Synthesis and Characterization of α-Al_2O_3 Platelet Nanoparticle as a Direct Product of Solution Combustion Method

Hamed Sadabadi, Adeleh Aftabtalab, Shirzad Zafarian, K.Venkateswara. Rao, V.Rajendar

Abstract*—Crystalline α-Al_2O_3 was synthesized by the solution combustion of aluminum nitrate and urea in aqueous media. The effect of conditions was further investigated in detail. The XRD analysis shows that the crystalline α-Al_2O_3 was obtained directly by SC method without annealing at high temperature above 1100°C, and the crystalline size can estimate from XRD result. The SEM micrograph shows the morphology of particles. Thermal behavior of α-Al_2O_3 was evaluated by applying Thermal gravimetric and differential thermal analysis (TG/DTA).

***Keywords:* α-Al_2O_3, Solution combustion, nanostructure, XRD**

I. INTRODUCTION

In practical applications and technologies, the un-agglomerated particles with sharp size distribution, is preferred especially for compacting or self-arranging the particles. A number of techniques such as: chemical reduction, sol-gel, reversed micelle, hot-soap, pyrolysis, and spray pyrolysis methods, for the preparations of nanoparticles that satisfy this requirement have been developed [1]. All these techniques require special chemicals and equipment [2]. To be industrially relevant, the process needs to be low-cost, continuous operation and high production rate. Solution Combustion synthesis(CS) or self-propagating high-temperature synthesis(SHS) is a time conserve, effective, and low-cost method for production of various industrially metal oxides. Corundum, α–Al_2O_3 platelet powder is one of the most important ceramic material with significant properties, such as: excellent mechanical strength at room temperature and high temperature, hardness and abrasion resistance, high melting point, chemical inertness, thermal shock resistance, and so on [3,4,9]. Such unique properties made α–Al_2O_3 one of the most commonly used materials for wide applications, such as electronics, metallurgy, optoelectronics, catalysts, [5] and fine ceramic composites reinforcement fillers in plastics for thermal conductivity enhancement. Many methods have been used to prepare α-Al_2O_3 platelet powder, including heating a mixture of Al_2O_3 and aluminum fluoride, growth in an HF–γ-Al_2O_3 system, synthesis in a molten Na_2SO_4 flux, K_2SO_4 flux, synthesis in a 1,4-butanedial solution by seeding, synthesis using electrostatic spray-assisted chemical vapor deposition, and synthesis using the flux method in a microwave field [5] and directly by CS method [4]. Boehmite undergoes dehydration at 500°C to form γ-Al_2O_3, which transforms to δ-Al_2O_3 and then to θ-Al_2O_3 before phase transformation to α-Al_2O_3. The θ to α-phase transformation of alumina is achieved by a nucleation and growth process [7]. α-Al_2O_3 obtains only after annealing above 1100°C [4-10].

Manuscript Received on February. 2013.

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II. EXPERIMENTAL

A. Materials

Aluminum nitrate nonahydrate extra pure purchased from E. merck (india) limited Co. Urea extra pure purchased from Thomas baker (chemicals) PVT limited Co (India).

B. Sample preparation

Nano-size α-Al_2O_3 was synthesized by the combustion of water solutions containing stoichiometric amounts of the aluminum nitrate and Urea, was calculated based on the total oxidizing to reducing valencies keeping the O/F ratio unity. To prepare the samples, 15.00 gm aluminum nitrate dissolve in 20, 10, and 5ml distilled water bearing sample number U20, U10, and U5 respectively. The 6.01 gm Urea added to solutions by stirring. Beaker then placed on hot plate to boiling, foaming, and flaming of solutions. Les amount of distilled water proportional to redox cause to procedure occur at short time and α-Al_2O_3 was prepared. The following formula shows the calculation of amount ratio of aluminum nitrate to Urea. All samples calcinated at 450°C for just 1hr without further heat treatment.

$2Al(NO_3)_3$ (aq)+ $5CH_4N_2O$(aq) ⟶ Al_2O_3+ $10H_2O$+ $5CO_2$+$8N_2$

4*375.13 (M_w) = 1500.52 gm Aluminum nitrate; 15.005 gm

10*60.06 (Mw) = 600.6 gm Urea; 6.006 gm

Urea as fuel

III. RESULT AND DISCUSSION

XRD patterns were recorded by a D8 Advance (Bruker,) X-ray diffractometer with Cu Kα irradiation by wavelength 1.54058 Å (40 kV, 40 mA) after calcinations of particles at 450°C. The morphology of the products were observed by SEM using a S-3400N (Hitachi High-Technologies, Japan) microscope, Thermal gravimetric analysis and differential thermal analysis (TG/DTA) has been performed in air at heating rate of 30°C/min, using thermal analyzer model XSTAR6000 (ultimac).

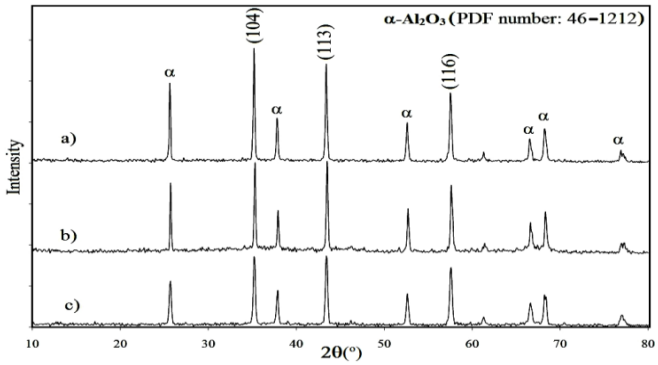
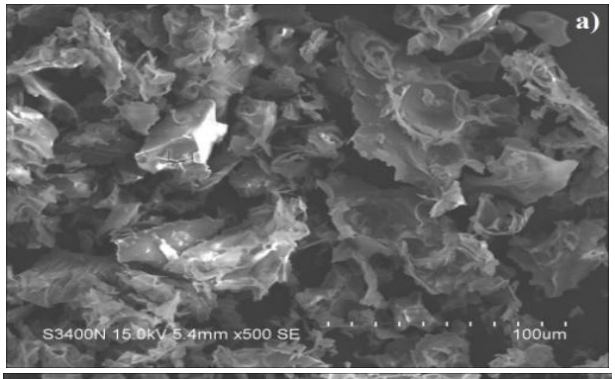


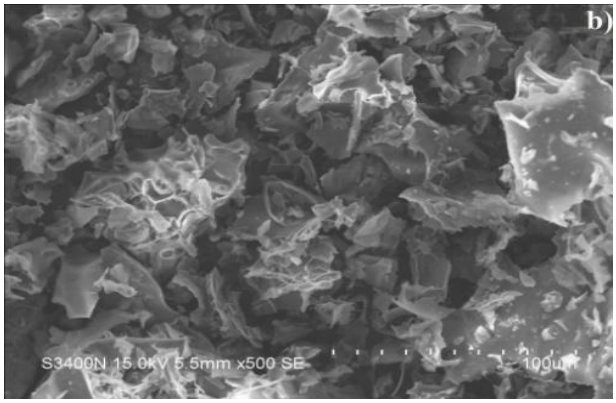
Fig.1. XRD pattern of α-Al_2O_3 prepared by solution combustion method of aluminum nitrate and Urea in (a) 20 ml, b) 10 ml, and (c) 5 ml distilled water.

XRD patterns of α-Al_2O_3 with different amount of water are shown in Fig 1. XRD result indicates that the α-Al_2O_3 obtains without any annealing process by using of Urea as fuel in solution combustion synthesis (SCS). The average crystal size was estimated by applying the debye-Scherrer equation to the apparent Full Width at Half Maximum intensity (FWHM) of (104), (113), and (116) peaks of α-Al_2O_3. The crystalline size of alumina has been changed by accelerating of process (decreasing in water amount) and size increases by decreasing in water.

Table.1. Crystalline size calculated by scherer equation.

Sample name	Water amount ml	Fuel	Size by **debye**-scherer equation nm
U20	20	Urea	29
U10	10	Urea	33.7
U5	5	Urea	33.8





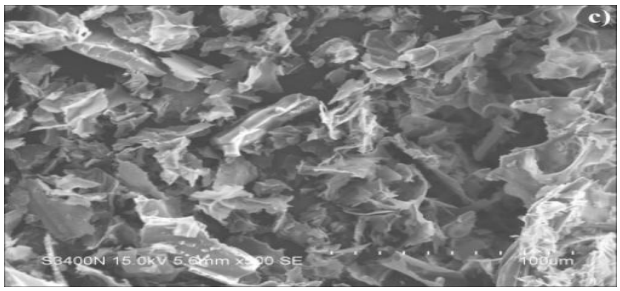


Fig.2. SEM micrograph of α-Al_2O_3 prepared by solution combustion method of aluminum nitrate and Urea in (a) 20 ml, b) 10 ml, and (c) 5 ml distilled water.

According to the SEM micrograph of the particles produced at different water amount (Fig.2.) it was determined that particle shape is platelet and constant by changing in the water amount and accelerating the process.

The DTA thermograph (Fig.3) indicates that α-Al_2O_3 decomposes exothermally up to 294.6°C and after 640.9°C, and between 294.6 to 640.9°C shows endothermic behavior. The soft slope of graph without any peaks before 800°C shows, there is no residual boehmite and γ-Al_2O_3 by this method. TG thermograph (Fig.4.) shows the sharp slope below 200°C, due to water evaporation, and above that, up to 800°C, slope mollified that indicate decomposition of urea and aluminum nitrate traces in the samples. The weight loss was 2.5% for samples (a) and (b), while the sample (c) shows 5% weight loss.

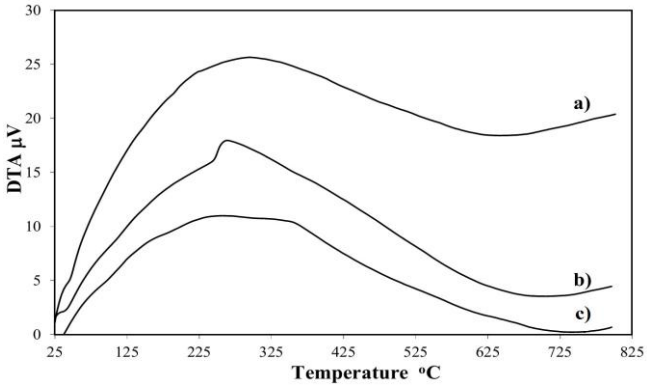


Fig.3. DTA graph of α-Al_2O_3 synthesized by solution combustion (a) in 20 ml,(b) in 10 ml, and (c) in 5 ml distilled water.

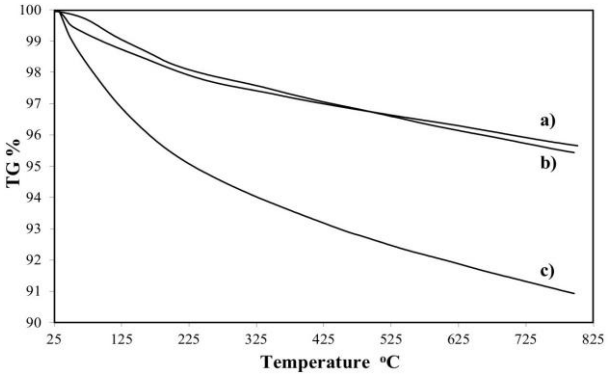


Fig.4. TGA graph of α-Al_2O_3 synthesized by solution combustion (a) in 20 ml, (b) in 5 ml, and (c) in 10 ml distilled water.

IV. CONCLUSION

Crystalline α-Al_2O_3 platelets were synthesized by heating, boiling, foaming, and flaming of a mixture of aluminum nitrate and urea in aqueous media. α-Al_2O_3 obtained directly as product of combustion and change in amount of water can change the size, but the morphology remains constant. TG/DTA thermograph the exist of single phase in all products of combustion method.

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