https://doi.org/10.70882/wt9szz52

SCIENCE FORUM (JOURNAL OF PURE AND APPLIED SCIENCES) 26, (5 Special) (2026), 745 - 751

Science Forum (Journal of Pure and Applied Sciences)

Journal Homepage: https://atbuscienceforum.com.ng/index.php/jpas

Sol–Gel Synthesis of Nanocrystalline γ-Al_2O_3 Physiochemical properties and Morphological Study

Bassima M. Mashkour

Ministry of Education, Wasit Education Directorate/ Kut/Wasit/Iraq

Abstract

Aluminium oxide (Al_2O_3) nano-crystalline powder was synthesized by sol–gel method using aluminium nitrate nonahydrate (1 M) as a precursor of aluminium, and then calcined at 800 °C. X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR) and field-emission scanning electron microscopy (FE-SEM) were used to study the structural and morphological features of the calcined product. The XRD pattern showed the presence of the cubic spinel type γ-Al2O3 phase with the three most intense reflections at 2θ = 37.63°, 46.46° and 67.58° corresponding to the planes (311), (400) and (440) respectively. The interplanar spacing values obtained from the measurements were found to be in good agreement with the standard values and the average crystallite size obtained from Scherrer equation was 16.9nm. FTIR spectrum showed the oxide framework by Al–O and Al–O–Al bands in the region 455-818 cm−1 and the presence of O–H bands due to surface hydroxyl groups and adsorbed moisture. FE-SEM micrographs revealed semi-spherical, partially agglomerated particles while the statistical analysis of sixty particles fitted with a Gaussian distribution yielded the mean particle diameter of 17.34 ± 5.09 nm, which was in good agreement with the crystallite size calculated from XRD. The results show that the easy and reproducible sol–gel method using the 1 M nitrate precursor with calcination at 800 °C is a suitable method for the synthesis of single-phase nanocrystalline γ-Al2O3, suitable for catalytic, adsorption and ceramic applications.

Keywords

γ-Al_2O_3;
sol–gel;
aluminium nitrate;
XRD;
FTIR;
crystallite size;
nanoparticles

EISSN: 3156-5190 (Online)
ISSN: 1119-4618 (Print)
JPAS
Faculty of Science, ATBU Bauchi

Corresponding Author: Bassima M. Mashkour, Ministry of Education, Wasit Education Directorate/ Kut/Wasit/Iraq
Email: etts341@gmail.com © 2026 Faculty of Science, ATBU Bauchi. All rights reserved

1. Introduction

Aluminium oxide (alumina, Al_2O_3) is one of the most widely studied ceramic oxides owing to its combination of high melting point, chemical inertness, mechanical hardness, wide band gap, and low cost. These properties have secured its use in catalysis and catalyst supports, adsorbents for water treatment, abrasives, wear-resistant coatings, dielectric layers, and biomedical components [1]–[3]. Alumina exists in several polymorphs, including the transition phases γ, δ, θ, and η in addition to the thermodynamically stable α phase (corundum). Among these, γ-Al_2O_3 occupies a special position because it crystallizes in a defective cubic spinel structure that provides a large specific surface area and abundant surface acid sites, making it the phase of choice for catalytic and adsorption applications [2], [3].

The phase composition, crystallite size, and morphology of alumina are strongly governed by the synthesis route and the thermal treatment applied to the precursor [4]. Numerous techniques have been reported for producing nanostructured alumina, including co-precipitation, hydrothermal processing, combustion synthesis, and sol–gel chemistry [4]–[7]. The sol–gel process is especially appealing because the processing is done at the solution level, thus resulting in high chemical homogeneity; the stoichiometry can be controlled at the solution level; and modest processing temperatures and simple equipment are required [1, 4]. Aluminium nitrate is a cheap, highly soluble salt which can be used as a convenient raw material for the preparation of the sol-gel process in aqueous solutions and decomposes cleanly when calcined without leaving any metallic impurities [5,8].

The final phase of sol–gel derived alumina is determined by the calcination temperature. The amorphous gel phase usually converts to γ-Al2O3 in the temperature range from about 700 to 900 °C, followed at higher temperatures by rather rapid crystallite growth and the concomitant decrease in surface area [6], [7], [9]. A calcination temperature of 800 °C is therefore a practical parameter where a well crystallized single γ phase can be achieved while the crystallites are still in the nanometre regime [6, [9]].

The nanocrystalline Al2O3 powder was prepared in the present work using aluminium nitrate aqueous solution (1 M) as starting material using sol–gel technique followed by calcination at 800 °C. The objectives of the study are to investigate the various structural properties of the product such as phase identification, lattice spacing, crystallite size, chemical bonding and the morphology of the particles by the XRD, FTIR and FE-SEM techniques and to compare the particle size as obtained from the FE-SEM technique with the lattice size of the crystallite as obtained from XRD.

2. Experimental Part

2.1. Materials

Aluminium nitrate nonahydrate (Al(NO3)3·9H2O, analytical grade) was used as a source of aluminium, citric acid monohydrate as chelating agent and aqueous ammonia solution as a pH adjuster. Throughout distilled water was used as the solvent. All chemicals were used as received and not purified.

2.2. Preparation of Al_2O_3 nanoparticles

The required amount of Al(NO3)3·9H2O was dissolved in distilled water with magnetic stirring at room temperature until a clear solution was achieved to make up a 1 M precursor solution. A complexing agent citric acid was then added and it was heated with constant stirring at around 80 °C. Ammonia solution was used as a diluting agent to adjust the pH so that hydrolysis and condensation of the aluminium species can be accelerated. Stirring and a slow heating continued until the sol slowly turned into a viscous transparent gel. The wet gel was placed in an oven at around 100 °C for a few hours to vacuum dry it of excess water and products of nitrate decomposition, yielding a dry xerogel. The xerogel was ground to fine powder and finally calcined in a programmable muffle furnace in air for 2 h at 800 °C and then cooled down to room temperature. The calcined product was a fine white powder that was kept in a sealed container before characterization.

2.3. Characterization techniques

The phase composition and crystal structure of the calcined powder was investigated by X-ray diffraction with Cu-Ka radiation (λ = 1.5406 Å) in the

range of 2θ from 10° to 80°. The broadening of the most intense peaks was used to estimate the average crystallite size, D, by applying the Scherrer equation[13] :

$$D = 0.9\lambda/(\beta \cos \theta)$$

where β is the FWHM of the diffraction peak, λ is the X-ray wavelength, and θ is the diffraction angle). Fourier Transform infrared spectroscopy was used to investigate chemical bonding and functional groups in the range 400-4000cm-1, using the KBr disc technique. The surface morphology and particle size were investigated using field-emission SEM, and the particle size distribution was determined by measuring the dimensions of sixty particles observed on the SEM images and fitting the distribution with a Gaussian function.

3. Results and Discussion

3.1. X-ray diffraction analysis

Figure 1 shows the XRD pattern of the Al_2O_3 powder calcined at 800 °C. The pattern displays the characteristic reflections of the cubic spinel γ-Al_2O_3 phase, with the three strongest peaks appearing at 2θ = 37.63°, 46.46°, and 67.58°, indexed to the (311), (400), and (440) planes, respectively. No diffraction lines belonging to α-Al_2O_3, boehmite, or any other secondary phase were detected, indicating that calcination at 800 °C was sufficient to complete the transformation of the dried gel into single-phase γ-alumina without initiating the transition toward the α phase, in agreement with the phase-evolution behaviour reported for nitrate-derived alumina gels [6], [7], [9]. The relatively broad profiles of the reflections are typical of γ-Al_2O_3 and reflect its nanocrystalline nature and intrinsically defective spinel lattice [2], [10].

The structural parameters extracted from the pattern are collected in Table 1. The experimental interplanar spacings of 2.388, 1.952, and 1.385 Å for the (311), (400), and (440) reflections are in close agreement with the corresponding standard values of 2.378, 1.971, and 1.394 Å, with relative deviations below about 1%, confirming the correct indexing of the cubic phase. The crystallite sizes calculated from the Scherrer equation for the three principal reflections were 13.26, 21.64, and 17.28 nm, giving an average crystallite size of 16.9 nm. This value lies within the range commonly reported for γ-Al_2O_3 obtained by sol–gel processing at comparable calcination temperatures [4], [6], and confirms that the material remains well within the nanocrystalline regime despite the relatively high calcination temperature.

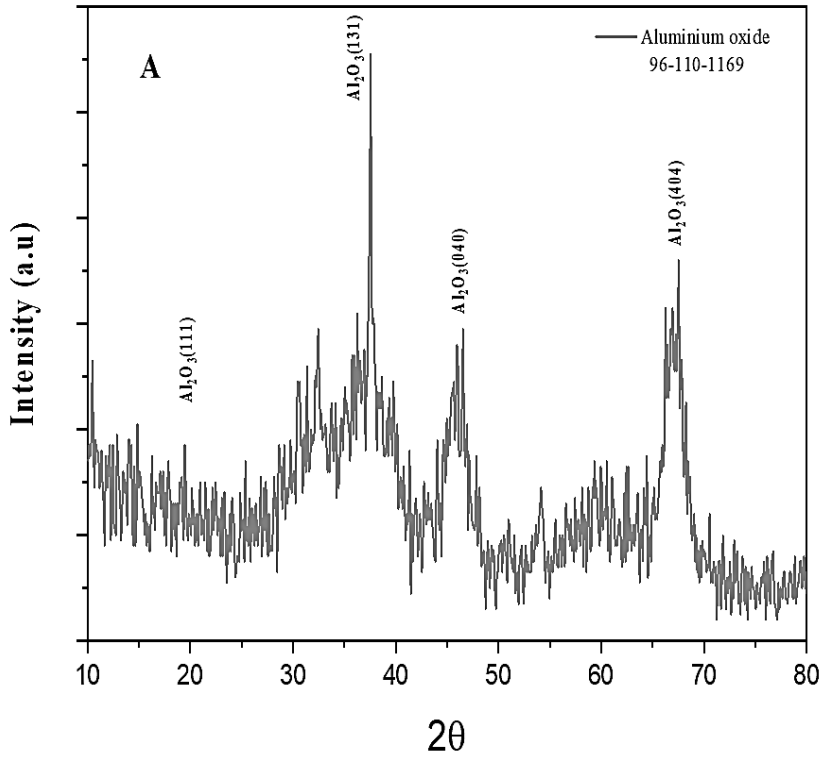


Fig. 1. XRD pattern of the Al_2O_3 powder calcined at 800 °C.

Table 1. Structural parameters of the γ-Al_2O_3 powder obtained from XRD.

2θ (deg.)	37.63	46.46	67.58
(hkl)	(311)	(400)	(440)
d_{hkl} exp. (Å)	2.388	1.952	1.385
d_{hkl} std. (Å)	2.378	1.971	1.394
Crystallite size (nm)	13.263	21.639	17.276
Average crystallite size (nm)	16.9		

3.2. FTIR analysis

The FTIR spectrum of the calcined powder is presented in Figure 2, and the band assignments are summarized in Table 2. The strong absorptions observed in the low-wavenumber region at 455.85, 505.28, and 636.42 cm^{-1} are attributed to the stretching and bending vibrations of Al–O and Al–O–Al bonds in the alumina framework; in particular, the band near 636 cm^{-1} is characteristic of the condensed AlO_6/AlO_4 network of the γ phase [11], [12]. The band at 817.71 cm^{-1} is assigned to the stretching vibration of tetrahedrally coordinated Al–O units, which is a signature of the spinel-type structure of γ-Al_2O_3 and is consistent with the diffraction results [11], [12]. The weaker feature near 1459.92 cm^{-1} arises from residual Al–O surface vibrations, while the bands at 1627.70 and 2925.62 cm^{-1} correspond to the bending and stretching modes of O–H in molecular water adsorbed on the particle surface. The broad absorption centred at 3450.19 cm^{-1} is due to the stretching vibration of Al–O–H surface hydroxyl groups, which are always present on high-surface-area transition aluminas exposed to ambient humidity [11], [14]. The absence of nitrate-related bands around 1380 cm^{-1} indicates that the precursor decomposed completely during calcination.

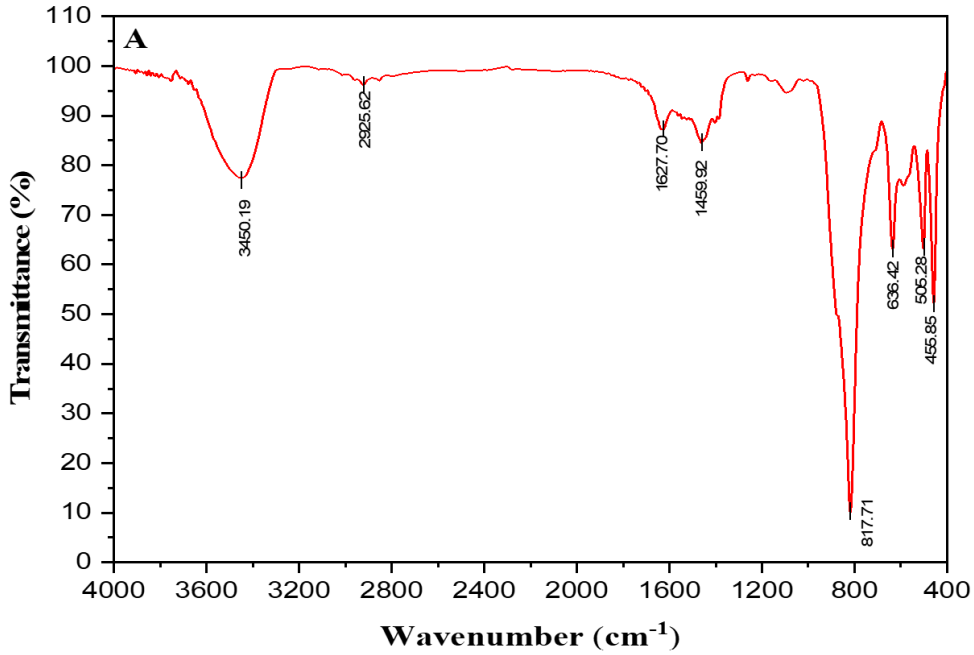


Fig. 2. FTIR spectrum of the Al_2O_3 powder calcined at 800 °C.

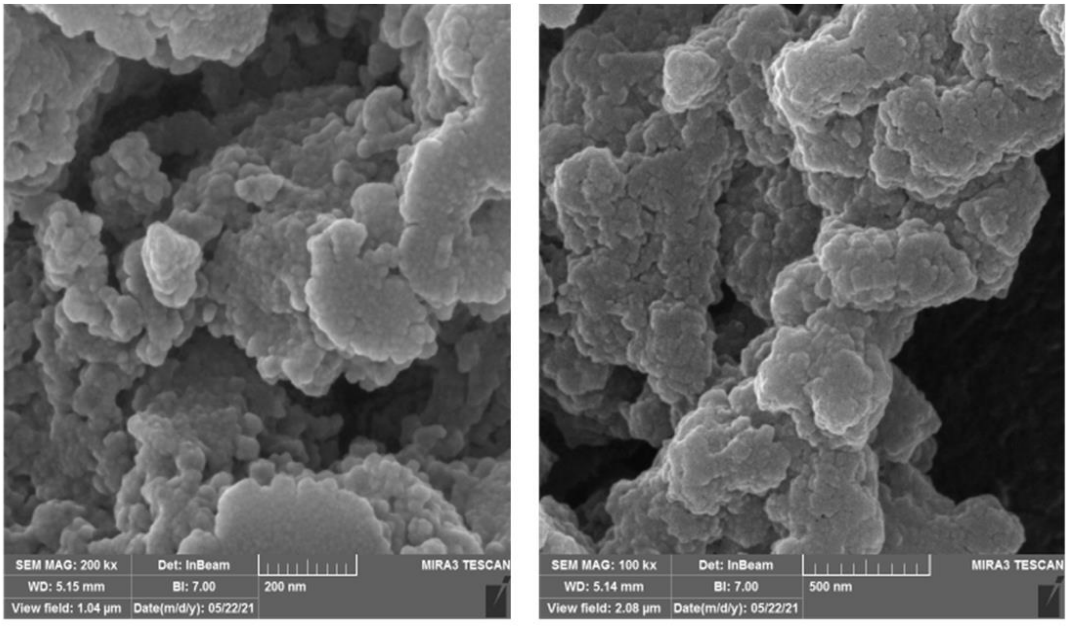
Table 2. FTIR absorption bands of the prepared Al_2O_3 powder and their assignments.

Absorption band (cm^{-1})	Vibration mode	Assignment
455.85	Al–O stretching	Aluminium oxide lattice
505.28	Al–O stretching	Aluminium oxide lattice
636.42	Al–O–Al bending	γ-phase alumina
817.71	Al–O stretching (AlO_4)	Aluminium oxide lattice
1459.92	Al–O vibration	Aluminium oxide
1627.70	O–H bending	Adsorbed water / hydroxyl
2925.62	O–H stretching	Hydroxyl groups
3450.19	Al–O–H stretching	Surface hydroxyl groups

3.3. Morphological analysis

Figure 3 shows a representative FE-SEM micrograph of the calcined powder. The product is composed of small (about 5 nm), semi-spherical particles which have a tendency to form soft agglomerates as seen in alumina prepared using sol-gel technology, due to the large number of hydroxyl groups present on the surface of each individual particle which cause bridging between particles during the drying and calcination processes [4, 5]. Even in this conglomeration the individual particles are well defined and fairly uniform in size.

The particle size was quantified by measuring 60 particles from the micrographs and the distribution was fitted to a Gaussian distribution (Figure 4), a result showing the statistical parameters in Table 3. The particle diameters were between 6.76 and 29.50 nm, and the mean, median and standard deviation were 17.34, 16.25 and 5.09 nm, respectively. The narrow, single-mode distribution is the result of the chemical homogeneity obtained from the solution-based sol-gel process. Notably, the mean particle diameter of 17.34 nm is very close to the mean crystallite size of 16.9 nm calculated from the broadening of the XRD which means that the particles observed by FE-SEM are close to a single crystallite and the agglomerates are composed of monocrystalline primary units. A similar correlation between crystallite size of Scherrer and microscopic particle size has been reported for the nanocrystalline γ-Al2O3 obtained from nitrate precursor [6, 10].



A B

Fig. 3. FE-SEM micrograph of the Al_2O_3 powder

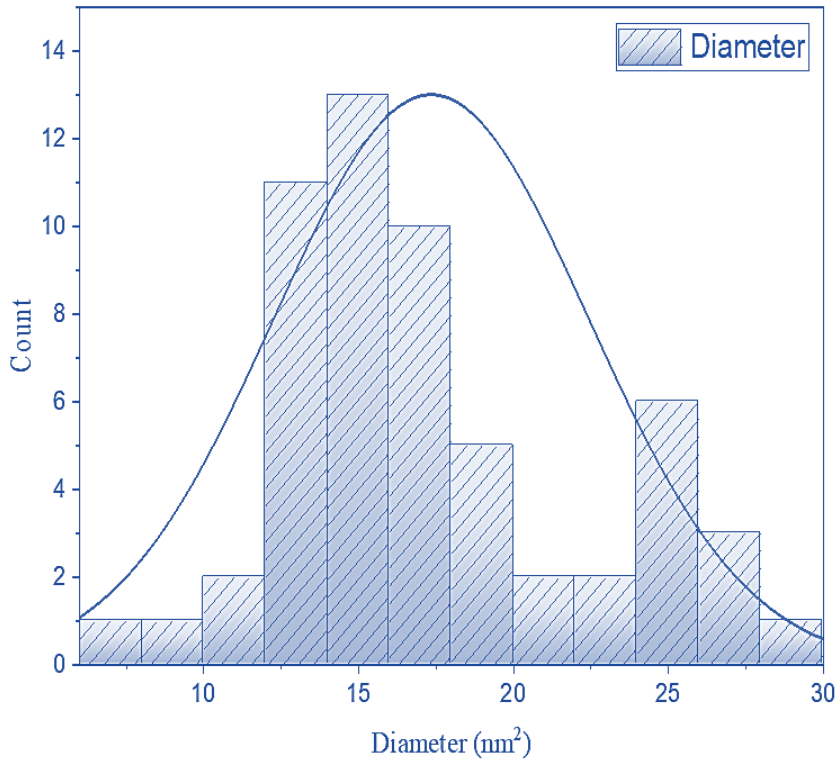


Fig. 4. Particle size histogram of the Al_2O_3 powder fitted with a Gaussian distribution.

Table 3. Statistical parameters of the particle size distribution of the Al_2O_3 powder.

	N (total)	Mean	St. dev.	Sum	Min.	Median	Max.
Diameter (nm)	60	17.341	5.089	988.44	6.757	16.247	29.498

4. Conclusions

Nanocrystalline aluminium oxide was successfully prepared by the sol–gel method from a 1 M aluminium nitrate solution followed by calcination at 800 °C. X-ray diffraction confirmed that the product is single-phase cubic γ-Al_2O_3, with the strongest reflections at 2θ = 37.63°, 46.46°, and 67.58° corresponding to the (311), (400), and (440) planes, and with experimental d-spacings deviating from the standard values by less than about 1%. The average crystallite size calculated from the Scherrer equation was 16.9 nm. FTIR spectroscopy supported the diffraction results, showing the Al–O and Al–O–Al framework vibrations of γ-alumina in the 455–818 cm^{-1} region together with O–H bands associated with surface hydroxyls and adsorbed water, with no evidence of residual nitrate. FE-SEM revealed semi-spherical, partially agglomerated particles whose Gaussian-fitted mean diameter of 17.34 ± 5.09 nm matches the XRD crystallite size, indicating essentially monocrystalline primary particles .

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