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Evaluation of α-Alumina Nanoparticles Prepared by Sol-Gel Method

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Abstract

Nanoparticles show mechanical, electrical, chemical and optical properties that are different and superior to bulk materials. In the present work, α-Alumina nanoparticles were synthesized using the nonorganic Sol-Gel method under controlled conditions. Because of the low cost of its raw materials, low manufacturing temperature and the high purity of the product, Sol-Gel method is the best in the manufacture of nanostructures like metal oxide nanoparticles. The precursor of the Sol-Gel process was aluminum nitrate with ethanol. The prepared nanopowder was evaluated by X-ray diffraction (XRD), scanning electron microscope (SEM), electron dispersive spectroscope (EDS) and Malvern Zetasize analyzer.

Keywords: Nano particles, Sol-Gel method, α-Alumina, XRD, SEM.

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1. Introduction

Alumina is the most important ceramic material due to its excellent properties such as thermal and chemical stability and its high mechanical properties such as strength, hardness and wear [1]. It can be classified into four phases; alpha, beta, gamma, and delta. Each phase reached at a different temperature during the synthesis procedure. alpha alumina is the most popular in applications due to its good properties such as high hardness, stability, and insulation [2].

Nanoparticles differ from micro and macro particles in that they have a larger surface area which leads to use as catalysts and adsorbents [2]. Also, they are used in many ways to improve the component properties such as reinforcement phase or protective coatings [3]. It was observed that when alumina nanoparticles are partially added to the coating, its corrosion resistance will improve [4]. Nanomaterials have been used in many industries such as electrical, mechanical and chemical industries, in addition to advanced technologies, including magnetic materials, conductors, structural and engineering materials [5]. There are many methods of nanomaterials synthesis processes, which can be classified as bottom-up and top-down. Bottom-up methods, such as sol-gel (wet chemical method), are considered the more effective and cost-effective means of synthesis [6]. The Sol-Gel process is a chemical method used for the production of different nanostructures, like metal oxide nanoparticles [7]. The beginning of Sol-Gel process is in the last century, started with Ebelman and Graham who studied of silica gels. Mirjalili et al. [8], investigated the preparation of α-alumina nanoparticles through the Sol-Gel method, utilizing various aqueous solutions of aluminum. The objective was to examine the influence of surfactant stabilizing agents on the aforementioned process. The findings of this investigation indicate that the introduction of surfactant stabilizing agents had a notable impact on both the morphology and size of the resulting nanoparticles, as well as the level of aggregation observed. Rogojan et al. [9], investigated the synthesis of alumina nanopowders by Sol-Gel method using different chemical precursors; inorganic and organic. They studied the powders in terms of crystallinity degree, crystal size, microstructure and morphology. Farahmand and Golabiyan [5], they investigated the production of alumina nanoparticles by the Sol-Gel technique, in which the precursor employed was iron (III) nitrate 9-hydrate. Characterization of surface morphology by scanning electron microscope showed nanosize particles of alumina with minimum agglomeration of sphere shape.

In this work a Sol-Gel process is used to produce α-alumina nanoparticles. The precursor of the process was aluminum nitrate with ethanol.

2. Experimental work

To synthesize α-alumina nanoparticles, 30 g of Aluminum nitrate $Al(NO_3)_3.9H_2O$ were first dissolved in (450 ml) distilled water with magnetic stirring at room temperature. Subsequently, a carefully measured volume of ethanol solution (42 ml) was gradually added drop by drop to the mixture. The temperature of the resulting solution was then elevated to 80°C while stirring was maintained at 1500 rpm. Throughout the synthesis process, the pH was maintained controlled within the range of 2 to 3. These experimental conditions yielded a yellow-colored solution, as shown in Fig.1.

Fig.1 Sol-Gel process.

As the stirring and evaporation process continues for approximately six hours, a white powdery precipitate gradually settled at the bottom of the container. Finally, the obtained white powder was subjected to calcination at a high temperature of 1200 °C in a furnace for a duration of two hours.

The resulting alumina nanopowder was examined and evaluated using X-ray diffraction (XRD), electron dispersive spectroscope (EDS), scanning electron microscope (SEM) and Malvern Zetasize analyzer. Figure 2 shows schematic of steps of Sol-Gel process to synthesis alumina nanoparticles, and Fig. 3 shows alumina nanoparticles prepared by Sol-Gel process.

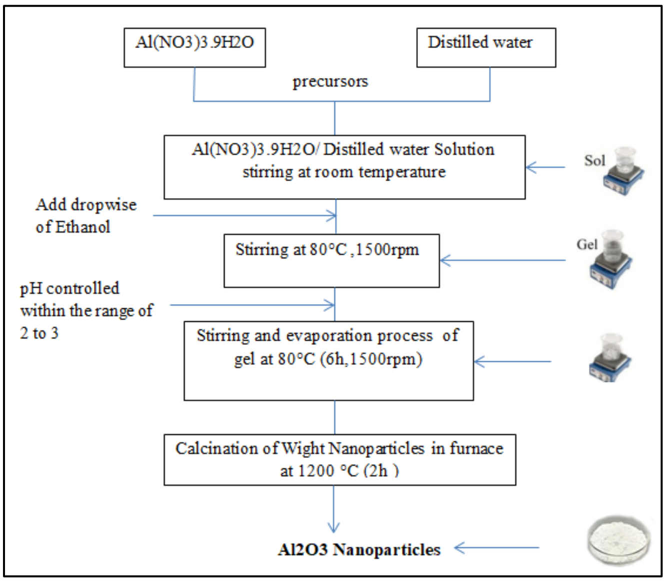

Fig. 2 schematic of steps of Sol-Gel process to synthesis alumina nanoparticles.

Fig. 3 Alumina nanoparticles prepared by Sol-Gel process.

3. Results and discussion

3.1. X-Ray diffraction test

X-ray diffraction (XRD) was used at 40 kV for the purpose of identifying the crystalline phases and estimating the sizes of the crystals. The X-ray diffraction patterns of alumina nanoparticles prepared are shows in Fig. 4. The observed peaks correspond to the (012), (104), (110), (113), (024), (116), (018), (300), and (119) planes, which are indicative of a rhombohedral structure associated with α-Al_2O_3, as established by using the standard data. By Scherrer method in equation (1), the average size of α-alumina nanoparticles was calculated using the FWHM data of each phase after correcting.

$$d = \frac{0.9\lambda}{B\cos\theta} \quad (1)$$

Where, d is the crystallite size, λ is the wavelength of X-ray radiation, θ is the diffraction angle (Bragg angle), and B is the peak width or full width at half maximum (FWHM). The mean crystallite calculated size of the prepared alumina was 31 nm. Figures 5, 6 which achieved by analysis XRD result using X' Pert High Score Plus program. Shows that the main peaks of the prepared alumina correspond exactly to the International Center of Diffraction Data (ICDD) of card number 00–010–0173 of α-Al_2O_3 with a rhombohedral crystal structure of α-alumina [10].

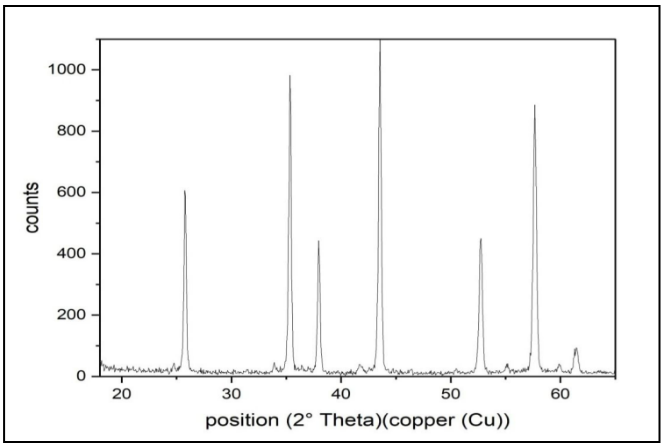

Fig. 4 the XRD pattern of the prepared alumina nanoparticles.

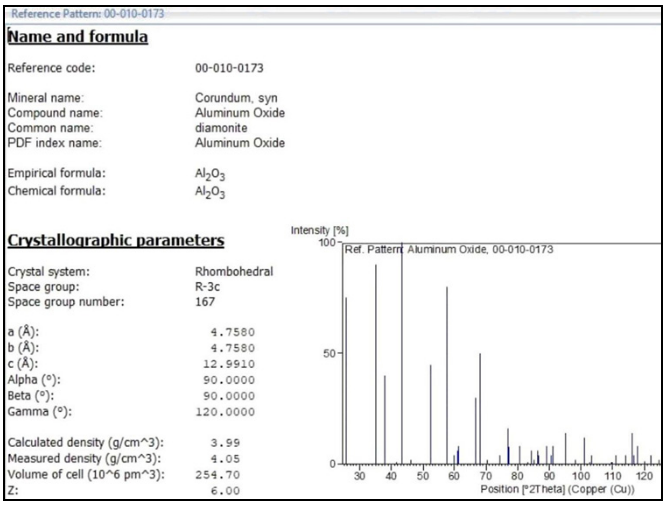

Fig. 5 PDF card no. 00-010-0173.

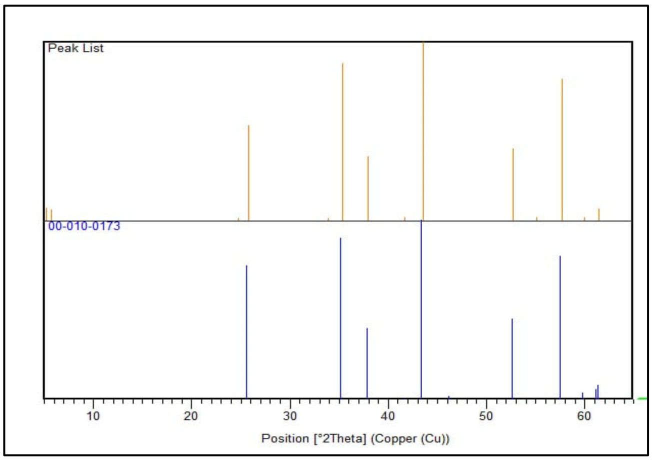

Fig. 6 convergence of PDF card no. 00-010-0173 and the XRD pattern of the prepared alumina nanoparticles.

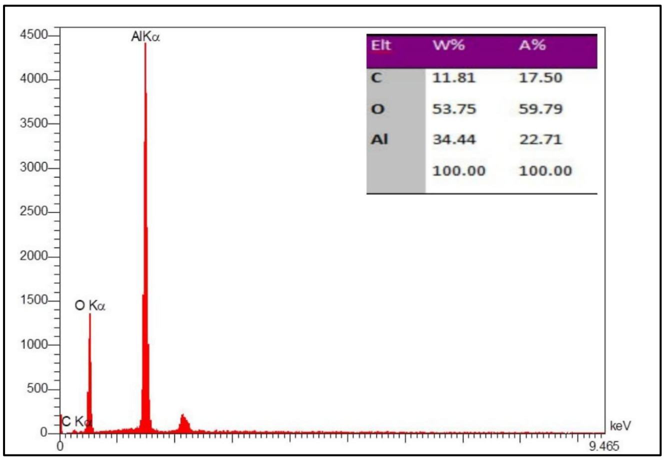

Fig. 7 EDS of the prepared α-Al2O3 nanoparticles.

3.2. Energy dispersive spectroscopy

Figure 7 includes the chemical composition of the prepared alumina nanopowder using the energy dispersive spectroscopy EDS connected to the SEM. The analysis shows that the powder is composed of aluminum and oxygen with a rare percentage of carbon.

3.3. Scanning electron microscopy

Scanning electron microscope of type VEGA3 TSCAN, 20 kV was used to study the morphological characterization of the prepared α-alumina nanopowder. The nanopowder was deposited on an aluminum foil substrate. Figure 8 shows the SEM images of the prepared powder, which show that the nanoparticles are in the form of agglomerations.

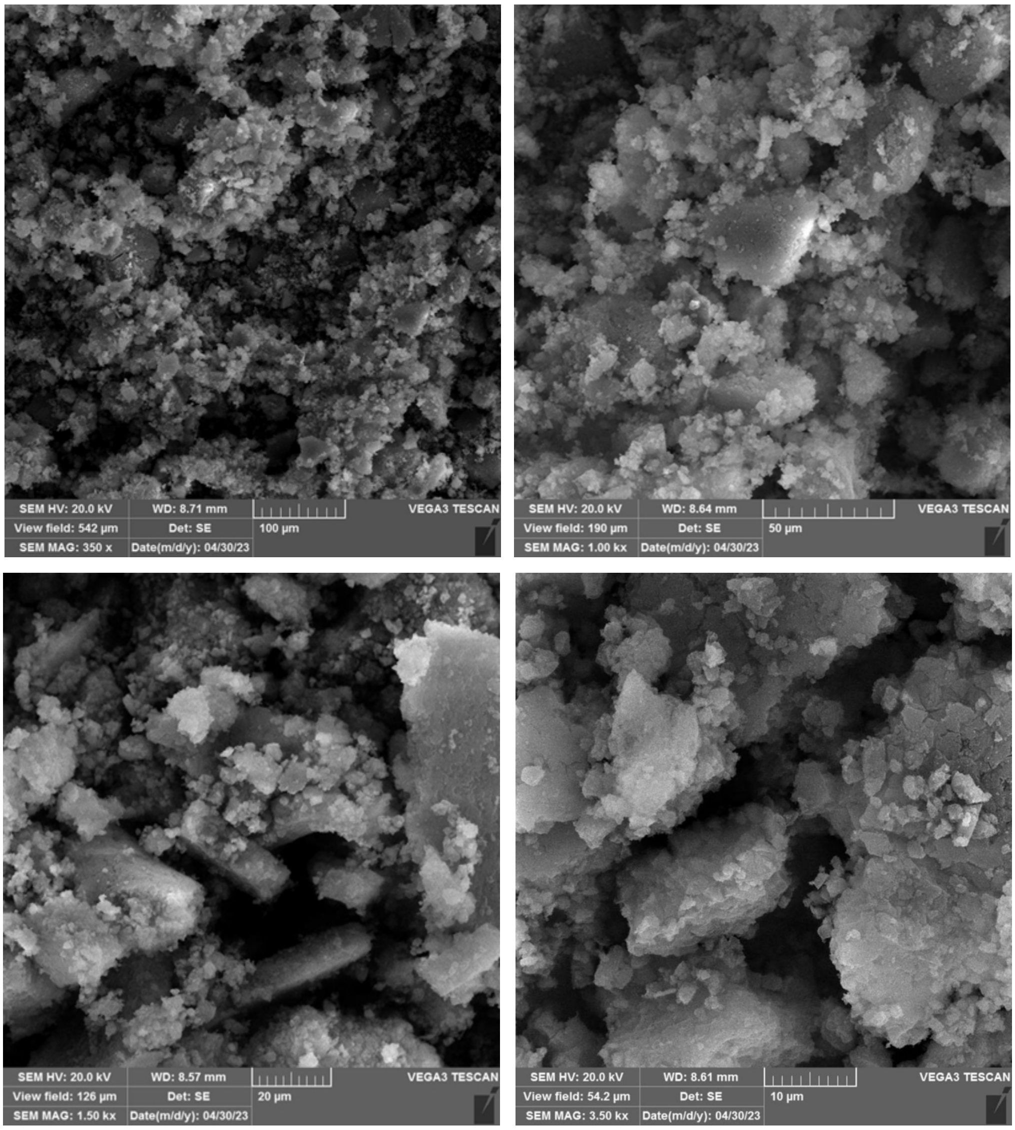

Fig. 8 SEM image of the alumina nanoparticles.

3.4. Particle size analysis

The principle of a Malvern Zetasize analyzer depends on the optical properties of diffraction and scattering phenomenon. There is a beam illuminates the particles dispersed uniformly in the liquid. Figure 9 shows the particle size distribution of the prepared α-alumina powder. It shows that the size distribution is concentrated around 221 nm, which is acceptable as a nanomaterial. By examining the SEM images in Fig. 8, it is clear that this size indicates agglomerations of nanoparticles.

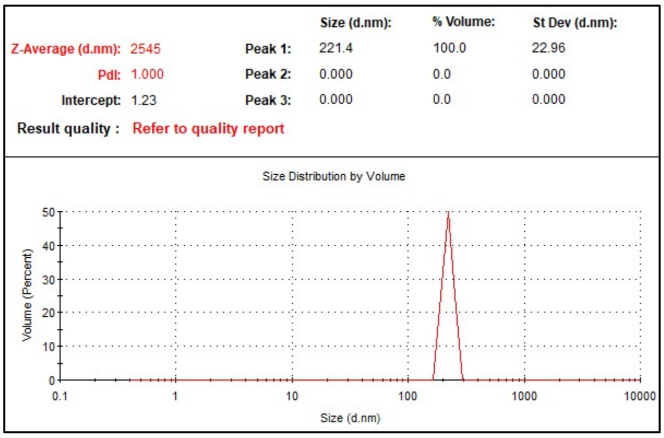

Fig. 9 Size distribution of the prepared Al_2O_3.

5. Conclusions

Alumina nanopowder was successfully synthesized using the inorganic Sol-Gel technique by employing a precursor consisting of an ethanol solution of aluminum nitrate. The X-ray diffraction (XRD) pattern shows a rhombohedral structure of (α-Al_2O_3) alongside an average particle dimension of approximately 31 nm, which was calculated via the Scherrer equation. Moreover, EDS analysis exhibits discernible peaks corresponding to the elements of aluminum (Al) and oxygen (O), as well as a few impurities within the prepared Al_2O_3 powder. Furthermore, the scanning electron microscopy (SEM) images and particle size analysis test show an agglomeration of the nanoparticles.

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