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Synthesis and characterization of sub-micron alumina by combustion of aluminum complexes with acetylacetone and salicylaldehyde

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Abstract

Sub-micron. alumina powders were synthesized by the combustion method using aluminum complexes of acetylacetone and salcylaldehyde. In this study, the powders were synthesized by solution combustion without the fuel. The phase transition and particle characteristics of the Al_2O_3 were determined by examining the crystalline structure before and after the heat treatment were investigated by Ft-IR and FESEM technique.

Keywords: Sub-micron alumina, Solution combustion synthesis, sub-micron aluminua complex.

1. Introduction

In recent years, increasing attention has been focused on the development of nano-crystalline Al_2O_3 powders. Because of low cost, fine particles size, high specific surface area and high activity of the surface [1-3]. Alumina is one of the most widely used ceramic materials as catalysts, catalyst supports and absorbents, and also wear resistant coating [4], [5]. Alumina exists in a number of metastable transition phases as well as the thermodynamically stable α-Al_2O_3 or corundum. These metastable alumina structures can be divided into two broad categories: a face-centered cubic arrangement of oxygen anions including γ-, η-, θ-, and δ-alumina, and a hexagonal close-packed arrangement of oxygen anions consisting of α-, κ-, and X-alumina [6-8]. Common methods to prepare γ-alumina nano powders are mechanical synthesis [9], vapor phase reaction [10], precipitation [11], combustion [12], and sol-gel [13] methods. Solution Combustion Synthesis (SCS) provides simple, low cost fast process, with energy and time saving to produce pure nano crystalline ceramic powders [14]. Solution combustion synthesis (SCS) is an effective method for the synthesis of nano scale materials and has been used in the production of various ceramic powders for a variety of advanced applications .Solution combustion synthesis makes use of salts such as nitrates , metal sulfates and carbonates , as oxidants and reducing reagents , fuels such as glycine, sucrose , urea , or other water soluble carbohydrates. Sulfate acts as an oxidizer for the fuel during the combustion reaction [15].

2. Experimental:

2.1. Synthesis of Sample 1:

8 ml of NH_3 (5M) was added to a stirred solution of salicylaldehyde (0.12 g, 12.5 mmol) in distilled water (10 ml). To this mixture was added a solution of $Al_2(SO_4)_3.16H_2O$ (0.092, 0.5 mmol) in distilled water (10 ml). The mixture was stirred for 30 min. After complete addition of Salicylaldehyde solution, pH value of the solution was kept basic using ammonia solution for 15 minutes. The product was filtered, washed with 100 ml of distilled water and dried by suction for a few minutes at 110°C (Sample 1).

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2.2. Synthesis of Sample 2:

This sample was prepared by the same method as for sample **1**, except that acetylacetone was used instead of Salicylaldehyde and a solid product was obtained after one week.

2.3. Synthesis of sub-micron alumina:

The requisite amount of the synthesized complexes of **1** or **2** (2g, mmol) has been mixed in minimum amount of deionized water. The resultant sol. was continuously stirred for several hours. Then the dish containing prepared precursor was heat treated at 600°C for 3 h, and remained in the furnace until the product was cooled to room temperature naturally after the combustion. First thermal dehydration was occurred until the solution was became free from water to form final product. The product was formed after solution combustion process was grounded thoroughly to form a homogeneous powder, without agglomerated particles. The obtained powder was kept for FT-IR, XRD & FE-SEM analysis. The phase identification of calcined powders was recorded by X-ray diffractometer (Philips X'pert MPD 3040) with Cu Kα radiation. The Fourier transform infrared spectra (FTIR) were measured on a Shimadzu FTIR spectrophotometer.using the KBr pellet method.

3. Results and discussion:

3-1. X-ray diffraction and FTIR analysis:

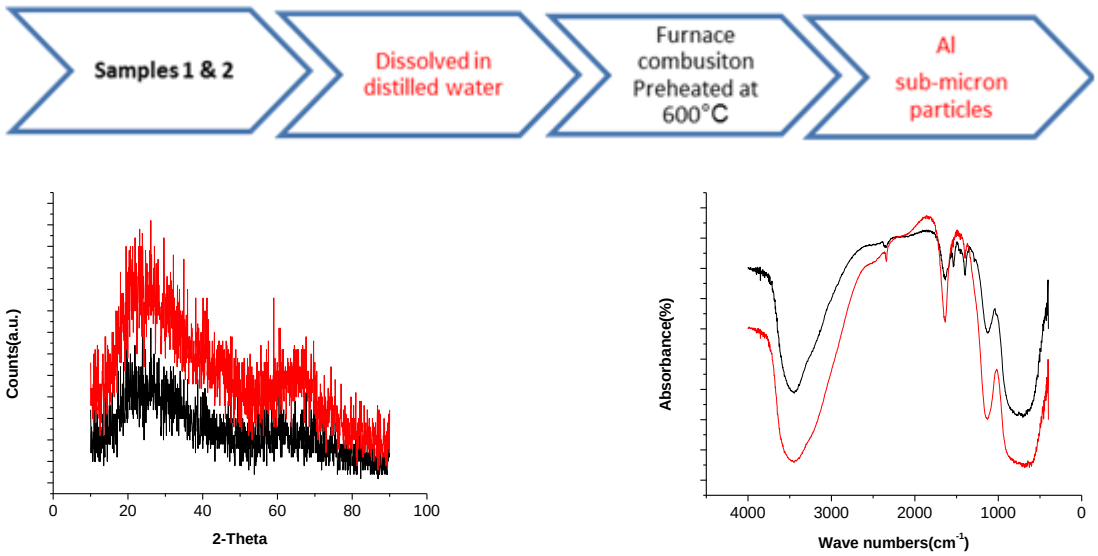

Fig.1. XRD spectrum of sample **1** and **2** at 600 ° C for 3 hours. Red (S_1) and black (S_2).

Fig.2. FT-IR spectrum of the samples **1** and **2** at 600 ° C for 3 hours. a) S_1 and b) S_2.

Phase in both samples based on the standard models are the γ and δ phases (pdf number 421468 and 461131). However, the interplanar spacing of the synthesized materials are calculated by Bragg's equation. According to the pattern, there is a left/right shift in the 2theta value: $\Delta 2\Theta = 24.5(S_2) - 24.43(S_1) = 0.07°$. So, according to the calculation, there is an expansion/contraction in the unit cell while increasing the heat treatment: $\Delta d = 7.29(S_1) - 7.26(S_2) = 0.03$ Å that is in a good agreement with SEM images.

The FTIR spectra of all samples are also recorded after heating at 600 °C. This clearly shows a broad absorption at around 3400 cm^{-1} and a small absorption at 1635 cm^{-1}, which are characteristic stretching vibration and deformation vibration of hydroxyl group (O-H), respectively. Peak localized at 1623 cm^{-1} are assigned to asymmetrical stretching vibration of carboxyl ions (COO-) and the broad absorption at around 400-900 cm^{-1}, which are characteristic stretching vibration and deformation vibration of (Al-O), respectively. Peak at around 1398 cm^{-1} is based on the vibration of the gelatinous boehmite which is converted to the poorly crystalline of AlOOH. The peak intensity increases indicating a delay in the transition phase of the gelatinous boehmite to gamma leading to the production of smaller nanoparticles [16-18].

3.2. Analysis (SEM):

Figure 3 shows the FESEM images of the synthesized materials (S_1). This figure shows the synthesized material has a porous structure with the clear porosity in a sphere shape in a size of about 600 nm. So the structure of the material is macro-porous.

While figure 4 shows the S_2 synthesized images shows that still the structure of the material is porous however the shape of the holes is different from S_1. Figure 4 shows that the holes have significant structure in a rectangular shape with the size in about 100 and 700 nm.

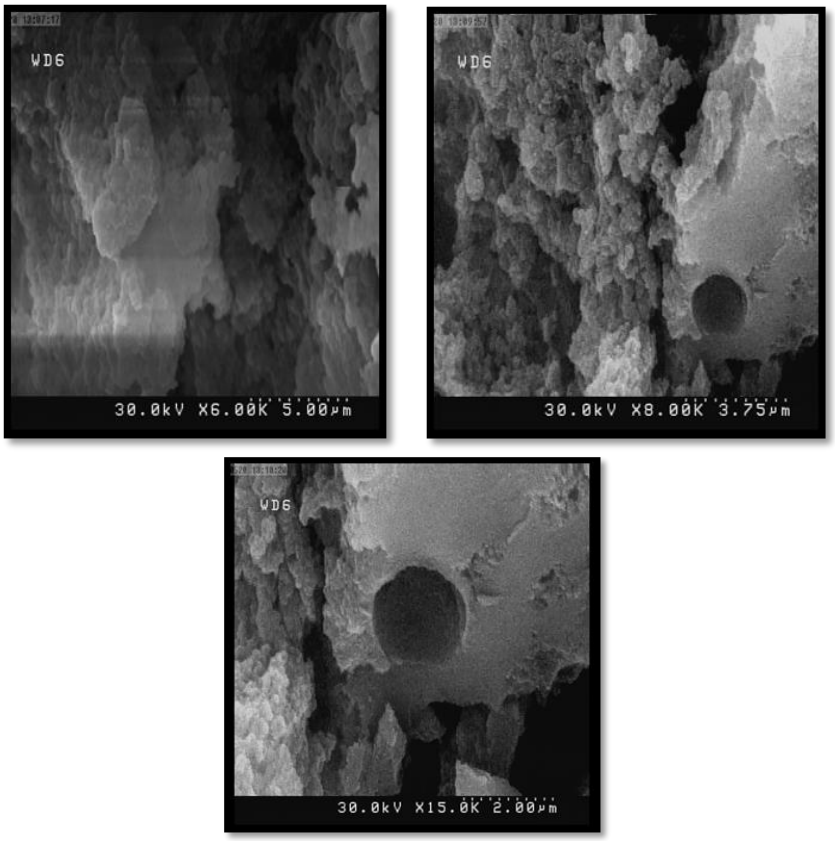

Fig.3. SEM photograph at 600 ° C for 3 hours Sample 1.

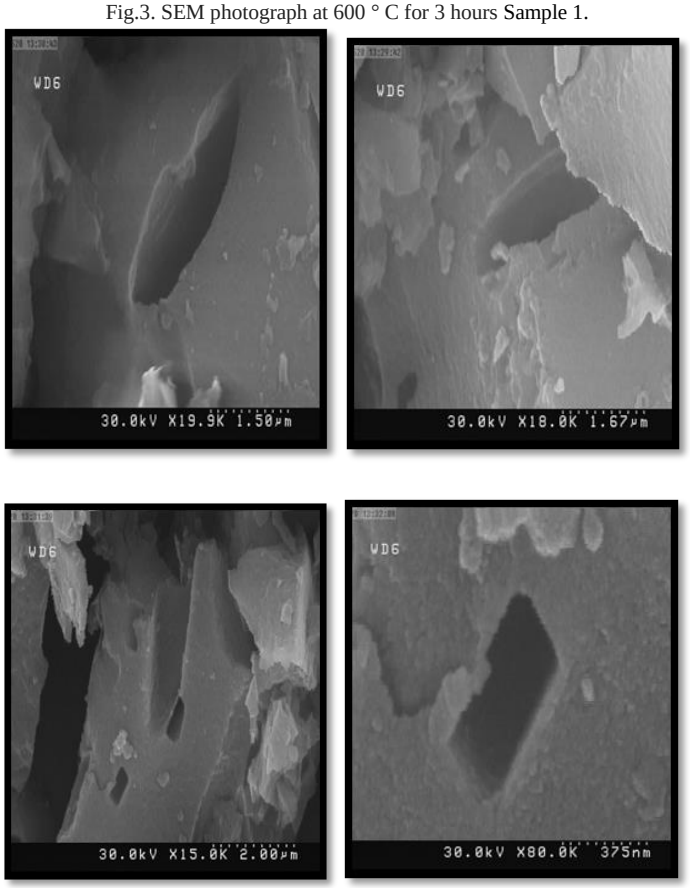

Fig.4. SEM photograph at 600 ° C for 3 hours Sample **2**.

Figure 5 shows the UV-Vis spectra of the synthesized materials. The figure shows that the both samples have the same absorption wavelength in about 230 nm. The absorption edge of the both samples, is 250 nm so the band gap is 4.96 eV that is in a good agreement with references. The UV-Vis spectra of the obtained materials are in a good agreement with the reference [19].

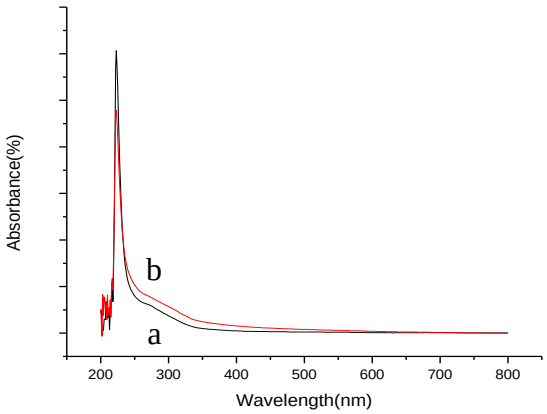

Fig. 5. UV-Visible spectrum of the samples **1** and **2** at 600 ° C for 3 hours. a) S_1 and b) S_2.

4. Conclusion:

In this work, the sub-micron-Al_2O_3 powder are produced by a combustion method using complexes as reagents. The XRD pattern of the submicron alumina showed its typical peaks related with its crystalline form. It is evident from the FESEM images that the synthesized materials are porous like while the porosity of the two samples are different. Also, we used FTIR and UV-Vis analysis to investigate the optical properties oh the synthesized materials. UV-Vis spectroscopy showed that the band gap for the both samples was 4.96 eV.

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