

## **A Simple Method for Synthesis of $\gamma$ -Aluminum Oxide Nanostructure Using PEG as a Neutral Surfactant**

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### **ABSTRACT**

Nano-sized  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> was synthesized successfully by a simple method using Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>·18H<sub>2</sub>O as aluminium source and in the presence of polyethylene glycol (PEG 2000). FTIR spectroscopy, X-ray diffraction (XRD), scanning electron microscope (SEM) and transmission electron microscope (TEM) were used to characterize the structure and morphology of synthesized powder. The results showed that PEG played a significant role in the decomposition of aluminum nitrate to  $\gamma$ -aluminum oxide nanoparticles with a size range of 10-15 nm. The experimental results showed that polyethylene glycol can avoid hard agglomerate during precipitation and that calcination temperature was the major factor to particles size of the final product and it played a significant role in the decomposition of aluminum nitrate to  $\gamma$ -aluminum oxide nanoparticles.

**Keyword:** Nanoparticles;  $\gamma$ -Alumina; Hydrothermal synthesis; Polyethylene glycol; Particles size; X-ray diffraction.

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### **1. INTRODUCTION**

In the past few years, much attention has been focused on the research field of nano-crystalline oxide materials, both because of their fundamental importance and the wide range of potential technological applications [1-6]. Alumina exists in a variety of metastable structures including  $\gamma$ -,  $\eta$ -,  $\delta$ -,  $\theta$ -,  $\kappa$ - and  $\chi$ -alumina, as well as its stable  $\alpha$ -alumina phase. Among these transitions,  $\gamma$ -alumina is one kind of extremely important nanosized materials

[7]. It is extensively used as catalyst, catalytic supports [8, 9] and adsorbents [9-11]. Chemical routes for production of Al<sub>2</sub>O<sub>3</sub> include sol-gel, hydrothermal processing and control precipitation of aluminum salts, alkoxides and metallic powders [12-15]. Polyethylene glycol (PEG) in aqueous solution is a highly mobile molecule with large exclusion volume, and mainly free of charges which can avoid the strong interaction between the

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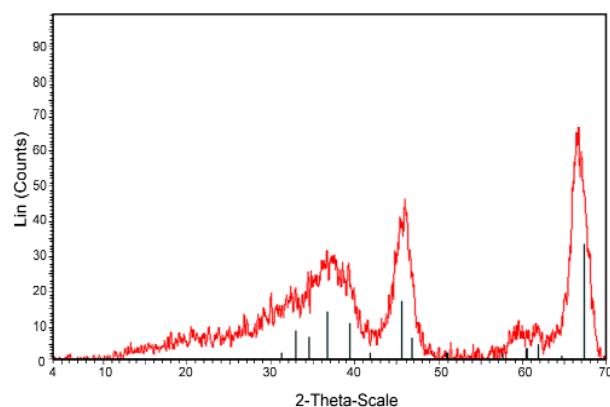
constituents [16, 17]. In continuation of our research on synthesis of metal-oxide in the presence of polyethylene glycol [18, 19], we report a new method for the synthesis of nano-sized aluminum oxide under hydrothermal condition and in the presence of polyethylene glycol in this work. XRD, SEM, TEM and FTIR were used to characterize the product. The experimental results showed that polyethylene glycol can avoid hard agglomerate during precipitation and that calcination temperature was the major factor for particle size of the final product. When the calculation temperatures were controlled between 400-600°C, the agglomerate-free nano-sized  $\gamma$ -alumina particles were obtained.

## 2. EXPERIMENTAL PROCEDURE

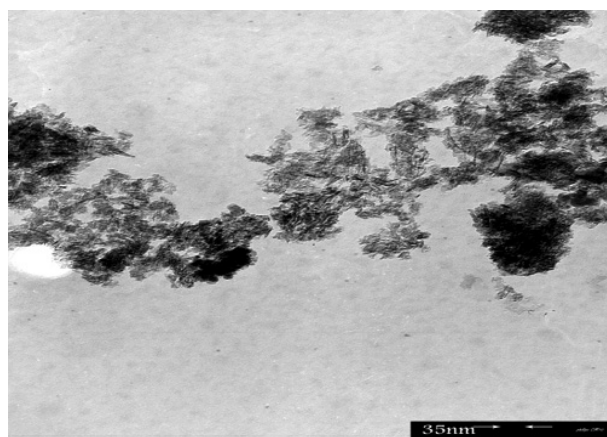
All chemicals are analytical-grade reagents without further purification. Aluminum sulfate pentahydrate,  $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ , (4 g) and polyethylene glycol (5 g) (abbreviated as PEG2000) were dissolved in 100 mL distilled water. An amount of ammonia (25%) was added into the above solution, giving rise to milky precipitates at pH= 9. The reaction mixture was stirred for 1 h at room temperature. Then the reaction mixture was placed in a Parr-Teflon lined stainless steel vessel. It was sealed and heated at 130°C for 48 h. The reaction mixture was gradually cooled to room temperature. The resulting precipitate was filtered and washed three to four times using double distilled water and ethanol to remove polyethylene glycol and other impurities. The synthesized  $\text{Al}_2\text{O}_3$  powder was dried at 100°C for 1 h and was calcinated at 400°C for 2 h. The sample was characterized by powder X-ray diffraction (Bruker, Advance D8) with  $\text{Cu K}\alpha$  ( $\lambda=1.5406 \text{ \AA}$ ) incident radiation. The size distribution and morphology of the sample was analyzed by scanning electron microscopy (SEM, Philips XL30) and Transmission electron microscopy (TEM, Philips CM10). Energy dispersive spectrometry (EDS) attached to SEM was employed to perform the elemental analyses of the nanostructure powder.

## 3. RESULTS AND DISCUSSION

Figure 1 illustrates the XRD patterns of the prepared powders. It indicates the diffraction peaks at  $2\theta$  values are attributed to the formation of  $\gamma$ -alumina and no characteristic peaks from other impurities were detected. The entire d-line patterns match with reported values (JCPDS Card Pattern: 01-1307). It is very clear from Figure 1 that the  $\text{Al}_2\text{O}_3$  precursor particles show nano-size nature as it is seen from line broadening due to the presence of small crystallite sizes.



**Figure 1:** The XRD patterns of synthesized  $\text{Al}_2\text{O}_3$  nano-sized.



**Figure 2:** TEM image of the synthesized  $\text{Al}_2\text{O}_3$  nano-sized.

The crystalline size was estimated from the broadening of  $\text{Al}_2\text{O}_3$  diffraction peak using

Scherrer's formula:

$$t = \frac{k\lambda}{B \cos \theta}$$

Where  $t$  is the average size of the crystallite, assuming that the grains are spherical,  $k$  is 0.9,  $\lambda$  is the wavelength of X-ray radiation,  $B$  is the peak full

width at half maximum (FWHM) and  $\theta$  is the angle of diffraction. The crystalline size of nano-sized  $\text{Al}_2\text{O}_3$  is found to be  $\sim 10$  nm. The TEM micrograph of nano-sized powder along with the electron diffraction pattern is shown in Figure 2. The morphology of the  $\text{Al}_2\text{O}_3$  nano-crystalline was shown in Figure 3.

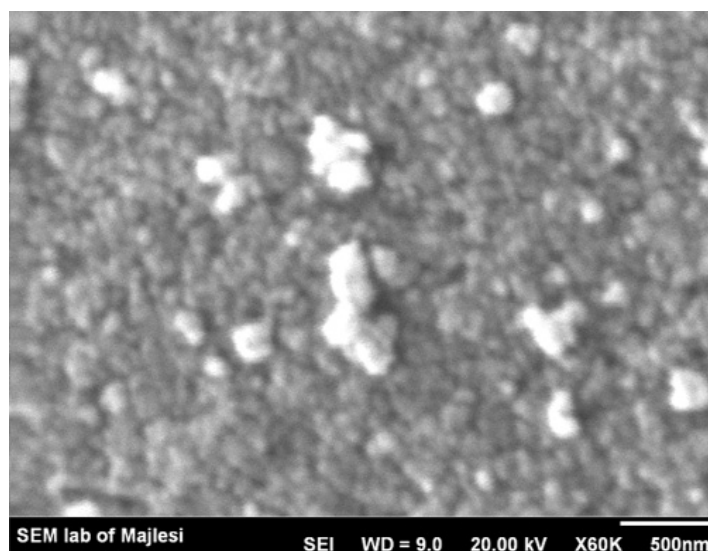


Figure 3: Scanning electron micrographs of synthesized.

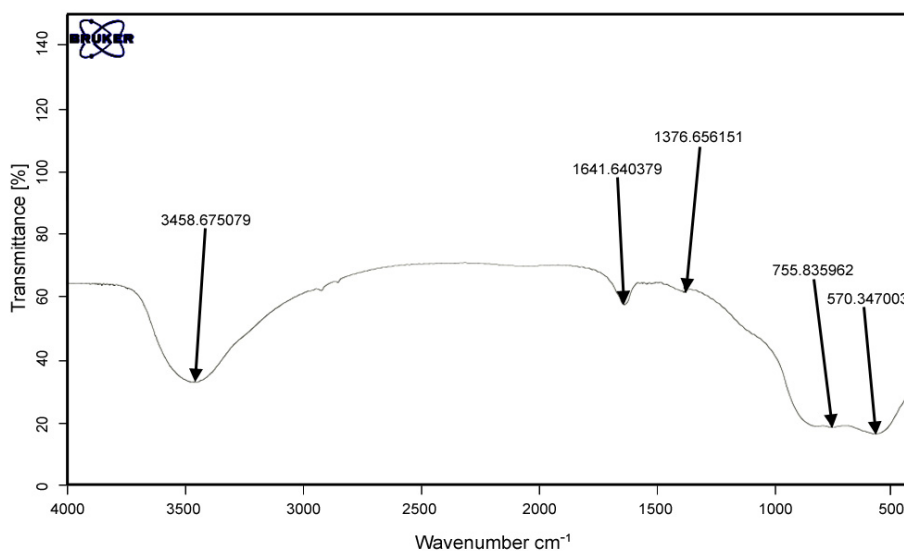


Figure 4: FTIR spectra of  $\gamma\text{-Al}_2\text{O}_3$ .

FTIR spectra (Figure 4) of calcined product shows that a broad band around  $3470\text{ cm}^{-1}$  and  $1650\text{ cm}^{-1}$ , which are assigned to stretching and bending modes of adsorbed water. The bonds in the region of  $500\text{--}750\text{ cm}^{-1}$  and  $800\text{ cm}^{-1}$  are assigned to  $\nu\text{-AlO}_6$  and  $\nu\text{-AlO}_4$  [13].

#### 4. CONCLUTIONS

Nanoparticles of  $\gamma\text{-Al}_2\text{O}_3$  were obtained by hydrothermally synthesis in the presence of poly ethylene glycol as a neutral surfactant. FTIR spectra and XRD pattern show the formation of  $\gamma$ -alumina. The broad XRD line indicates that the  $\gamma\text{-Al}_2\text{O}_3$  particles are of nano size.

#### ACKNOWLEDGMENT

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