

**SYNTHESIS AND CHARACTERIZATION OF LANTHANUM OXIDE
NANOPARTICLES**

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ABSTRACT

In this investigation, we report facile synthesis of metal oxides (La_2O_3) as nanoparticles. The process contained two steps: first preparation of complex $[\text{La}(\text{acacen})(\text{NO}_3)(\text{H}_2\text{O})]$ (where acacen= acetylacetonatoethylenediamine. Then the precursors were calcination. In first step, complex synthesized using a methanolic solution of $\text{La}(\text{NO}_3)_3$ and acetylacetonatoethylenediamine. Then desired product was collected by suction filtration and characterized using Fourier transform infrared (FT-IR), Ultra Violet-Visible (UV-vis) spectroscopy and scanning electron microscope (SEM).

KEYWORDS: nanoparticles, FT-IR, Ultra Violet-Visible, SEM.**INTRODUCTION**

In recent years, the synthesis of inorganic materials with specific size and morphology has attracted considerable attention. Their excellent physical and chemical properties in various fields, such as solar cells, catalysis, photo detectors, sensors, light emitting diodes and laser communication, have made them attractive.^[1] and promising materials. It is well documented that materials with nano-scale grain size show different properties relative to the same material in bulk form.^[2-3] Preparation of various nano-materials have many different synthetic approaches such as chemical vapor deposition (CVD), thermal evaporation, chemical bath deposition, laser ablation hydrothermal, homogeneous precipitation in an organic matrix, sonochemical and sol-gel method. The synthesis, production and manipulation of materials on the Nano scale is one of the favorite areas of research which also attracts the industrialists for designing and fabricating new functional materials with novel special properties. Among the lanthanides, lanthanum has been extensively examined for its unique properties.^[4] and lanthanum has been synthesized in various compositions such as $\text{La}(\text{OH})_3$, LaF_3 , $\text{La}_2(\text{CO}_3)_3$, LaPO_4 , LaBO_3 , LaOF , $\text{La}_2\text{Sn}_2\text{O}_7$, La_2O_3 nanoparticles.^[5] Lanthanum oxide (La_2O_3) powders have a lot of attractive properties for industrial and technological applications such as: 1- Nanometer lanthanum oxide can be used in piezoelectric materials to increase product piezoelectric coefficients and improve product energy conversion efficiency; 2- Nano-lanthanum oxide can be used for the manufacture of precision optical glass, high refraction optical fiber, all kinds of alloy materials; 3- Nano-lanthanum oxide can be used for the preparation of organic chemical products

catalysts, and in automobile exhaust catalyst; Nanometer Lanthanum oxide can improve the burning rate of propellant, is a promising catalyst; As regards the photoelectric conversion efficiency of nanolanthanum oxide is high, it can be used in lightconverting agricultural film; Also, nano-lanthanumoxide can be used in electrode materials and in light-emitting material (blue powder), hydrogen storage materials, laser materials etc. In the present work, we report the synthesis of $[\text{La}(\text{acacen})(\text{NO}_3)(\text{H}_2\text{O})]$, then this compound has been convert to Lanthanum oxidesnanoparticle as La_2CO_5 at 600°C and La_2O_3 at 900°C

Experimental**Material and characterization**

All chemicals and solvents were reagent grade or better, obtained from Merck, and used without further purification. Infrared spectra ($4000\text{--}250\text{ cm}^{-1}$) of solid samples were taken as 1% dispersion in KBr pellets using a JASCO FT-IR 460 PLUS. Electronic absorption spectra in DMF solution were taken at room temperature on a JASCO 7850 spectrometer. The crystallite sizes of selected samples were estimated using the Scherer method. SEM images of the samples were collected on Philips XL30 equipped with an EDX microanalysis (Tarbiat Modares Uni.).

Synthesis of $[\text{La}(\text{acacen})(\text{H}_2\text{O})(\text{NO}_3)]$

Synthesis of $[\text{La}(\text{acacen})(\text{H}_2\text{O})(\text{NO}_3)]$ to a methanolic solution (10 ml) of $\text{La}(\text{NO}_3)_3$ (0.1 M) was added drop wise aqueous solution acetylacetonatoethylenediamine (0.1 M). The resulting solution was stirred at $55\text{--}60^\circ\text{C}$ for 2h. The solid (desired product) was collected by

suction filtration, washed with MeOH solution, then air dried (Yield %34). Anal. Calc: C, %32. 22; H, %5.86; N, %9. 4). Found: C, 32.12; H, 5.73; N, 9.54. IR (KBr, cm^{-1}): 3419 and 2928 $\nu(\text{CH})$, 1644 $\nu(\text{N-O})$, 1530- $\nu(\text{C}=\text{C})$ and $\nu(\text{C}=\text{N})$, 3454 $\nu(\text{O-H})$, 1303 $\nu(\text{conjugated NO}_3^-)$, 900 $\nu(\text{C-O})$

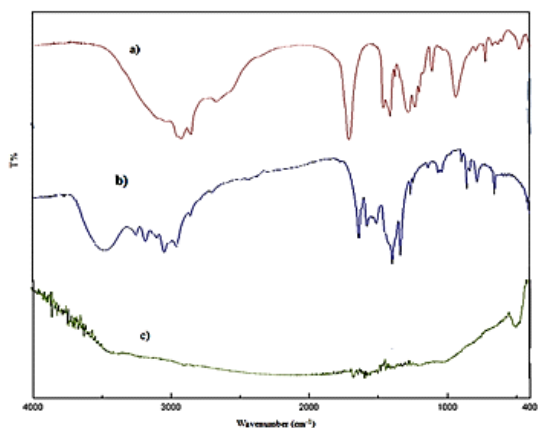
Synthesis of nanoparticles La_2O_3

Lanthanum oxide nanoparticles were prepared from [La (acacen) (H_2O) (NO_3)] using the physical method. First 1.0 g of compound dissolved in oleic acid solution (5mL) as a surfactant, then calcinated at 600°C and 900°C for 4h, to get La_2O_3 nanoparticles respectively. The final products were collected and washed with distilled water and absolute ethanol several times, dried in air and kept for further characterization.

RESULTS AND DISCUSSION

FT-IR spectroscopy

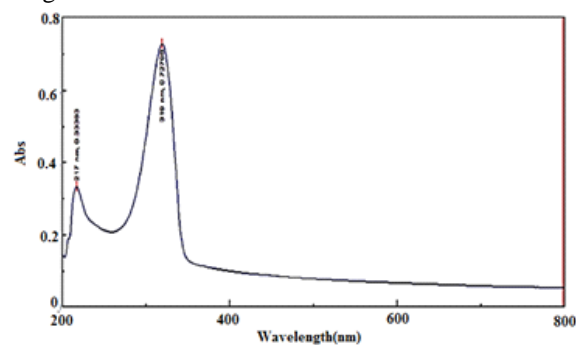
The broad bands at $2600\text{-}3000\text{ cm}^{-1}$ and 1712 cm^{-1} assigned to stretching vibration of O-H and C=O group in oleic acid respectively. As it can be observed in all the bands of the oleic acid and [La (acacen) (H_2O) (NO_3)] were disappeared when the product was calcinated in 900°C and only broad band was appeared that attributed to lanthanum oxide. The strong band at 500 cm^{-1} is assigned to the $\nu(\text{La-O})$, indicating formation of La_2O_3 . Strong bands at 1585 , 1505 and 1625 cm^{-1} [6-8], related to the stretching vibrations of C=C, C=N, C=O can be shared on acacen ligand. And stretching frequency observed in the 1374 cm^{-1} attributed to NO_3^- group. Weak absorptions in the region 3158 and 3226 cm^{-1} assigned to N-H vibrations of the acacen ligand. FT-IR band observed at $2925\text{-}2000\text{ cm}^{-1}$, [9] attributed to overlap of stretching vibration frequency of the acidic OH screw with CH bands. The bands observed at 3006 cm^{-1} , 1711 cm^{-1} , 1378 cm^{-1} and 1465 cm^{-1} assign to CH, C=O, CH₂ and CH₃ groups, respectively.



Electronic Excitation Study

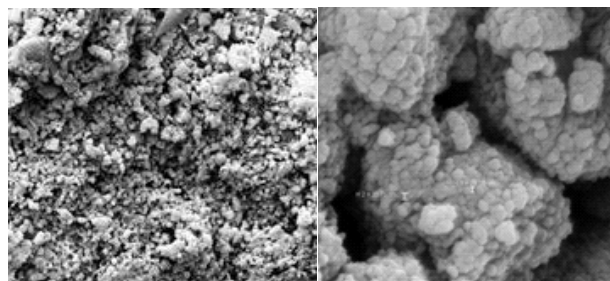
The electronic spectrum of the [La (acacen) (H_2O) (NO_3)] (1) showed in Fig2. Absorption bands in 217 and 319 nm can be assigned to the intra ligand transition of the aromatic ring and $\pi \rightarrow \pi^*$ transition of the acacen and nitrate ligands, respectively. There was any transition in

visible region due to Lanthanum (III) has d^0 electron configurations. [10,13]



Surface Morphology

The morphology of the La_2O_3 nanoparticles was characterized using scanning electron microscope (SEM) at 600°C . Show below the SEM images of La_2O_3 in absence and presence of oleic acid respectively. As can be seen the images are clear examples and nano carbonate oxide relatively spherical, porous and beneath the high porosity for use as a catalyst for much is suitable. So that the particle size is found $29.0\text{-}35.73\text{ nm}$ and $22.33\text{-}26.89\text{ nm}$ respectively that in order to the effect of surfactants on the image more uniform and stronger link between particles surface area that can provide a better view. [14-17]



CONCLUSION

Metals, rare earths, has a strong tendency to combine with non-metallic element such as oxygen, nitrogen, carbon and hydrogen, and various factors such as geometry and central atom, the structural features of the ligand molecules, with a solvent can play key roles in the formation of structures them. The nano-sized particles have different properties than the bulk material can be explained by the fact that the smaller the particle size, the number of atoms will be higher. As a result, the surface energy of atoms within a material volume with a different energy and this will cause different properties. The unique properties of nanometer particles, not only because of their high level, but also for other reasons such as changes in the crystal lattice, the link between nuclear energy and so is also relevant. It should be noted that the production of nano-materials and in the summary, we prepared Lanthanum oxide nanoparticles in the presence and absence of Oleic acid as a surfactant. The size of these nanoparticles was measured using FTIR and SEM.

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