cm^{-1}, were not observed in the present study [21]. Surface carbonation also contributes to the differences in band position reported by different authors. The bands at 1121.44 cm^{-1} support carbonate compound present at the surface [22].

3.4 Field Emission Gun Scanning Electron Microscopy (FEG-SEM) and EDS

The grain size, shape and surface properties like morphology were observed using FEG-SEM with different magnifications. The FEG-SEM images of La_2O_3 nanorods, which were prepared using this Method, are shown in fig. 5, respectively.
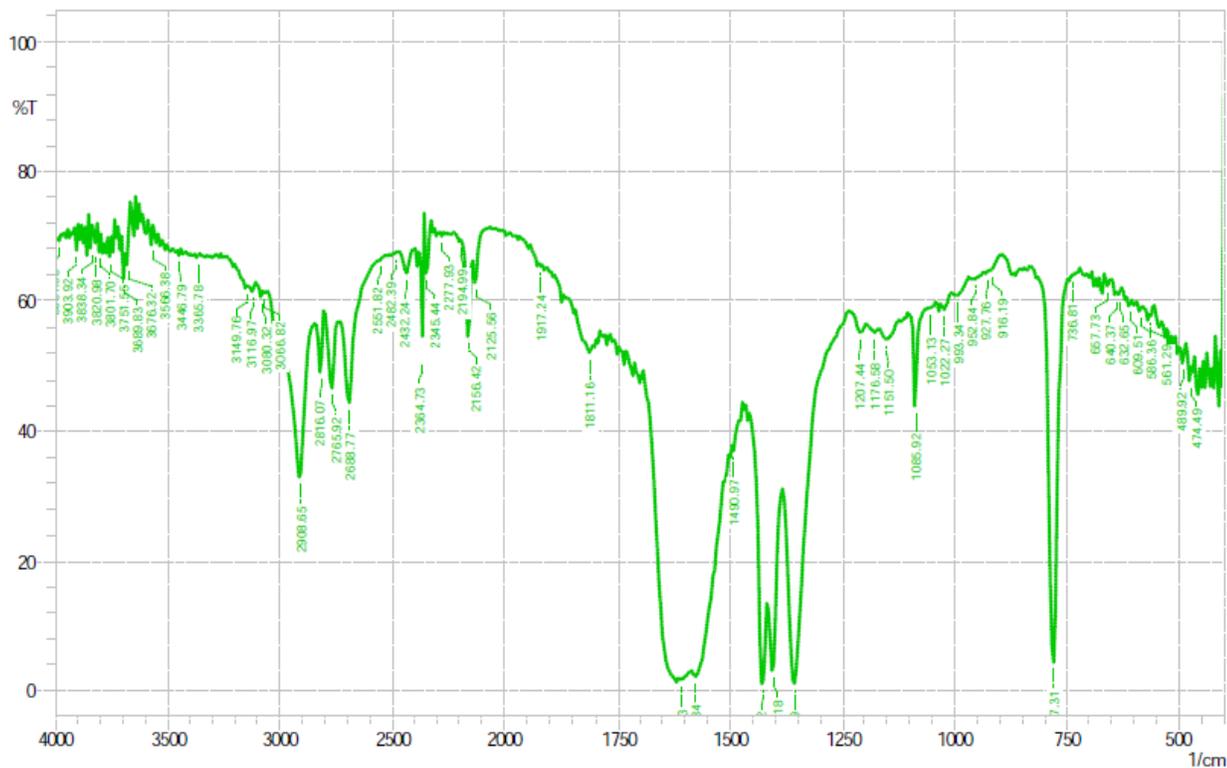
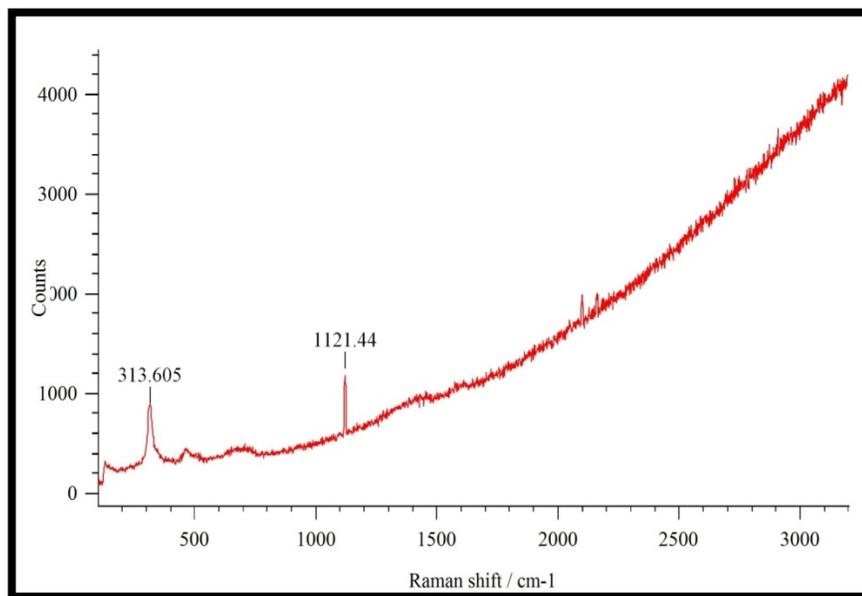**Fig. 3:** IR Spectra of La$_2$O$_3$ Nanoparticles.

Fig. 4: Raman spectrum of La 2 O 3 Nanoparticles.

EDS spectrum of La$_2$O$_3$ shows the peaks for lanthanum and oxygen elements indicating the formation of La$_2$O$_3$ nanoparticles. Peak indexing of the elements is oxygen 0.52 keV and lanthanum 4.62 keV.

The compositions in mass percentage of the elements are oxygen 35.15% and lanthanum 58.42 %. The observed composition matches the theoretically calculated composition.
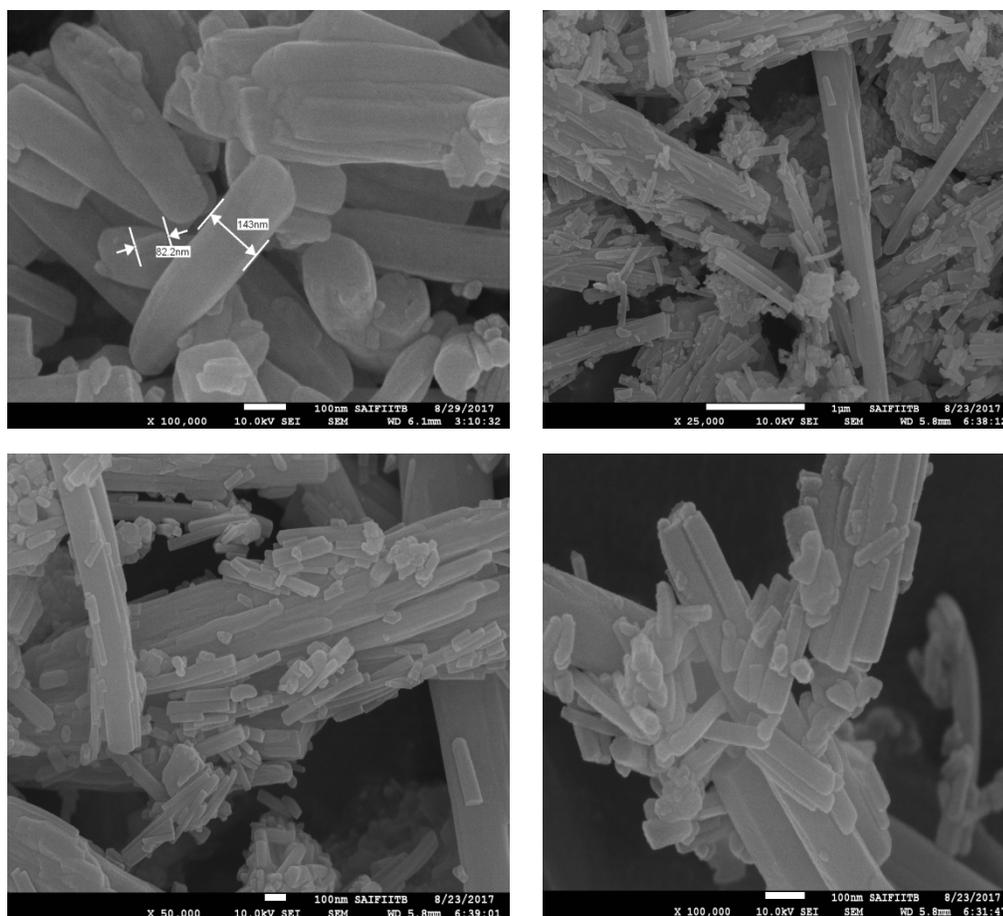
Fig. 5: FEG-SEM Images of La_2O_3 Nanorods Synthesized by Precipitation Method. It shows that the nanorods are agglomerated and porous properties. It shows that the size of the porous or porosity increases as the fuel ratio increases.
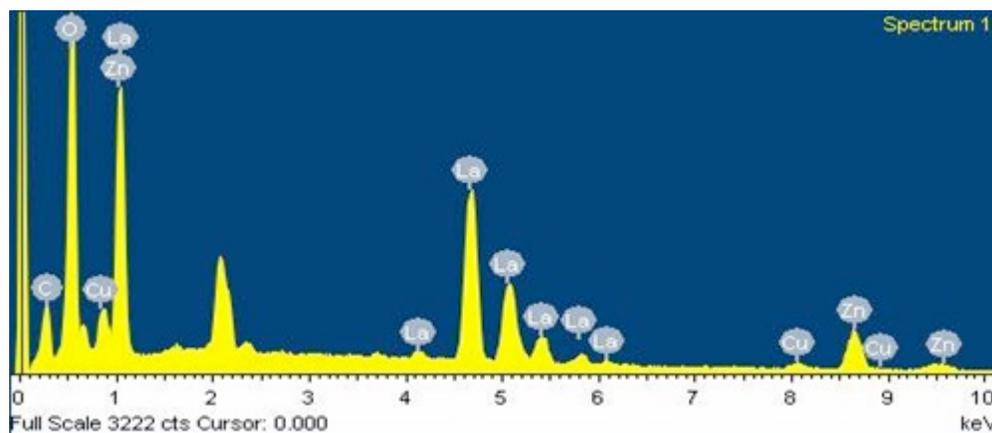

Fig. 6: EDS spectrum of La_2O_3 Nanoparticles.

3.5 High Resolution Transmission Electron Microscopy (HR-TEM) Analysis:

The HR-TEM analysis ndicates the agglomerated sample in Nano range. The below figure shows the HR-TEM micrograph of the sample synthesized using Precipitation Method.From HR-TEM analysis, it has been found that the nanorods samples are not good in crystal due to severe agglomeration. However, the nanorods are well below Nanometer range to conclude that the obtained materials are Nanorods.
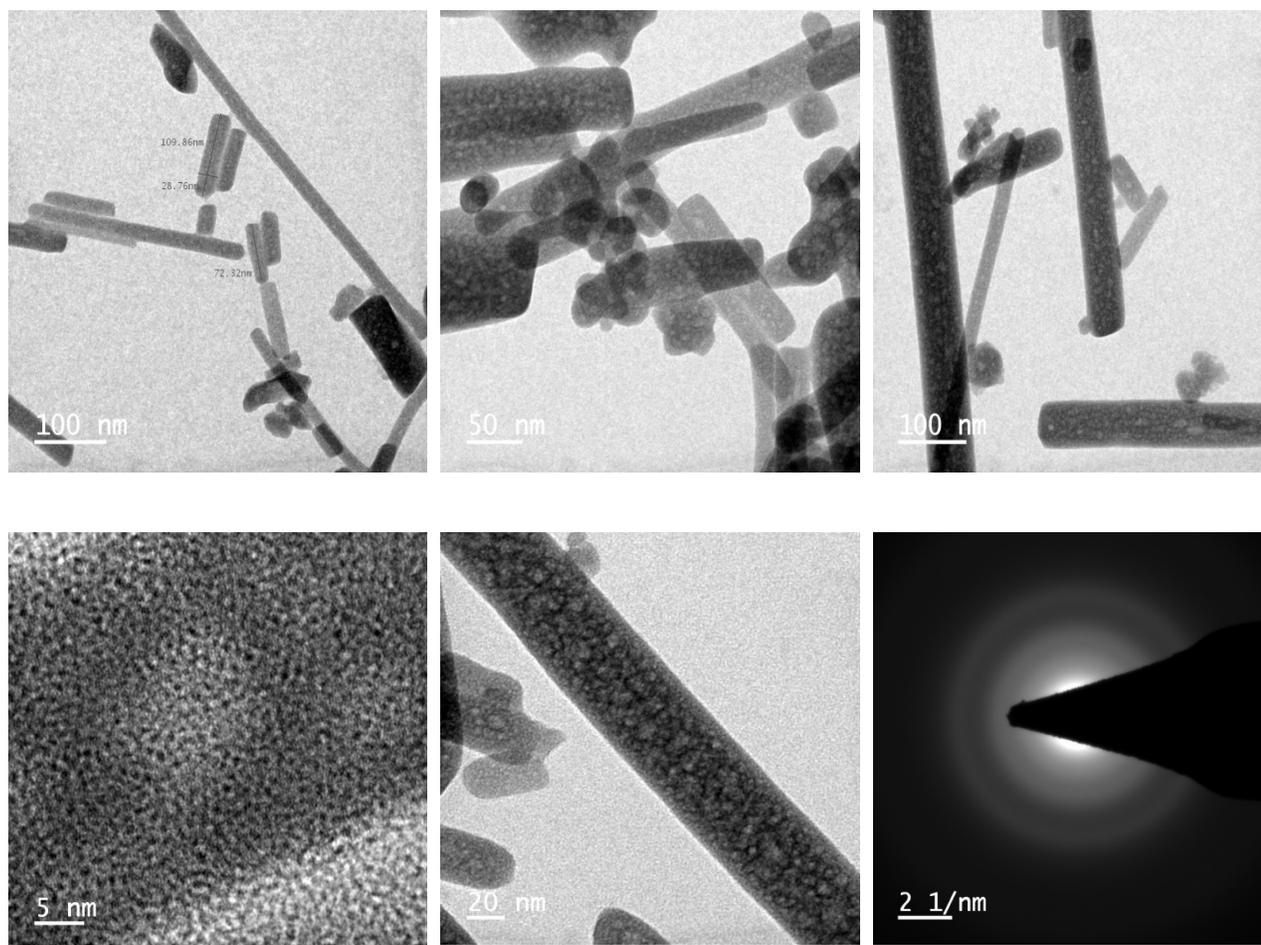
Fig. 7: (a) and 6 (b) HR-TEM Images of La$_2$O$_3$ Nanorods Synthesized by Precipitation Method.

4 Conclusions

La2O3 Nanorods have been successfully synthesized by Precipitation method using Acetamide as fuel and CTAB as surfactant. The average Nanorods sizes of samples synthesized using this method are 30 nm and length near to 200 nm. FTIR analysis shows La-O band at 777 cm-1. Structural properties examined by FEG-SEM reveal that porous and porosity were good in network of Nanorods La2O3. The EDS shows that the compositions in mass percentage of the elements are oxygen (35.15%) and lanthanum (64.42 %.) From the above HR-TEM characterizations, we inferred that nanorods are well below Nanometer range and the obtained materials are Nanorods.

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