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Synthesis and characterization of Alumina (Al_2O_3) nanoparticles prepared by simple sol-gel method

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ABSTRACT: Alumina is one of the most widely used ceramic materials as catalysts, catalyst supports and absorbents, and also wear resistant coating. This study focused on fabricating and characterizing of alumina ceramic nanoparticles fabricated using new and simple sol-gel method. Aluminium oxide (Al_2O_3) nanoparticles were synthesized by iron (III) nitrate 9-hydrate as precursor. Physicochemical properties were done using X-ray diffraction (XRD), high resolution transmission electron microscopy (HRTEM), field effect scanning electron microscopy (FESEM) and electron dispersive spectroscopy (EDS). As there are many forms of transition aluminas, XRD technique was used to identify α-alumina and γ-alumina. The mean particle size of the sphere-like as-prepared sample was around 28 nm estimated by XRD technique and direct TEM observation. The surface morphological studies from SEM depicted grain particles with less agglomeration to sphere-like shape nano-sized Al_2O_3 by increasing annealing temperature. The obtained prticles are spherical and in non-agglomerated state. EDS shows peaks of aluminium and oxygen in prepared Al_2O_3.

Keywords: *Aluminium oxide (Al_2O_3); Chemical synthesis; Nanocrystal; Phase transition; Sol-gel method*

INTRODUCTION

Ultrafine particles are of considerable interest for a wide variety of applications, ranking from catalyst to luminescence ceramics, due to their unique and improved properties primarily determined by size, composition and structure. Nanometer-sized particles hold great potential for use in electronic, chemical or mechanical industries, as well as in relevant technologies, including superconductors, catalysts, magnetic materials, structural and engineering materials. Aluminium oxide (Al_2O_3) nanoparticles have important applications in ceramic industry (Zieliński, *et al.*, 1993, Hench, 2003) and can be used as an brasive material, in heterogeneous catalysis, as an absorbent, and as a biomaterial (Travitzky, *et al.*, 2003, Ganguly, *et al.*, 2003, Martínez, *et al.*, 2003). Alumina is an electrical insulator but has a relatively high conductivity for a ceramic material. Being chemically inert and white, alumina is favored filler for plastics. Also, it is a common ingredient in sunscreen and lots of cosmetics. Alumina is a catalyst itself for converting hydrogen sulfide waste gases into elemental sulfur. Also, alumina is used as a catalyst support for many industrial applications. The most common occurring crystalline form, alpha-

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alumina, makes it suitable for use as an abrasive and as a component in cutting tools. The other phases, gamma-, delta-, eta- and theta- alumina are also existed. There are lots of production method for alumina powders like sol-gel process (Sakka, 1990, Reddy, *et al.*, 2007, Touati, *et al.*, 2000, Bujdák and Rode, 2003, Karim, *et al.*, 2011, Rogojan, *et al.*, 2011, Mirjalilia, *et al.*, 2011), hydrothermal method (Elinaga and Futamura, 2004, Suchanek, 2010), co-precipitation process, mechanical milling, vapor phase reaction, and combustion method (Lang, *et al.*, 2008, Kobayashi, *et al.*, 2005). As the process temperature goes higher the more phase transition to gamma-, delta-, theta-, and alpha-alumina can be occurred. These transitions were accompanied by an abrupt decrease in specific surface area by the sintering process among particles, especially from gamma to alpha phase. Used as a structural material alpha alumina with high strength is needed. Also, the gamma alumina with large specific surface area is suitable for the heat-resisting catalyst support. It is not easy to get nano-sized alpha alumina by the calcination process which accompanied with grain growth of the nano particles. One of the major problems related to the use of alumina catalysts is the deactivation by coke formation and pore plugging which limits the diffusion of substrates and products in and out the catalyst particles. This work describes the synthesis of nanostructured alumina particles by the nonsurfactant templating sol-gel techniques and the structural and morphological characterization of these nanoparticles are done by using XRD, HRTEM, FESEM and EDS analyses.

EXPERIMENTAL DETAIS

Al_2O_3 nanoparticles were synthesized by ethanol solution of aluminium nitrate Firstly, 10 g $Al(NO_3)_3.9H_2O$ was completely dissolved in 150 mL pure water with stirring at room temperature. 14 mL of ethanol solution was then added drop by drop to the solution and synthesis temperature was increased to 80°C. The color of solution changed from orange color to dark brown color. The pH was maintained between 2 and 3 during the synthesis. The white product was evaporated for 3 hours, cooled to room temperature and finally calcined at 500°C for 5 hours. All analyses were done for samples without any washing and more purification. The specification of the size, structure and optical properties of the as-synthesis and annealed Al_2O_3 nanoparticles were carried out. X-ray diffractometer (XRD) was used to identify the crystalline phase and to estimate the crystalline size. The XRD pattern was recorded with 2θ in the range of 4-85° with type X-Pert Pro MPD, Cu-K_α: λ = 1.54 Å. The morphology was characterized by field emission scanning electron microscopy (SEM) with type KYKY-EM3200, 25 kV and transmission electron microscopy (TEM) with type Zeiss EM-900, 80 kV. The Al and O elemental analysis of the samples was performed by energy dispersive spectroscopy (EDS) type VEGA, 15 kV. All the measurements were carried out at room temperature.

RESULTS AND DISCUSSION

X-ray diffraction (XRD) at 40Kv was used to identify crystalline phases and to estimate the crystalline sizes. Figure 1 shows the X-ray diffraction patterns of the powder before and after heat treatment. Fig. 1(a) shows the XRD pattern of aluminium oxide before annealing. A γ →α-Al_2O_3 phase transformation took place in calcination more than 1000°C. Fig. 1(b) shows the XRD pattern of aluminium oxide at1000°C. As you can see, the broad γ picks were appeared with increasing temperature. α- Al_2O_3 was the only phase present for the powder calcined above 1000°C. The exhibited picks correspond to the (012), (104), (110), (113), (024), (116), (018), (300) and (119) of a rhombohedral structure of α- Al_2O_3 is identified using the standard data. The mean size of the ordered Al_2O_3 nanoparticles has been estimated from full width at half maximum (FWHM) and Debye-Sherrer formula according to the equation as follows:

$$D = \frac{0.89\lambda}{B\cos\theta} \quad (1)$$

where, 0.89 is the shape factor, λ is the x-ray wavelength, B is the line broadening at half the maximum intensity (FWHM) in radians, and θ is the Bragg

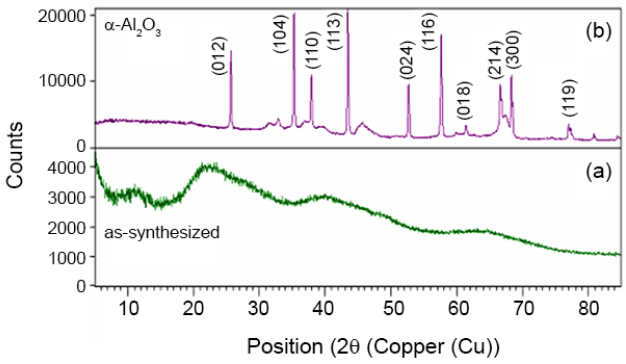
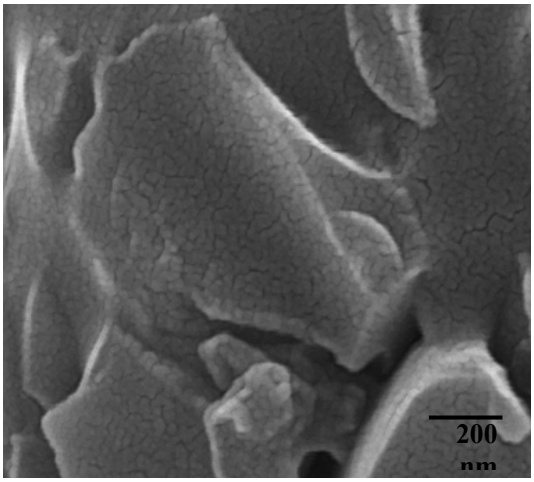


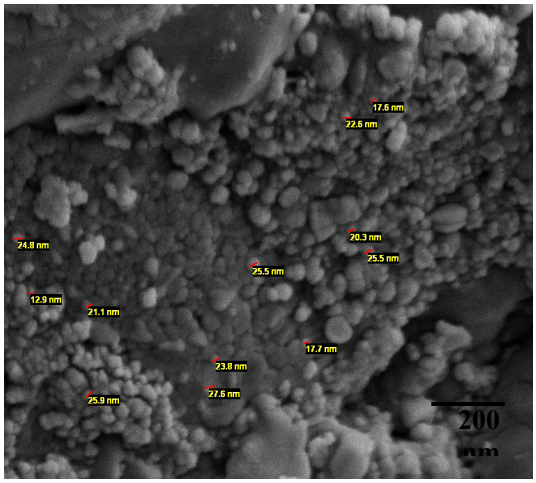
Fig. 1. XRD pattern of Al_2O_3 nanocrystals, (a) as-synthesized and (b) annealed at 1000°C

angle. The mean size of as-prepared Al_2O_3 nanoparticles was around 28 nm from this Debye-Sherrer equation.

SEM analysis was used for the morphological study of nanoparticles of Al_2O_3 samples. These analyses



(a)



(b)

Fig. 2. SEM images of the (a) as-prepared (b) annealed Al_2O_3 nanoparticles at 500°C.

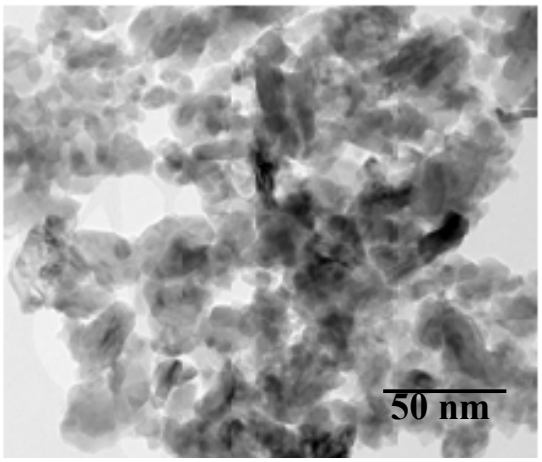


Fig. 3. TEM image of the as-prepared Al_2O_3 nanoparticles

show high homogeneity emerged in the surface by increasing annealing temperature. By increasing annealing temperature the morphology of the particles changes to the sphere-like shape with the smallest diameter of 21 nm. Fig. 2(a) shows the SEM image of the as-prepared Al_2O_3 nanoparticles with less agglomeration. Fig. 2(b) shows the SEM image of the annealed sphere-like shape nanoparticls Al_2O_3 at 500°C for 5 hours.

TEM analysis was carried out to confirm the actual size of the particles, their growth pattern and the distribution of the crystallites. Fig. 3 shows the as-synthesized TEM image of Al_2O_3 nanoparticles with less agglomeration prepared by sol-gel route. The mean particle size of nanoparticles was determined about 28 nm.

Energy dispersive spectroscopy (EDS) of Al_2O_3 prepared by wet synthesis is shown in Fig. 4 which confirms the existence of Al and O with weight percent. EDS was used to analyze the chemical composition of a material under SEM. EDS shows peaks of alu-

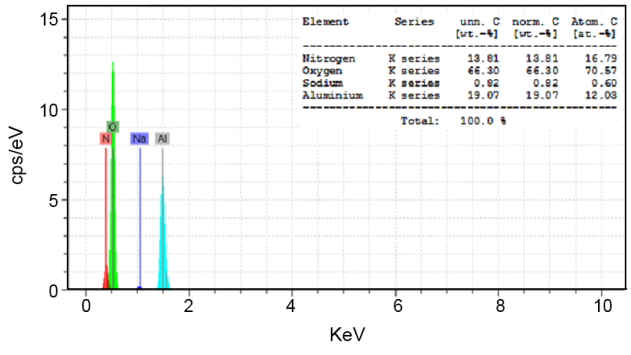


Fig. 4. EDS spectra of the as-synthesized Al_2O_3.

minium and oxygen and indicates fewer impurities in prepared Al_2O_3.

CONCLUSIONS

Novel α-Al_2O_3 nanoparticles ceramic were successfully synthesized by the nonsurfactant templating sol-gel techniques using an ethanol solution of aluminium nitrate. XRD spectrum shows rhombohedral structure of α-Al_2O_3 annealed at 1000°C. From SEM images, it is clear that with increasing temperature, the morphology of the particles changes to sphere-like shaped. TEM image exhibits that the uniform as-synthesized Al_2O_3 nanoparticles prepared by sol-gel route with an average diameter about 28 nm. EDS shows peaks of aluminium and oxygen and indicates fewer impurities in prepared Al_2O_3.

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