International Journal of Thin Film Science and Technology

Volume 9
Issue 2 *May 2020*

Article 5

2020

# Controlled Synthesis and Characterization of Lanthanum Nanorods

Jayadeep Tejani
*National Rubber Corporation, Canonsburg, PA 15317, USA*, amankhan255@gmail.com

Rahul Shah
*Dr. Indu Dayal Meshri College of Science and Technology, Patan – 384265, Gujarat, India*, amankhan255@gmail.com

Hiral Vaghela
*Government Science College, Gandhinagar-382016, Gujarat, India*, amankhan255@gmail.com

Shailesh Vajapara
*Department of Chemistry, Hemchandracharya North Gujarat University, Patan- 384265, India*, amankhan255@gmail.com

Amanullakhan Pathan
*Department of Chemistry, Hemchandracharya North Gujarat University, Patan- 384265, India*, amankhan255@gmail.com

Follow this and additional works at: https://digitalcommons.aaru.edu.jo/ijtfst

## Recommended Citation

Tejani, Jayadeep; Shah, Rahul; Vaghela, Hiral; Vajapara, Shailesh; and Pathan, Amanullakhan (2020) "Controlled Synthesis and Characterization of Lanthanum Nanorods," *International Journal of Thin Film Science and Technology*: Vol. 9 : Iss. 2 , Article 5.
Available at: https://digitalcommons.aaru.edu.jo/ijtfst/vol9/iss2/5

This Article is brought to you for free and open access by Arab Journals Platform. It has been accepted for inclusion in International Journal of Thin Film Science and Technology by an authorized editor. The journal is hosted on Digital Commons, an Elsevier platform. For more information, please contact rakan@aaru.edu.jo, marah@aaru.edu.jo, u.murad@aaru.edu.jo.

International Journal of Thin Films Science and Technology

http://dx.doi.org/10.18576/ijtfst/090205

# 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

Received: 2 Jan. 2020, Revised: 10 Feb. 2020, Accepted: 19 Feb. 2020

Published online: 1 May 2020

**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:** $La_2O_3$nanorods, 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 $La_2O_3$Nanoparticles 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

© 2020 NSP
Natural Sciences Publishing Cor.

## 2 Experimental Details

### *2.1 Materials*

Analytical-grade Lanthanum chloride ($LaCl_3$), 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 $La_2O_3$ 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 $^0$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 $^0$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 $^0$C.

## 3 Results and Discussion

### *3.1 XRD Spectra of $La_2O_3$ Nanoparticles*

Figure-2 shows how to prepare the XRD spectra of the $La_2O_3$Nanorods using Precipitation method. This result indicates that the structure of the $La_2O_3$ 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 16.55$^o$, 23.55$^0$, 28.9$^0$, 33.44$^0$, 44.79$^0$, 48$^0$and 56.99$^0$ 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].

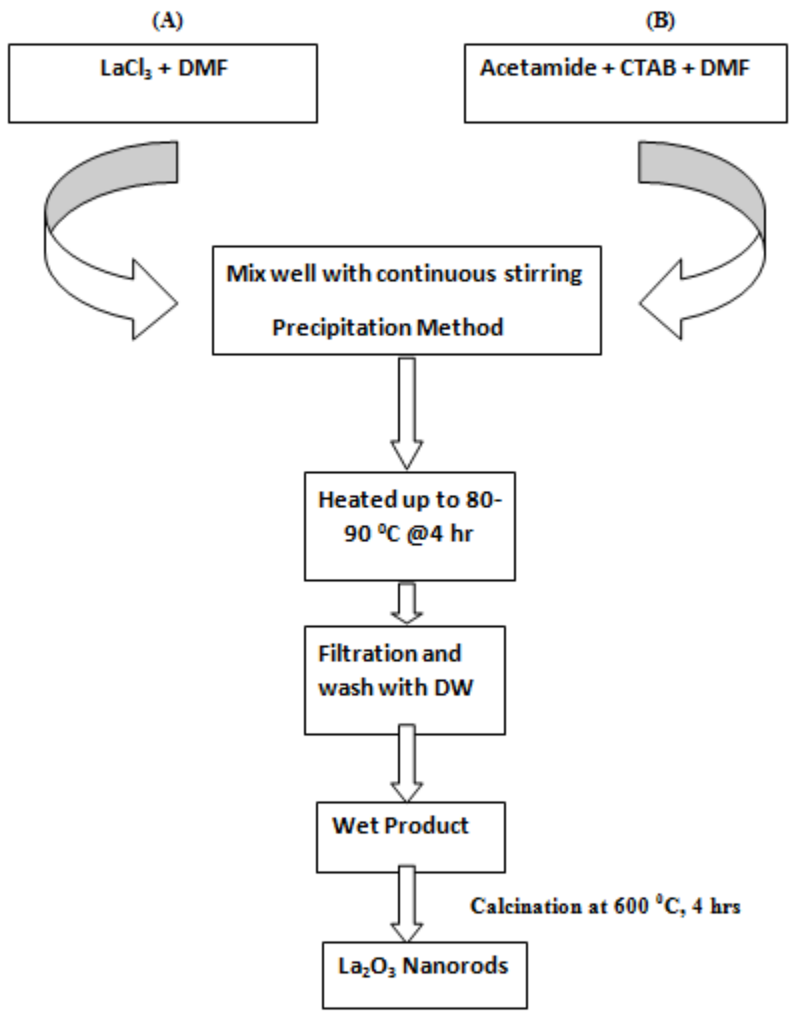


**Fig. 1:** Schematic diagram of Precipitation method for $La_2O_3$ Nanorods.

© 2020 NSP
Natural Sciences Publishing Cor.

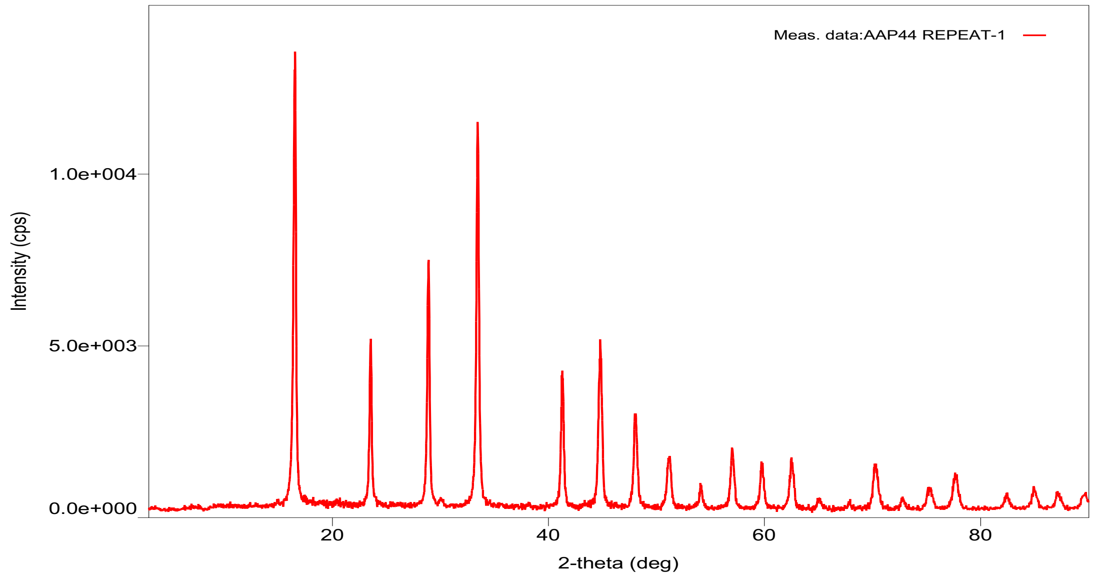


**Fig: 2:** XRD Spectra of $La_2O_3$ Nano rods Synthesized by Precipitation Method.

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

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

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.

© 2020 NSP
Natural Sciences Publishing Cor.

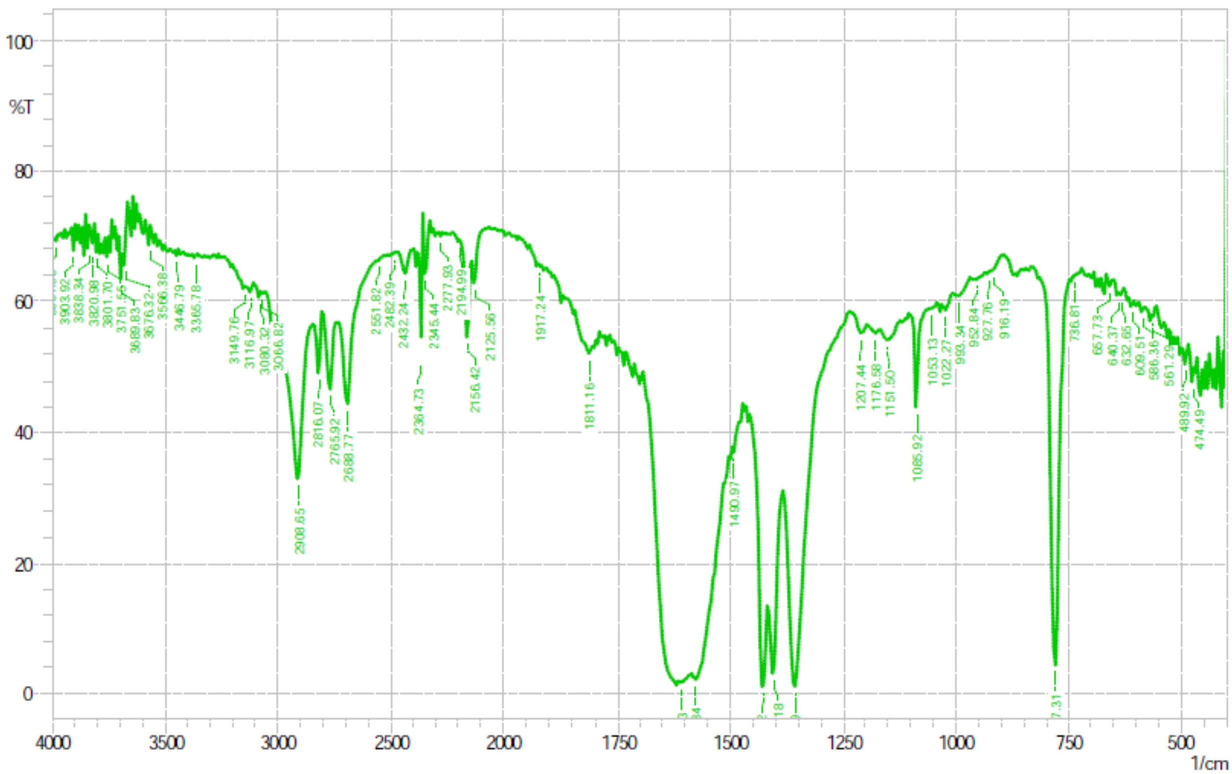


**Fig. 3:** IR Spectra of $La_2O_3$ Nanoparticles.

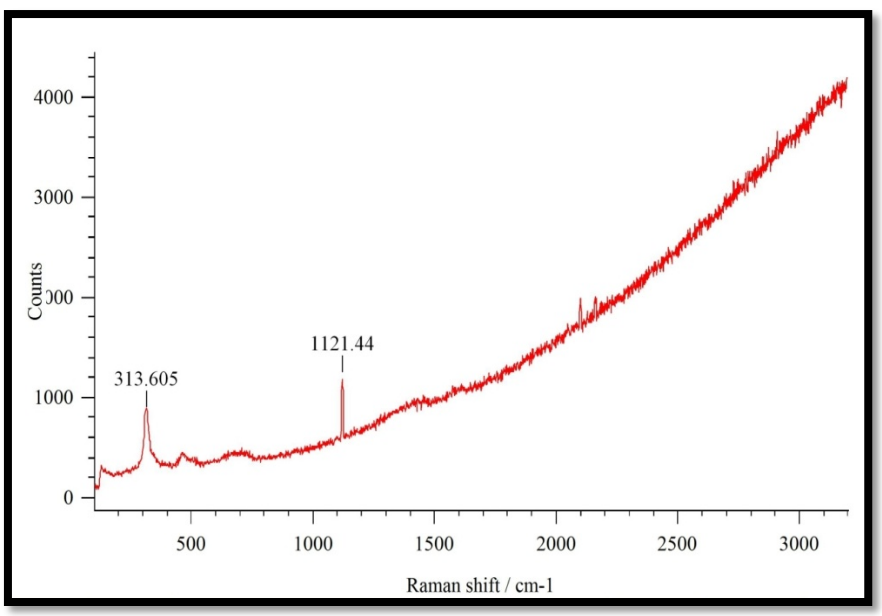


**Fig. 4:** Raman spectrum of La 2 O 3 Nanoparticles.

EDS spectrum of $La_2O_3$ shows the peaks for lanthanum and oxygen elements indicating the formation of $La_2O_3$ 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.

© 2020 NSP
Natural Sciences Publishing Cor.

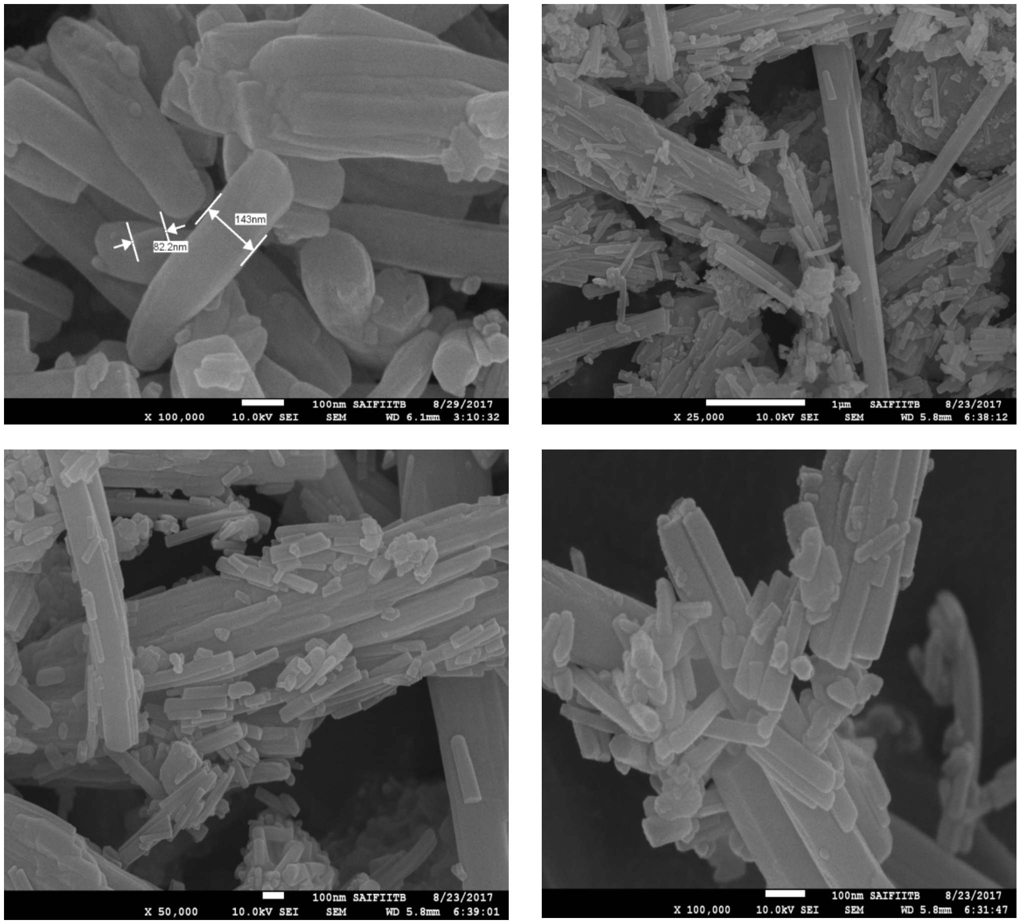


**Fig. 5:** FEG-SEM Images of $La_2O_3$ Nanorods Synthesized by Precipitation Method. It shows that the nanorods are agglomerated and porous properties. It shows that the size of the porous or porosity increases as the fuel ratio increases.

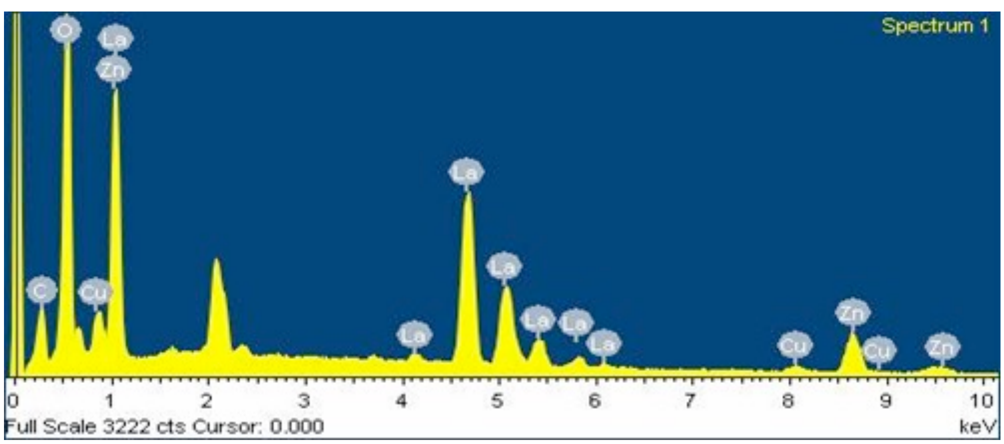


**Fig. 6:** EDS spectrum of $La_2O_3$ Nanoparticles.

### *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.

© 2020 NSP
Natural Sciences Publishing Cor.

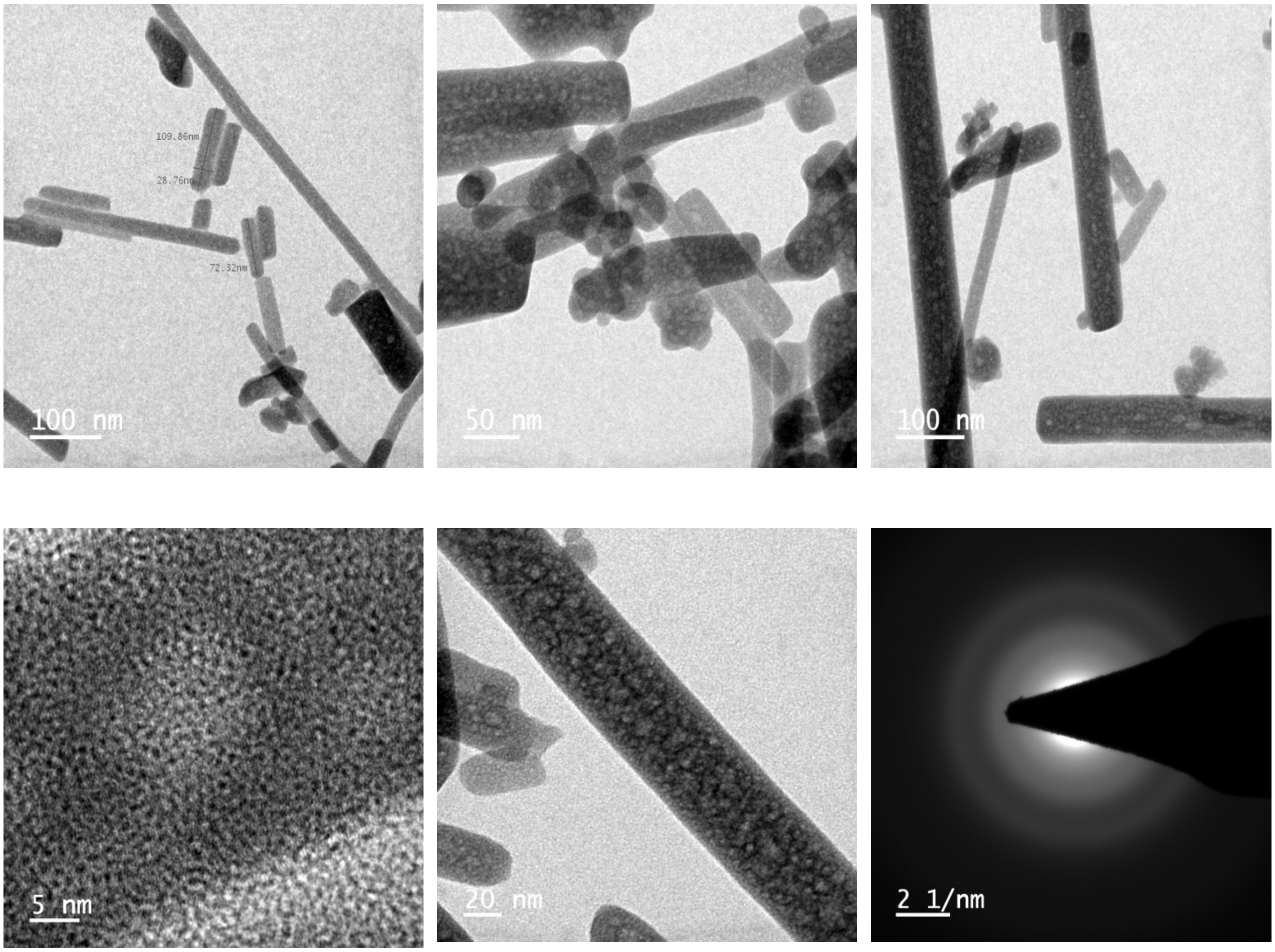


**Fig. 7:** (a) and 6 (b) HR-TEM Images of $La_2O_3$ Nanorods Synthesized by Precipitation Method.

## 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.

## References

[1] Gao Y, Masuda Y, Koumoto K. Micropatterning of lanthanum- based oxide thin film on self-assembled mono layers. J. Colloid Interface Sci., **274**, 392(2004).

[2] Nejad S J, Abolghasemi H, Moosavian M A, Golzary A, Maragheh M G. Fractional factorial design for the optimization of hydrothermal synthesis of lanthanum oxide nanoparticles under supercritical water condition. J. Supercrit. Fluids., **52**, 292(2010).

[3] Wang X G, Wang M L, Song H, Ding B J. A simple sol-gel technique for preparing lanthanum oxide nanopowders. Materials Letters., **60**, 2261(2006).

[4] Khanjani S, Morsali A. Synthesis and characterization of lanthanum oxide nanoparticles from thermolysis of nanostructured supramolecular compound. J. Mol. Liquid., **153**, 129(2010).

[5] R. Vali, S.M. Hosseini, First-principles study of structural, dynamical, and dielectric properties of A-La2O3, Comp. Mater. Sci., **31,** 125–130(2004).

[6] A.V. Vishnyakov, I.A. Korshunova, V.E. Kochurikhin, L.S. Sal'nikova, Catalytic activity of rare earth oxides in flameless methane combustion, Kinet. Catal., **51** 273–278(2010).

[7] H.L. Wan, X.P. Zhou, W.Z. Weng, R.Q. Long, Z.S. Chao, W.D. Zhang, M.S. Chen, J. Z. Luo, S.Q. Zhou, Catalytic performance, structure, surface properties

© 2020 NSP
Natural Sciences Publishing Cor.

and active oxygen species of the fluoride-containing rare earth (alkaline earth)-based catalysts for the oxidative coupling of methane and oxidative dehydrogenation of light alkanes, Catal. Today., **51** 161–175(1999).

[8] O. Yavapao, S. Thongtem, A. Phuruangrat, T. Thongtem, A simple microwave assisted hydrothermal synthesis of lanthanum hydroxide nanowires with a high aspect ratio, J. Ceram. Process. Res., **13**, 466–469(2012).

[9] Pechini, M. P. "US Pat.. **3**, 330- 697(1967)."

[10] Yi, Xie, Qian Yitaia, Li Jinga, Chen Zuyaoa, and Yang Li. "Hydrothermal preparation and characterization of ultrafine powders of ferrite spinels MFe2O4 (M= Fe, Zn and Ni)." Materials Science and Engineering B., **34(1)**, L1-L3(1995).

[11] Wang, Hong-Wen, and Shong-Chung Kung. "Crystallization of nanosized Ni–Zn ferrite powders prepared by hydrothermal method." Journal of Magnetism and Magnetic Materials., **270( 1-2)**, , 230-236(2004).

[12] Krishnaveni, T., S. R. Murthy, F. Gao, Q. Lu, and S. Komarneni. "Microwave hydrothermal synthesis of nanosize Ta2O5 added Mg-Cu-Zn ferrites." Journal of materials science., **41(5)**, 1471-1474(2006).

[13] Tsoutsou, D., G. Scarel, A. Debernardi, S. C. Capelli, S. N. Volkos, L. Lamagna, S. Schamm, P. E. Coulon, and M. Fanciulli. "Infrared spectroscopy and X-ray diffraction studies on the crystallographic evolution of La2O3 films upon annealing." Microelectronic Engineering., **85(12)**, 2411-2413(2008).

[14] Kim, Woo Chul, Sam Jin Kim, Seung Wha Lee, and Chul Sung Kim. "Growth of ultrafine NiZnCu ferrite and magnetic properties by a sol–gel method." Journal of magnetism and magnetic materials., **226**, 1418-1420(2001).

[15]http://www.intel.com/technology/silicon/45nmtechnology.htm

[16] Pathan, Amanullakhan A., Kavita R. Desai, and Chandra P. Bhasin. "Improved Photocatalytic Properties of NiS Nanocomposites Prepared by Displacement Method for Removal of Rose Bengal Dye." Current Nanomaterials., **2(3)**,169-176(2017).

[17] Tejani J, Shah R, Vaghela H, Kukadiya T, Pathan AA. Conditional optimization of displacement synthesis for pioneered ZnS nanostructures. J Nanotech Adv Mater., **6(1)**, 1-7(2018).

[18] Pathan, Amanullakhan A., Kavita R. Desai, and C. P. Bhasin. "Synthesis of La2O3 Nanoparticles Using Glutaric Acid and Propylene Glycol for Future CMOS Applications." International journal of Nanomaterials and Chemistry., **3**, 21-25(2017).

[19] Phases, Powder Diffraction File Inorganics. "Alphabetical index, inorganic phases. Swarthmore, Pennsylvania: JCPDS." International Centre for Diffraction Data (1988).

[20] Pathan, Amanullakhan A., Kavita R. Desai, Shailesh Vajapara, and C. P. Bhasin. "Conditional Optimization of Solution Combustion Synthesis for Pioneered La2O3 Nanostructures to Application as Future CMOS and NVMS Generations." Advances in Nanoparticles., **7( 01)**, 28(2018).

[21] Boldish, Steven I., and William B. White. "Vibrational spectra of crystals with the A-type rare earth oxide structure-I. La2O3 and Nd2O3." Spectro chimica Acta Part A: Molecular Spectroscopy., **35(11)**, 1235-1242(1979).

[22] Cui, Jianlan, and Gregory A. Hope. "Raman and Fluorescence Spectroscopy of CeO2, Er2O3, Nd2O3, Tm2O3, Yb2O3, La2O3, and $Tb_4O_7$." Journal of Spectroscopy., (2015).

© 2020 NSP
Natural Sciences Publishing Cor.