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Applied Physics A

Influence of Calcination on Sol-Gel Synthesis of Lanthanum Oxide Nanoparticles

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Abstract:	<p>A facile sol-gel technique was employed to synthesize Lanthanum Oxide nanoparticles (La₂O₃ NPs) effectively using micro-sized La₂O₃ powder, 20% nitric acid and high molecular weight polyethylene glycol (PEG) as starting materials and later their characteristics were studied via several characterization techniques, namely X-Ray diffraction (XRD), environmental scanning electron microscopy (ESEM), energy-dispersive X-ray spectroscopy, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), Fourier Transform infrared (FTIR) spectroscopy and photoluminescence (PL) spectroscopy. To investigate the influence of calcination temperatures on the particle size and other properties, the synthesized La₂O₃ nanopowder was calcined at several temperatures including 600 °C, 750 °C and 900 °C. The obtained results indicated that calcination temperatures affected significantly the crystallinity, particle size and lattice strain. The XRD patterns confirmed the hexagonal phase of the La₂O₃ NPs with lattice constant values, $a = b = 0.3973$ nm and $c = 0.6129$ nm. The average particle size of the La₂O₃ NPs estimated from ESEM images were in good agreement with the average crystal size obtained from the XRD data. The crystallinity as well as the mean crystallite size of the La₂O₃ NPs increased, while the lattice strain decreased with increasing calcinations temperatures. The Photoluminescence spectra of La₂O₃ NPs illustrated a strong emission band located at the wavelength of 364 nm which is typical green band and the wavelength of this band was not affected by the temperatures of the calcination.</p>

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Influence of Calcination on Sol-Gel Synthesis of Lanthanum Oxide Nanoparticles

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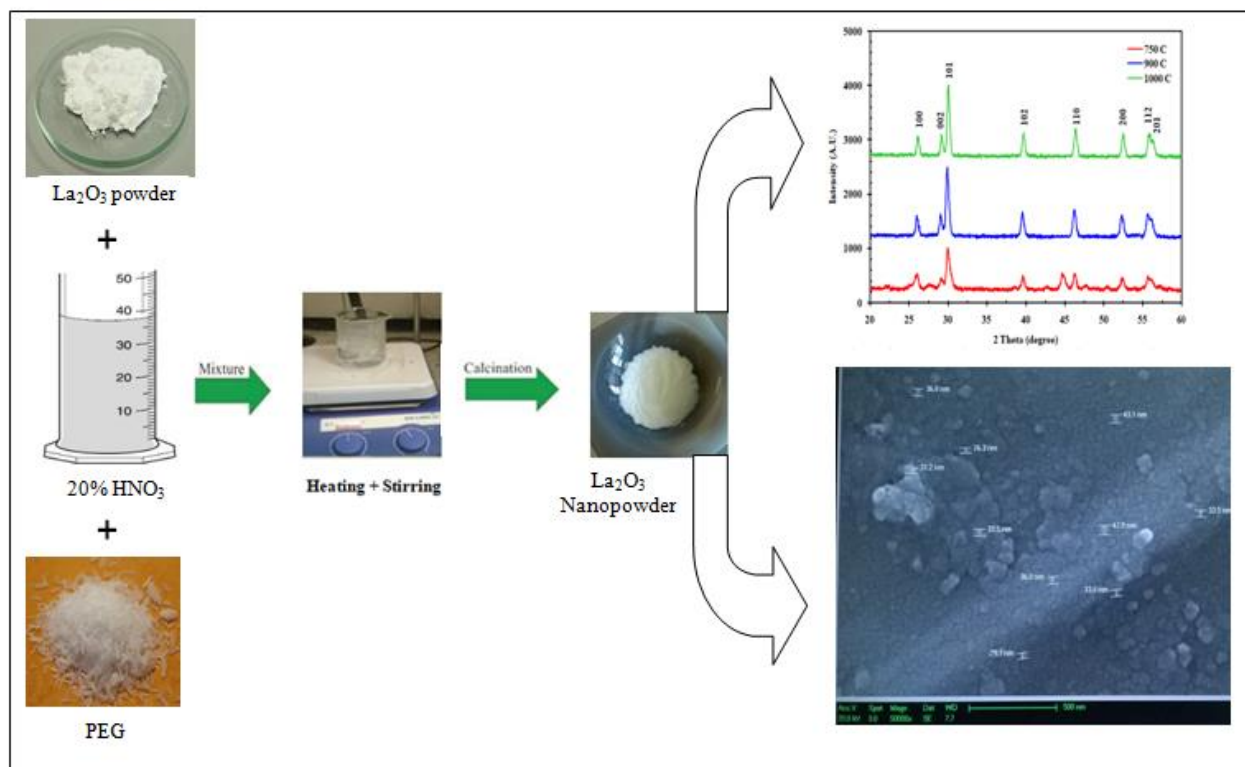
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Abstract:

A facile sol-gel technique was employed to synthesize Lanthanum Oxide nanoparticles (La_2O_3 NPs) effectively using micro-sized La_2O_3 powder, 20% nitric acid and high molecular weight polyethylene glycol (PEG) as starting materials and later their characteristics were studied via several characterization techniques, namely X-Ray diffraction (XRD), environmental scanning electron microscopy (ESEM), energy-dispersive X-ray spectroscopy, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC), Fourier Transform infrared (FTIR) spectroscopy and photoluminescence (PL) spectroscopy. To investigate the influence of calcination temperatures on the particle size and other properties, the synthesized La_2O_3 nanopowder was calcined at several temperatures including 600 °C, 750 °C and 900 °C. The obtained results indicated that calcination temperatures affected significantly the crystallinity, particle size and lattice strain. The XRD patterns confirmed the hexagonal phase of the La_2O_3 NPs with lattice constant values, $a = b = 0.3973$ nm and $c = 0.6129$ nm. The average particle size of the La_2O_3 NPs estimated from ESEM images were in good agreement with the average crystal size obtained from the XRD data. The crystallinity as well as the mean crystallite size of the La_2O_3 NPs increased, while the lattice strain decreased with increasing calcinations temperatures. The Photoluminescence spectra of La_2O_3 NPs illustrated a strong emission band located at the wavelength of 364 nm which is typical green band and the wavelength of this band was not affected by the temperatures of the calcination.

Graphical Abstract



Key Words

Sol-gel technique, X-Ray diffraction, crystallinity, hexagonal phase, crystal size.

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Highlights

- La_2O_3 NPs were synthesized successfully via a facile sol-gel technique.
- The calcination temperatures affected significantly the crystallinity, particle size and lattice strain.
- The crystallinity and the mean crystallite size of the La_2O_3 NPs increased, while the lattice strain decreased with increasing calcinations temperatures.

- The thermograms of all samples showed three main thermal transitions associated with weight loss.
- In PL spectra a strong emission band located at the wavelength of 364 nm is typical green band.

1. Introduction

Rare earth elements are distinguished by their high density, high melting point, high thermal conductance and conductivity. Because of their 4f orbital electron, they possess unique physical as well as chemical properties and have wide applications in electronics, medical, biomedical and agronomical fields [1, 2]. In plenty of areas of physics, chemistry, and materials science, metal oxides keep exceptionally significant contributions [3]. They can indicate metallic, semiconductor, or insulator character; because they can take an extensive number of structural geometries with a specific electronic structure. They have vast applications in technology, including fabrication of microelectronic circuits, sensors, piezoelectric devices, fuel cells, coatings against corrosion, and as catalysts. Metal oxides (MOs) are used as catalysts to prepare different products in chemical and petrochemical industries [4, 5]. They also used as catalysts or sorbents to carry away CO, NO_x, and SO_x species formed during the combustion of fossil fuels [5]. The greater portion of semiconductor industry uses of MOs to generate electronic components, like chips used in computers [6]. If the metal oxide's size is taken down to nanometer range, a number of properties depending on size initially develop because of surface chemistry [7-9]. As metal oxide nanoparticles (NPs) have confined size, so they can express unique chemical as well as physical properties. Particle size has impact on three momentous fundamental properties of a material. Firstly, the structural characteristics, exemplary lattice symmetry and unit cell parameters [10]. Normally, bulk MOs is stable with specific crystallographic structures. When the particle size decreases, the stability of thermodynamic as well as unit cell parameters alters and hence structural transformations may happen [11]. Secondly, particle size of MOs is connected to the electronic properties. If the size of particle reduces, the energy of exciton levels of a semiconductor MOs displaces and therefore the optical band gap of the MOs varies [12]. When the structural and electronic properties of MOs vary, they evidently switch the physical as well as chemical properties of a MOs. This is the third group of characteristics that is dominated by the size of the particles [13].

Lanthanum (III) oxide, La₂O₃, called lanthania is an odourless, white solid and is a rare earth metal oxide. Lanthania is not soluble in water, but soluble in dilute acid. La₂O₃ has the greatest

1 optical band gap of the rare earth oxides, 4.3 eV, the minimum lattice energy and an extremely
2 high dielectric constant, 27 [14]. Because of these interesting properties, lanthania has a lot of
3 potential applications in different cases, for instance fuel cells, dielectric layers in devices, optics,
4 magnetic data storage and magnetic resonance imaging (MPI), ceramics, gas sensors, catalysis,
5 automobiles, biosensors, automobiles, water treatment, biomedicine, protective and optical
6 coating, preparing several perovskite nanostructures, photoelectric conversion, optical filters, light
7 converting agricultural films, in light emitting materials (blue powder), for preparing high
8 refraction optical fibres, precision optical glasses and other alloys materials, etc. [15-19]. A several
9 methods have developed for the synthesizing of La₂O₃ ultrafine powders, nanopowders, nanorods,
10 nanowires, nanosheets, nanoneedles, nanobundles and nanoparticles, incorporate with sol-gel [20,
11 21], hydrothermal [22, 23], sonochemical [24, 25], microwave [26], solvo-thermal [27], laser
12 deposition [28], thermal decomposition [29], chemical precipitation [30], reverses micelles [31],
13 arc-discharge [32], green carbonation [33], starch template [34] and other chemical and physical
14 techniques. All these methods have their own advantages and disadvantages. Amongst these
15 methods, the sol-gel synthesis process is a facile, inexpensive and does not require any specialist
16 equipment. Goharshadi *et al.* synthesized lanthania nanoparticles [35] using LaCl₃, 7H₂O as the
17 starting materials, ammonia solution as hydrolysis agents and hexadecyltrimethyl ammonium
18 bromide as a surfactant using hydrothermal process. The obtained average particle size was below
19 30 nm. La₂O₃ NPs were also prepared by Nejad *et al.* using same process starting from lanthanum
20 nitrate, La(NO₃). 6H₂O and the obtained mean particle size was around 50 nm [22]. A simple sol-
21 gel method has been employed by Wang *et al.* [36] to synthesis La₂O₃ nanoparticles by mixing
22 commercial powder La₂O₃, polyethylene glycol (PEG) and nitric acid. They showed that particle
23 size is strongly influenced by calcination temperature as well as concentration of PEG in the
24 solution. The average particle size of La₂O₃NPs decreased with increasing PEG concentration.
25 Niasari *et al.* studied the synthesis of lanthanum carbonate, La₂(CO₃)₃ for the preparation of
26 La(OH)₃ and La₂O₃ NPs via sonochemical method [37]. They reported the variation of surfactant,
27 calcination temperature as well as sonication time on the morphology and particle size of the NPs
28 and concluded that sonication time of 30 min and calcination temperature of 600 °C are optimum
29 condition to synthesize La₂(CO₃)₃ and La₂O₃, respectively and addition of surfactant is not
30 required. Synthesis of nanostructure La₂O₃ as a heterogeneous catalyst has been reported by Zhou
31 *et al.* [38] via sonochemical technique for biodiesel synthesis.

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In this study, we reported surfactant assisted sol-gel technique to synthesis La₂O₃ NPs
using surfactant PEG as template agent and micro-sized lanthanum oxide powder as precursors.
As-synthesized products were characterized by XRD, ESEM, TGA, DSC, PL and FTIR
spectroscopy. The influence of calcination temperatures on average particle size was also

investigated. A deeper understanding the properties and the synthesis technique will be useful for developing the potential applications of La₂O₃ NPs.

2. Experimental details

2.1 Raw materials and synthesis process La₂O₃ NPs

All the chemical reagents used in this synthesis process were analytical grade, purchased from Sigma Aldrich, UK, and were used as direct without further purification.

In this synthesis process, the sol-gel technique was used to prepare La₂O₃ NPs. In a typical process, 2.00 g micro-sized ($\approx 300 \mu\text{m}$) commercially purchased La₂O₃ powder was kept in a pre-cleaned glass beaker. The required amount ($\approx 18.5 \text{ ml}$) of 20% aqueous solution of HNO₃ was added drop wise to the La₂O₃ powder with continuous and vigorous magnetic stirring until all La₂O₃ powder reacted with HNO₃ to produce an aqueous solution of lanthanum nitrate (La(NO₃)₃·6H₂O). Then various amounts of PEG were added to produce a solution with different PEG concentrations, such as 30 g/l, 40 g/l, 45 g/l, 50 g/l, 60 g/l and 70 g/l. The solution was then further stirred for 15 minutes using a magnetic stirrer. The solution was then kept in a water bath at 80 °C with continuous stirring for approximately 2.0 hours until most of the water was evaporated and a clear gel was formed. The obtained product was allowed to cool down to room temperature. The whole experimental procedure was carried out in the fume cupboard for safety reasons. Thereafter, the product (clear gel) was placed in an oven at 90 °C for 72 h to obtain a dry gel. The dry gel was then grounded using an agate mortar and pestle to obtain a white powder. In order to remove the organic phase and decompose the La(NO₃)₃, the powder was heat treated in a furnace at 300 °C. Eventually, the resultant powder was calcined at the several temperatures including 750 °C, 900 °C, and 1000 °C. The final product was La₂O₃ NPs. The flow chart for the preparation of La₂O₃ NPs via the sol-gel technique is illustrated in Fig. 1. Within six trial samples, best three samples (PEG concentration: 30 g/l, 45 g/l and 60 g/l) in each calcination temperature, such as 750 °C, 900 °C and 1000 °C were taken for different measurements.

2.2 Characterization of Samples

An Equinox 3000 X-ray powder diffractometer was used to perform the structural characterization of the as synthesised La₂O₃ NPs in the 2 θ range from 10° to 60°, and step of 0.03°, with graphite monochromatic Cu K α radiation ($\lambda = 0.154 \text{ nm}$). The operating voltage and current was 35 kV and 25 mA, respectively. The surface morphology of all NPs samples was investigated using a Philips XL 30 ESEM equipped with Oxford INCA EDX FEG electron microscope operated at 20 kV at different magnifications. Specimens were prepared by dispersing

La₂O₃ NPs in absolute ethanol under ultra-sonication, dropping a little solution onto thin Cu coating film which was set previously on a steel stab and evaporating the solvent naturally in air. Then resulting specimens were sputter coated with a thin layer of platinum in vacuum. The EDS of the samples was performed by a Philips XL 30 ESEM and EDX FEG electron microscope operated at 10 kV. The FTIR spectra were recorded in transmittance (%) mode at room temperature by using a double beam IR spectrophotometer (Nicolet 8600 FTIR spectrometer) in the wavenumber range from 400 to 4000 cm⁻¹. To obtain the IR spectra, a ratio of 1:100 of the sample and potassium bromide, KBr were used. The background using a control sample of KBr was always taken prior to each measurement. The applied resolution and number of scan was 4 cm⁻¹ and 100 per minute, respectively. TG and DSC analysis were performed at room temperature 25 °C to 1000 °C at a heating rate of 10 °C /min under argon gas flow by a NETZSCH Thermal Analysis STA 449C. A QuantaMasterTM 510 spectrofluorometer (HORIBA Jobin Yvon GmbH, Germany) was used to record photoluminescence spectra at room temperature with excitation wavelength, $\lambda_{ex} = 300$ nm.

3. Results and discussion

3.1. XRD analysis

Fig. 2 illustrates the X-ray diffraction patterns of La₂O₃ NPs prepared with PEG concentration of 60 g/l calcined at 750 °C, 900 °C and 1000 °C, and all these X-ray diffraction patterns reveal the overall crystal structure and phase purity of the La₂O₃ NPs. The diffraction peaks of all samples were indexed to (100), (002), (101), (102), (110), (200), (112), and (201) reflections corresponding to the pure hexagonal phase with lattice constant, $a = b = 0.3973$ nm and $c = 0.6129$ nm (JCPDS card No. 83-1348). The strong and sharp diffraction peaks of all samples confirm the good crystallinity of the La₂O₃ NPs. The broader peaks suggest the nanometer-sized structures of the La₂O₃ NPs. It is observed that with increasing calcination temperature from 750 °C to 1000 °C the height of the peak increases and full width at half maximum (FWHM) decreases, that is diffraction peaks become stronger and sharper indicating the crystallinity of the samples improve and the size of particles getting larger [36, 37].

The lattice constants, a , b and c of the unit cell for hexagonal structure of La₂O₃, were calculated by the following equation [39]:

$$\frac{1}{d_{hkl}^2} = \frac{4}{3} \left(\frac{h^2 + k^2}{a^2} \right) + \frac{l^2}{c^2} \quad (1)$$

1 where d_{hkl} is the interplanar spacing of the atomic planes and hkl is Miller indices. The calculated
2 values of a , b and c is listed in **Table 1**. The obtained results are well agreement with the reported
3 values [40].
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5 The width of the diffraction peak varies with the crystallite size of the material. The
6 crystallite size, D of sample material can be estimated by using the Scherrer's formula [41]:
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$$9 \quad D = \frac{M\lambda}{\beta \cos\theta_B} \quad (2)$$

10 where D is the diameter of the crystallite, β is the FWHM of the selected diffraction peak, θ_B is the
11 Bragg angle, λ is the wavelength of the X-ray used, and M is a constant of which the value depends
12 on the shape of the crystallite.
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14 The FWHM of the XRD peaks may also contain contributions from lattice strain, ε_{str} and
15 the strain induced in powders owing to crystal imperfection and distortion can be calculated by
16 using the Stokes-Wilson equation [42]:
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$$19 \quad \varepsilon_{str} = \frac{\beta_c}{4 \tan\theta} \quad (4)$$

20 The values of average crystallite size and lattice strain determined by using Equations (3) and (4)
21 and values are tabulated in **Table 2**. It is observed from the data in the **Table 2** that the average
22 crystallite size decreases while lattice strain increases with increasing concentration of PEG in the
23 sol. On the contrary, average crystallite size increases while lattice strain decreases with increasing
24 calcination temperature. The similar result was reported by Wang *et al.* [36].
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27 A variety of properties of the materials is strongly affected by crystallographic defects or
28 irregularity within a crystal structure. This defects or irregularity is known as dislocation. The
29 dislocation density, δ for La_2O_3 NPs can be determined by the expression [43]:
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$$32 \quad \delta = \frac{15\beta \cos\theta}{4aD} \quad (5)$$

33 In crystalline solids, atoms or molecules exist in a periodic nature and repeat fixed distance
34 positions, called unit cell parameter. The unit cell volume, V for hexagonal La_2O_3 NPs can be
35 calculated by the relation below [44]:
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$$38 \quad V = \frac{\sqrt{3}}{2} a^2 c \quad (6)$$

39 The calculated values of δ and V for all samples are listed in **Table 2**.
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1 The crystallinity of the sample material can be represented by the crystal index, C_i and can
2 be calculated by the following equation [35]:

$$3 \quad C_i = \frac{I_{max}}{\beta_c} \quad (7)$$

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7 In this case I_{max} is the maximum intensity of the XRD peak and β_c is the value of corrected FWHM.

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9 The obtained value of dislocation density, unit cell volume and crystal index are listed in Table 2.
10 It is found that dislocation density is higher for samples calcined at 900 °C presenting the higher
11 concentration of crystal imperfections in the samples. It is apparent that samples calcined at
12 temperature 1000 °C have larger unit cell volume as compared to the other samples.
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18 19 20 **3.2 Surface Morphology and Particle Size Measurement**

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22 Fig. 3, Fig. 4 and Fig. 5 show SEM micrographs of dispersed La₂O₃ NPs prepared with
23 PEG concentration of 60 g/l calcined at 750 °C, 900 °C and 1000 °C, respectively at different
24 magnifications. In this case, to get average crystallite size for every sample, at least ten crystallites
25 were considered and their diameter were measured from a software connected with ESEM which
26 was marked in every samples. The nanoparticles were dispersed in ethanol with the help of an
27 ultrasonic bath (probe power level 3 and power density of 0.275 W/mL) for 20 min. From all
28 Figures, it can be seen that the La₂O₃ NPs are uniformly dispersed and mostly spherical in shape
29 with diameters from approximately 32 nm to 56 nm. This can be attributed to the fact that the
30 acoustic waves transmitted by ultra-sonication are effective in dispersing La₂O₃ NPs owing to the
31 transient cavitations and acoustic streaming that can possibly redefine the shape and structure of
32 NPs and change the surface morphology [45]. From the results [shown in supplementary Figures],
33 it can be seen that the particle size of the as synthesized La₂O₃ NPs decreased with increasing PEG
34 concentration. This can be attributed to the enhanced steric hindrance by the adsorption of polymer
35 polyethylene glycol on the surfaces of lanthanum oxide particles. That is the polyethylene glycol
36 polymer surrounds the crystal nuclei of lanthanum nitrate and prevents their growth which creates
37 steric hindrance effect resulting to reduce particle-particle aggregation. When the concentration of
38 PEG is not enough to fully cover the crystal nuclei, the growth of crystal is not impeded
39 effectively by adsorbing PEG. As a result, bigger particles will be acquired. On the other hand, if
40 the concentration of PEG rises, then the coverage degree of PEG on the crystal nuclei will increase
41 and consequently average particle size is thinly decreased. At the same time, the particle-particle
42 agglomeration is gradually reduced [36].
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1 The change of the average particle size with calcinations temperature is summarized in
2 **Table 3** to understand the influence of particle size on the calcination temperature clearly. It can be
3 concluded from the result shown in **Table 3** that the particle size is gradually increased with the
4 increase in calcination temperatures. This is fact can be explained as that at low temperature the
5 particles grow gently while at higher temperature particles grow very rapidly to form
6 agglomeration. The similar type of results was found by earlier researchers [36].
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10 **3.3 Chemical Composition Study**

11 The EDS spectra of NPs La_2O_3 NPs prepared with PEG concentration of 60 g/l calcined at
12 750 °C, 900 °C and 1000 °C are represented in **Fig. 6**, respectively. The EDS spectrum shows
13 presence of lanthanum, oxygen, carbon, and platinum. The small carbon peak was originated from
14 the tiny carbon tape mounted on a stainless steel stab which was applied during the sample
15 preparation for SEM. The Pt peak was resulted from platinum coating of the samples which was
16 used to make the sample highly conductive. The weight percentage (wt. %) of the different
17 elements present in all samples are listed in **Table 4**. It is observed from the results shown in **Table**
18 **4**, that highest weight percentage of Lanthanum (La) is present in the sample while a good amount
19 of Oxygen (O) is also observed. The theoretical percentage of lanthanum in La_2O_3 NPs is 85.3%
20 and that of oxygen is 14.73 % while the EDX results showed on the average 75.03 % of lanthanum
21 and 19.76 % of oxygen. These obtained results confirmed a good agreement with previous work
22 [35]. The EDX analysis indicates no presence of any impurity.
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37 **3.4 Thermal analysis of La_2O_3 NPs**

38 **Fig. 7** illustrates the TG-DSC curves of the lanthanum dried gel powder prepared at PEG
39 concentration of 60 g/l. The thermogram of **Fig. 7** shows four different regions indicated by letter
40 A, B, C and D. These regions are indication of different weight losses of the sample at different
41 range of temperature. The weight loss approximately 1.5 % in the first region (A) between
42 temperature 30 to 250 °C corresponds to the removal of surface absorbed water of the sample and
43 because of this a small endothermic peak in DSC curve is obtained at about 95 °C. In the second
44 region (B) between temperature 250-400 °C, DSC curve indicates a sharp endothermic peak at
45 380°C and the corresponding weight loss in the in this region is 8.25 %. This can be explained as
46 the evaporation of the surfactant (PEG), whose boiling point is 260 °C. The weight loss (2.75 %) in
47 the third region, C is due to the oxidation of the residual organic compounds from surfactant, PEG.
48 A small endothermic peak in this region at a temperature 510 °C is also observed. Finally, in the
49 fourth region (D) an endothermic peak at 750 °C in DSC curve is obtained and the corresponding
50 weight loss in this region is around 3.5 %. This is because of the final decomposition of lanthanum
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1 compound of the sample [36]. That is, PEG and all others lanthanum compound of the sample
2 decomposed completely at 750 °C. Hence, 750 °C was chosen as the minimum calcination
3 temperature.
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6 **3.5 Fourier Transform Infrared Spectroscopy analysis**

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9 The FTIR spectra in the wavenumber range 4000-400 cm^{-1} of the as-prepared La_2O_3 NPs
10 samples are depicted in Fig. 8. At the spectrums of the samples with PEG concentration of 60 g/l
11 calcined at 750 °C, 900 °C and 1000 °C in Fig. 8, a broad band is observed at the wavenumber of
12 3439 cm^{-1} (A), 3424 cm^{-1} (A) and 3442 cm^{-1} (A), respectively and these bands show the presence
13 of O-H stretching vibration which is due to absorbed moisture on the surface of the samples [36].
14 The absorption peaks at around 1459, 1460 and 1464 cm^{-1} (B) as well as sharp absorption bands at
15 1362, 1363 and 1363 cm^{-1} (C) in the samples is found because of the asymmetric and symmetric
16 stretching of COO^- , respectively [37]. The band at 1067 cm^{-1} (D) in three samples is for C-O
17 stretching vibration but in the same samples, the sharp absorption bands at around 856 cm^{-1} (E) is
18 owing to C-O bending vibration. The broad absorption bands at 648, 678 and 663 cm^{-1} (F) in three
19 samples is observed because of the La-O stretching vibration while the small bands at 510, 510
20 and 514 cm^{-1} (G) in the same samples is due to La-O bending vibration. The presence of above
21 mentioned bands therefore confirmed the existence of La_2O_3 [38]. The location of all the band
22 frequencies and their corresponding modes of vibration are recorded in Table 5 for better
23 comparison.
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40 **3.6 Photoluminescence Spectroscopy Study**

41 The Photoluminescence (PL) spectra of different La_2O_3 NPs samples are illustrated in Fig.
42 9. From Figures 9 it is seen that the strongest emission band is found to be located at the
43 wavelength of 364 nm for all samples. The observed emission band at 364 is a typical green band.
44 This may be ascribed to recombination of a delocalized electron near to the conduction band with
45 a single charged state of a surface oxygen vacancy [46]. The higher fluorescence intensity of the
46 La_2O_3 sample is an indication of the higher surface-to-volume ratio and due to higher density of
47 single ionized oxygen vacancies, resulting from the small particle size as has been suggested by G.
48 Wang *et al.* [47]. A small band is also observed at 465 nm for all samples.
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4. Conclusions

La₂O₃ nanoparticles were successfully synthesised via a simple sol-gel technique and the effect of calcinations temperature on the size of nanoparticles was also investigated. All of the diffraction peaks of La₂O₃ NPs obtained from XRD data were indexed to (100), (002), (101), (102), (110),(200), (112), and (201) reflections corresponding to the pure hexagonal phase. The average crystal size calculated by the Debye Scherrer's equation was found to be in the range of 18.14-29.31 nm while that of estimated from ESEM micrographs for different dispersed La₂O₃ NPs samples were found from 32.59 nm to 55.73 nm. The particle size was found to be increased with increasing calcination temperature while that of decreased with increasing concentration of the PEG in the solution. The EDS study confirmed the presence of Oxygen and Lanthanum in the synthesized nanoparticles. The thermograms of all samples showed three main thermal transitions associated with weight loss. The FTIR spectroscopy suggested that all samples exhibited a broad band in the range of 3442-3424 cm⁻¹ associated with O-H stretching vibrations in absorbed water molecules. Absorption bands in the frequency range 1067 cm⁻¹ associated with C-O stretching vibrations were observed, while C-O bending vibrations appeared in the range of 856-845 cm⁻¹. La-O stretching and bending vibrations were observed in the wavenumber range of 678-648 cm⁻¹ and 514-510 cm⁻¹, respectively. The Photoluminescence spectra of different samples of La₂O₃ NPs presented a strong emission band located at the wavelength of 364 is typical green band.

Conflict of interest statement

The authors declare that they have no conflict of interest.

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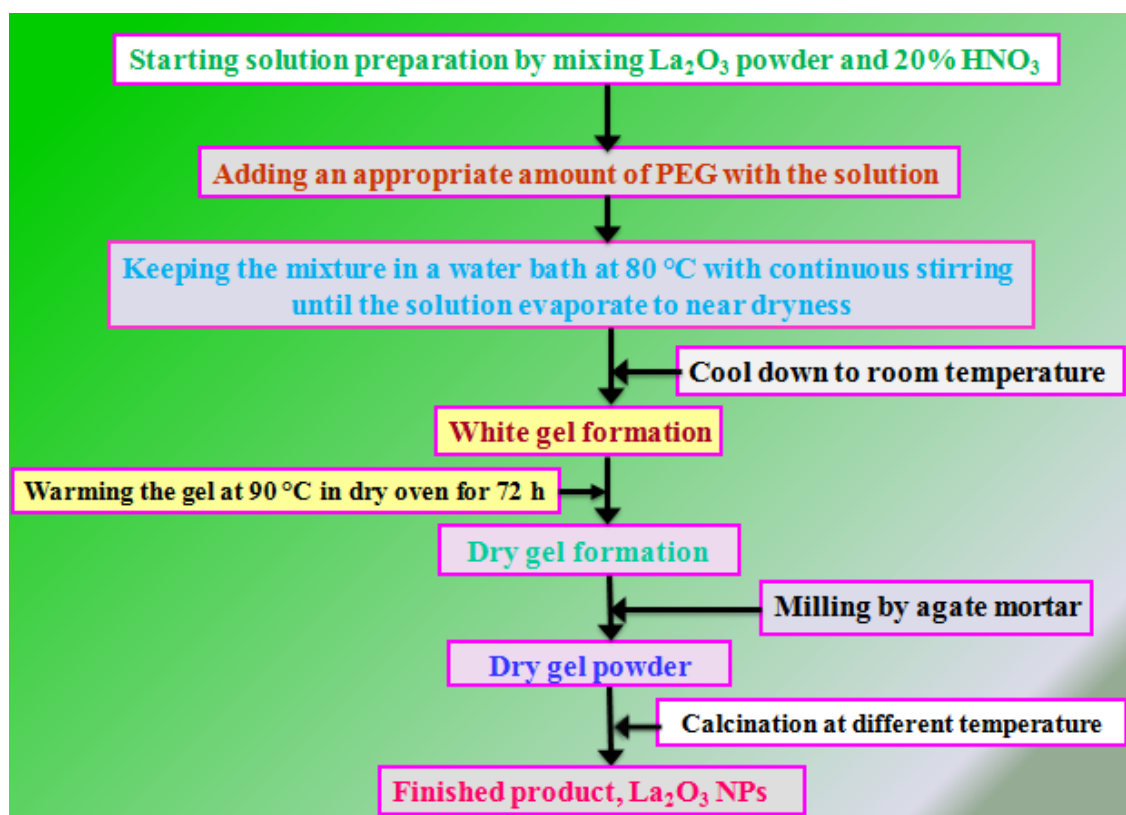


Fig. 1. Flow chat for the synthesis of La_2O_3 NPs via sol-gel technique.

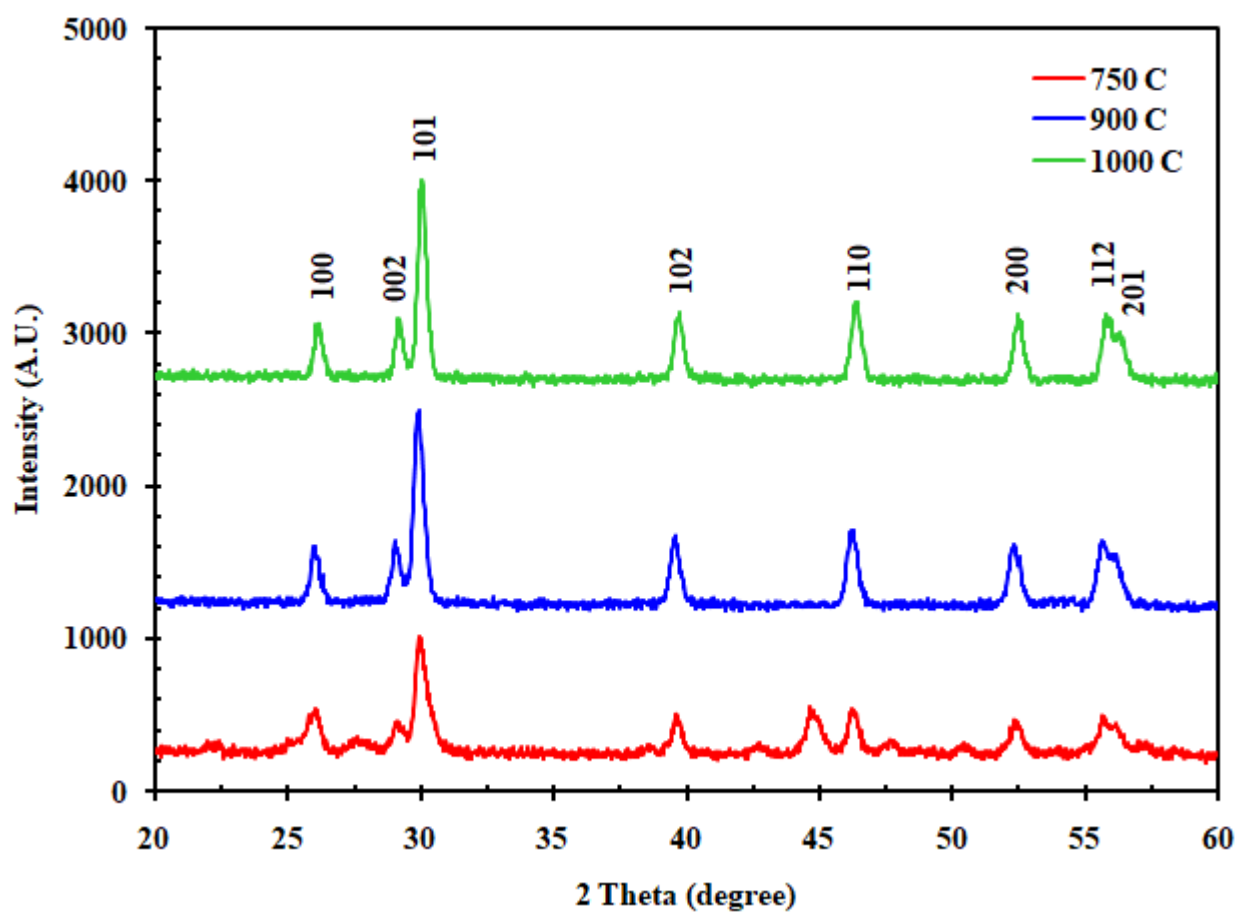


Fig. 2. XRD patterns of La_2O_3 NPs samples calcined at $750\text{ }^\circ\text{C}$, $900\text{ }^\circ\text{C}$ and $1000\text{ }^\circ\text{C}$ [PEG concentration 60 g/l].

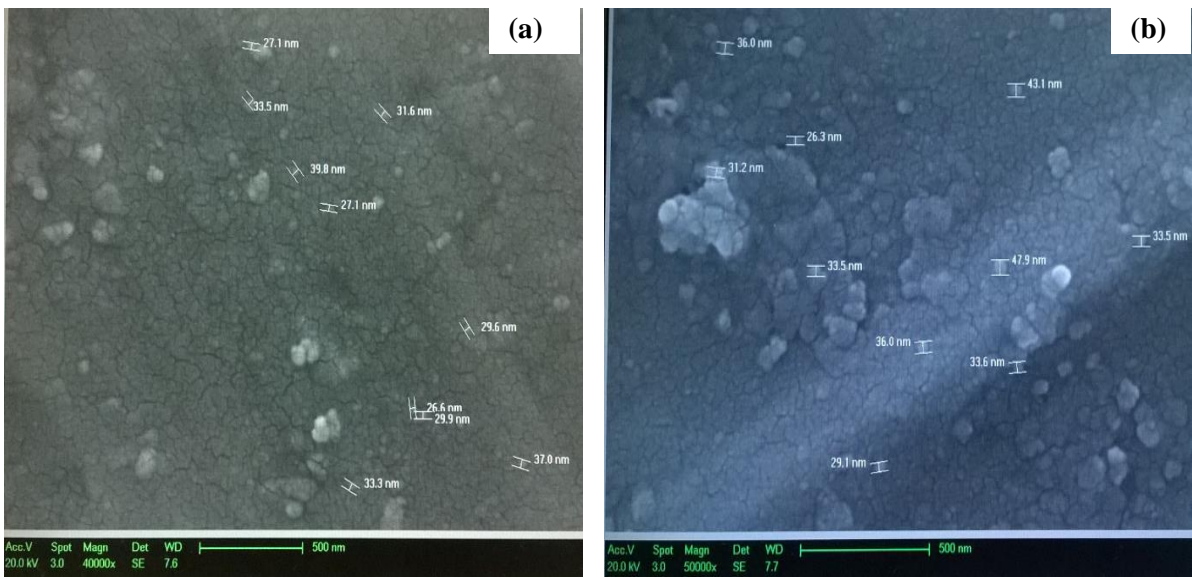


Fig. 3: SEM micrographs of dispersed La_2O_3 NPs sample calcined at 750°C , at magnification (a) 40k \times , (b) 50k \times .

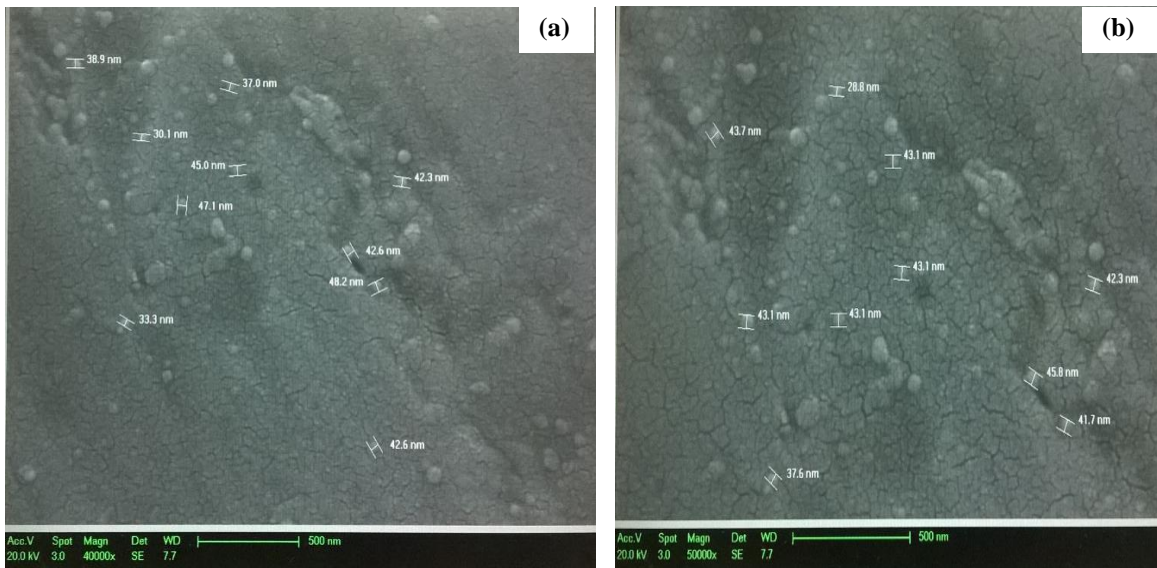


Fig. 4: SEM micrographs of dispersed La_2O_3 NPs sample calcined at 900°C , at magnification (a) 40k \times , (b) 50k \times .

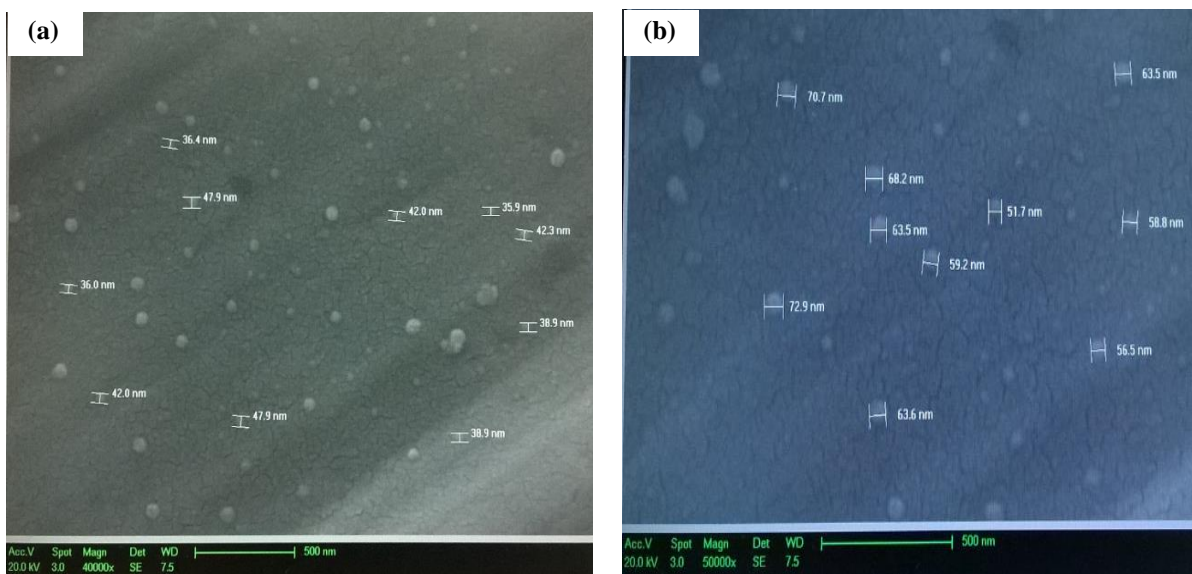


Fig. 5: SEM micrographs of dispersed La_2O_3 NPs sample calcined at 1000°C , at magnification (a) 40k \times , (b) 50k \times .

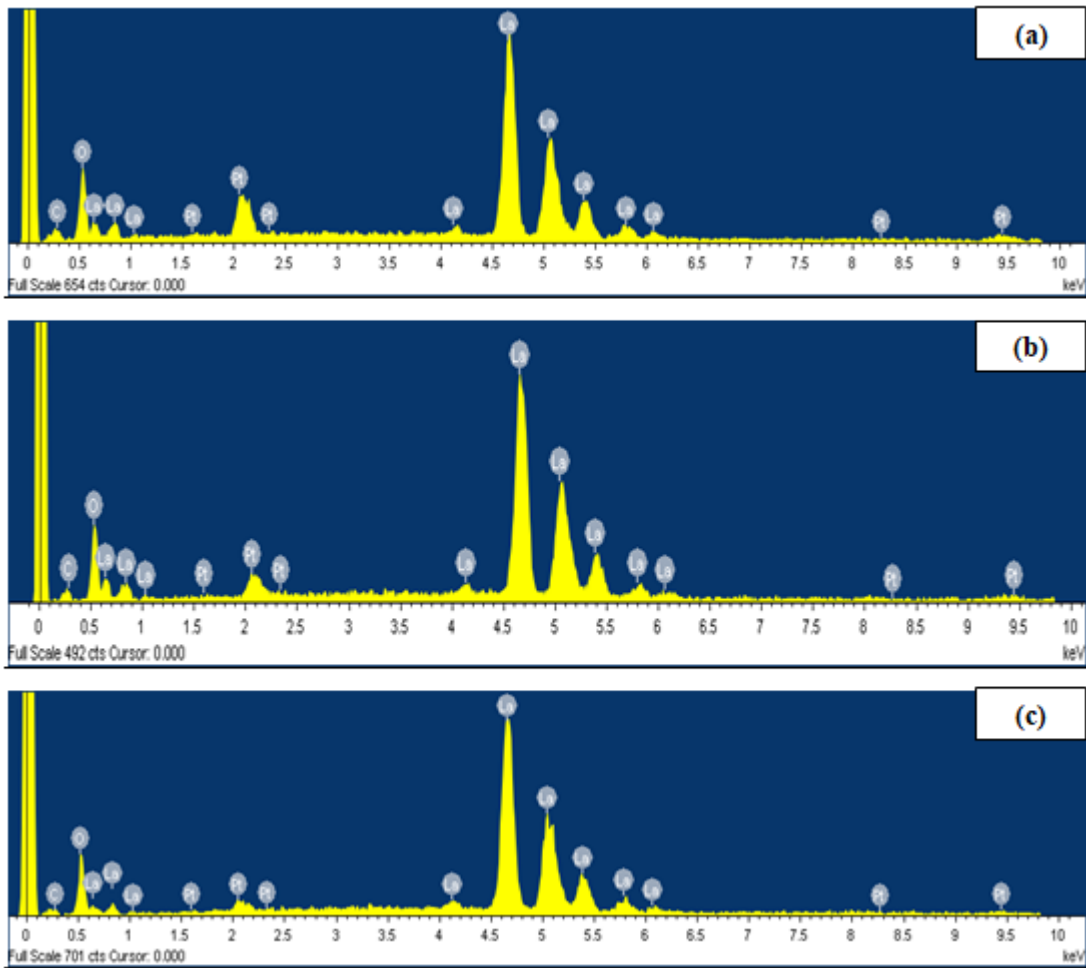


Fig. 6: EDS spectra of La_2O_3 NPs samples calcined at 750 °C, 900 °C and 1000 °C [PEG concentration 60 g/l].

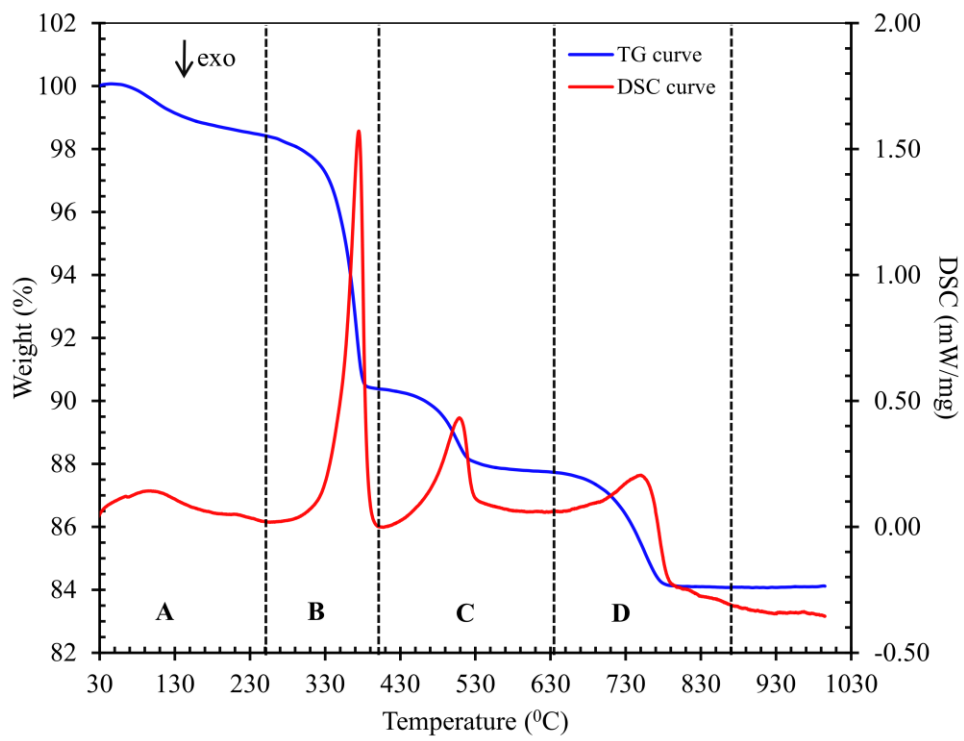


Fig. 7: TG-DSC traces of the lanthanum compound dried gel powder prepared at PEG concentration of 60 g/l.

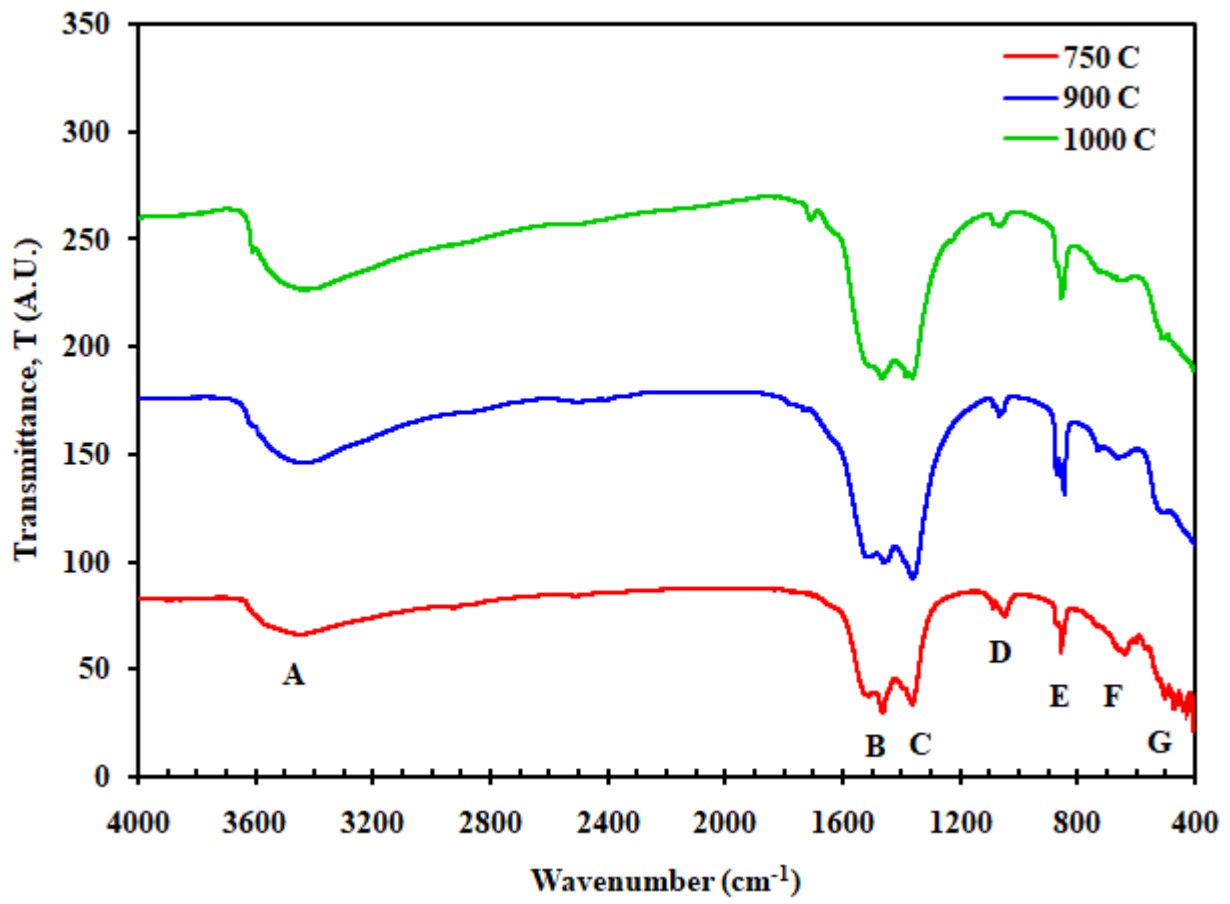


Fig. 8: FTIR spectra of La₂O₃ NPs samples calcined at 750 °C, 900 °C and 1000 °C [PEG concentration 60 g/l].

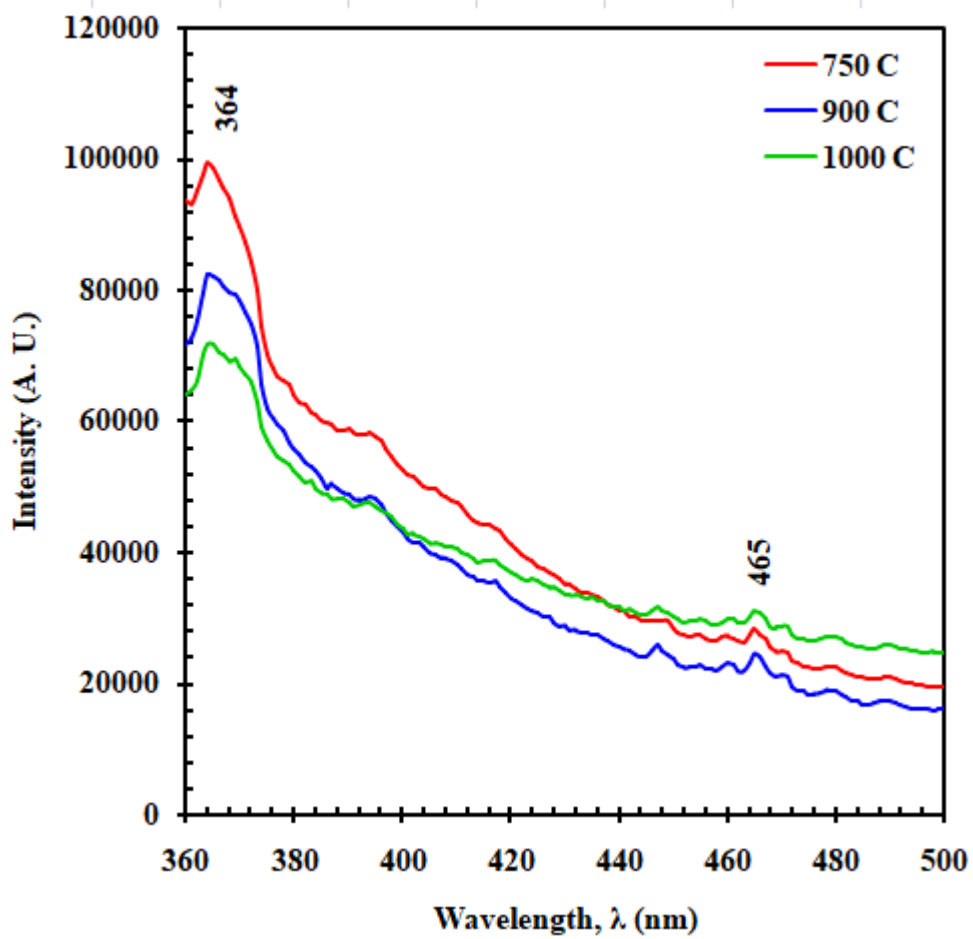


Fig. 9: PL spectra of La₂O₃ NPs samples calcined at 750 °C, 900 °C and 1000 °C [PEG concentration 60 g/l].

Table 1. Determination of interplanar distance and lattice constants of La₂O₃ NPs.

Sample (Calcination Temp.)	Interplanar distance, d_{hkl} (Å)			Lattice constants, a and b (Å)			Lattice constants, c (Å)			Average values of a or b (Å)	Average of c (Å)
	(100)	(002)	(110)	(100)	(002)	(110)	(100)	(002)	(110)		
750 °C	3.4219	3.0688	1.9630	3.9513	3.9397	3.9260	6.0801	6.1377	6.2081	3.9390	6.1420
900 °C	3.4144	3.0750	1.9595	3.9426	3.9274	3.9190	6.0741	6.1499	6.1936	3.9297	6.1392
1000 °C	3.4174	3.0664	1.9595	3.9461	4.0885	3.9190	6.057	6.1328	6.1936	3.9845	6.1278

Table 2. Crystallographic parameters of La₂O₃ NPs.

Sample	Crystallite size, D (nm)	Lattice strain, ϵ	Dislocation density, δ	Unit cell volume, $V(\text{Å}^3)$	Crystal index, C_i	c/a ratio
750 °C	18.14	0.0077	0.0240	82.5274	1561.16	1.5593
900 °C	25.37	0.0055	0.1081	82.0993	4056.49	1.5623
1000 °C	29.31	0.0048	0.0089	84.2509	3775.58	1.5379

Table 3. Average particle size for different dispersed samples of La₂O₃ NPs:

Sample	Magnification(k \times)	Particle size (nm)	Average particle size (nm)
750 °C	40	31.55 \pm 3.49	32.59 \pm 4.04
	50	35.02 \pm 4.58	
900 °C	40	40.81 \pm 4.59	41.71 \pm 3.90
	50	41.23 \pm 3.21	
1000 °C	40	40.82 \pm 3.60	55.73 \pm 4.32
	50	62.86 \pm 5.05	

Table 4. The weight percentage (wt. %) of the different elements present in different samples of La₂O₃ NPs.

Sample	Element present in Wt%		
	La	O	C
750 °C	67.81	25.23	6.96
900 °C	69.83	24.04	6.13
1000 °C	80.20	15.92	3.88

Table 5. Assignments of FTIR absorption peaks for La₂O₃ nanoparticles samples.

Assignments	Wavenumber (cm ⁻¹)		
	As prepared La ₂ O ₃ nanoparticles		
	750 °C	900 °C	1000 °C
O-H stretching vibration (A)	3439	3424	3442
Asym. stretching of COO ⁻ (B)	1509, 1459	1510, 1460	1464
Symmetric stretching of COO ⁻ (C)	1362	1363	1363
C-O stretching vibration (D)	1067	1067	1067
C-O bending vibration (E)	856, 845	856, 845	855
La-O stretching vibration (F)	648	678	663
La-O bending vibration (G)	510	510	514



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The Editor

Journal of Applied Physics A

Submission of Full-Length Article to Journal of Applied Physics A

With reference to the above, I attach herewith an article entitled “Influence of Calcination on Sol-Gel Synthesis of lanthanum oxide nanoparticles” for submission to *Journal of Applied Physics A* as a full-length research paper.

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