High purity Alpha Alumina nanoparticle: Synthesis and characterization

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Abstract - High purity crystalline alpha alumina (α-Al_2O_3), platelet powder, synthesized by the combustion synthesis, aluminum nitrate was used as the source of aluminum and urea as oxidizer in an aqueous medium. X-Ray diffractometer applied to study crystalline phase. Scanning Electron Microscopy (SEM) and Energy dispersive X-ray spectroscopy analysis (EDAX) was used for morphological and chemical characterization of nanoparticel. Further study on crystalline structure of α-$Al_2O_3$was done by applying Transmission Electron Microscopy (TEM) and Selected Area Electron Diffraction (SAED). Size distribution of powder investigated using Particle Size Analyzer (PSA). Thermal gravimetric and differential thermal analysis(TG/DTA)evaluated thermal behavior of α-Al_2O_3.

Index Terms: α-Al_2O_3, combustion synthesis, High purity, Nanostructure, platelet structure, SEM, TEM.

1 INTRODUCTION

Alpha alumina, known as corundum, has been attraction attention as one of most important ceramic material due to its significant properties such as: high strength at elevated temperature, hardness, high melting point, thermal conductivity, chemical inertness, abrasion resistance and so on [1-3]. Such excellent properties made α-Al_2O_3 nanoparticle promising material for a wide range of applications such as: electronics [4], optoelectronics [5], and reinforcement filler in composites [6,7]. Recently, α-Al_2O_3 nanostructures such as: nanotube [8], nanowire, nanobelt [9], nanoplatelet [10] and spherical nanoparticle [11] have been received attraction in mechanical applications. Different methods used to synthesis alpha alumina nanostructures including: Chemical Vapor Deposition (CVD) [9], Combustion Chemical Deposition (CCD) [11], Atomic Layer Deposition (ALD) [12], and Spray pyrolysis [13], and. All these techniques require specific equipments, while industrial processes must be affordable, time-consuming and abundant production rate [14]. Solution combustion synthesis (SCS) is time efficient, low-cost, safe and environmental friendly procedure for metal oxide synthesis [15]. Furthermore, in techniques mentioned above approaching alpha phase, High-temperature annealing above 1100^o C of production is mandatory [16-19]. In present work, α-Al_2O_3 platelet nanostructure with high purity obtained directly from solution combustion procedure.

2 EXPERIMENTAL PROCEDURE

2.1. Materials:

Aluminum nitrate nonahydrate extra pure provided from E. Merck (India) limited Co. Urea extra pure purchased from Thomas Baker (chemicals) PVT limited Co (India).

2.2. Sample preparation

Crystalline α-Al_2O_3 platelet nanostructure was synthesized by solution combustion. Mixture solution of aluminum nitrate and urea prepared by dissolving 15.0 gm aluminum nitrate in 20 ml distilled water using magnetic stirrer, 6.0 gm urea slowly added to the solution through vigorous stirring. Transparent solution placed on a pre-heated hot plate. Oxidizer to fuel ratio (ψ factor) kept unit in this experiment. Obtained alpha alumina powder dehydrated at 450o C in an air furnace to improve purity.

3 RESULTS AND DISCUSION

D8 Advance (Bruker) X-ray diffractometer used to record XRD patterns, with Cu Kα irradiation by wavelength 1.54Å (40 kV, 40 mA). Morphological study and chemical characterization of powder were observed by Scanning Electron Microscopy (SEM) using a S-3400N (Hitachi High-Technologies, Japan) microscope, Transmission electron microscopy (TEM), High resolution

TEM (HR-TEM), and selected area electron diffraction (SAED) patterns obtained using a JEM-2100 (JEOL, Tokyo, Japan) microscope. The SZ-100 nanoparticla series instruments (Horiba, Kyoto, Japan) used to study particle size and thermal gravimetric analysis and differential thermal analysis (TG/DTA) performed in air at heating rate of 30° C/min, using a thermal analyzer model XSTAR6000 (Hitachi High-Tech Science Corporation, Tokyo).

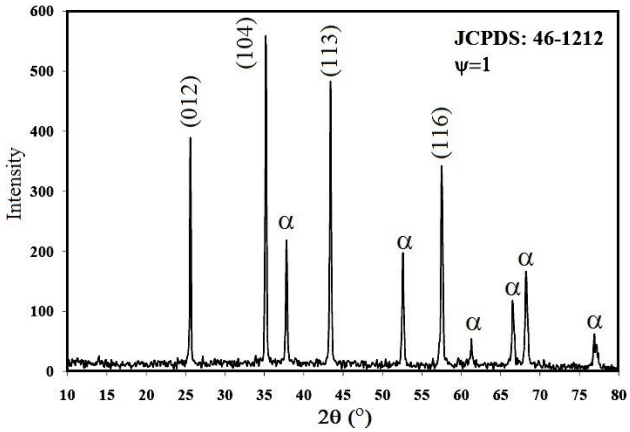


Fig.1. XRD pattern of α-Al_2O_3 synthesis by solution combustion method of aluminum nitrate and Urea in 20 ml distilled water.

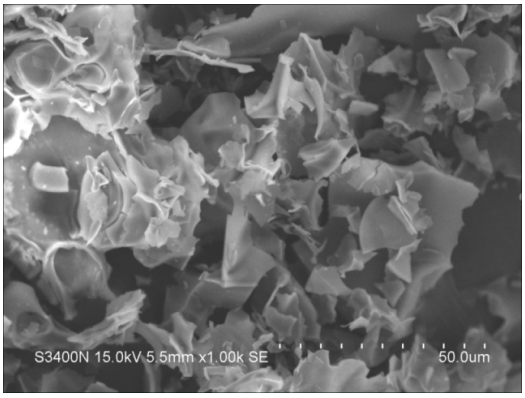
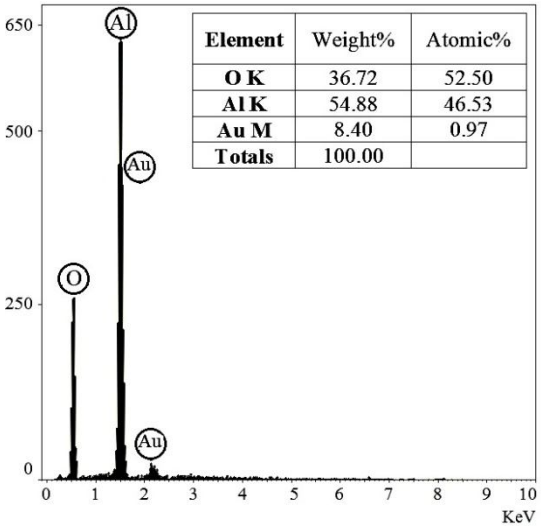


Fig.2. SEM images of α-Al_2O_3 sample, indicates platelet morphology.



Element	Weight%	Atomic%
O K	36.72	52.50
Al K	54.88	46.53
Au M	8.40	0.97
Totals	100.00	

Fig.3. EDAX spectrum of α-Al_2O_3 synthesized by solution combustion method.

Scanning Electron Microscopy (SEM) with Simultaneous EDAX was employed to study platelet morphology of product (Fig.2.) and EDAX spectrum confirms high purity of α-Al_2O_3 (Fig.3.) and reveals that particle is composed of Al and O elements. Au element observed is due to preparation process for SEM, because alumina is ceramic material.

Fig.4. and Fig.5. show the TEM micrographs of α-Al_2O_3 powder obtained from the solution combustion of Aluminum nitrate (oxidizer) and Urea (fuel) in 20 ml aqueous medium and desiccated at 450°C for 1 h. This shows that most crystallites exhibit hexagonal platelet morphology. The side lengths of the platelets were ~80 nm for 20 ml aqueous.

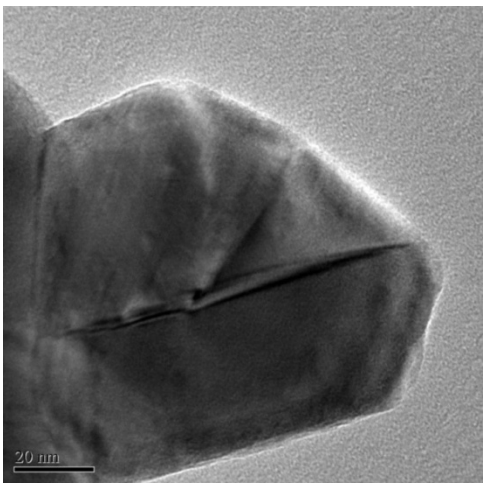


Fig.4. TEM micrograph of α-Al2O3 nanoparticles shows hexagonal shape synthesized by SCS.

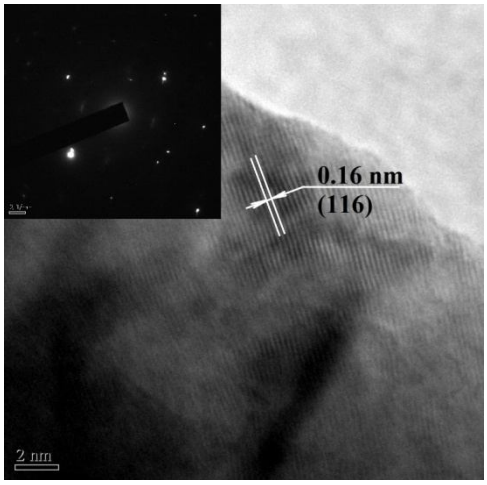


Fig.5. HR-TEM micrograph and SAED pattern of α-Al_2O_3 nanoparticles illustrates d-spacing.

Further characterization of platelet was applied using Selective Area Electron Diffraction (SAED), as shown in Figure.6. The regular squarepattern and strong diffraction spots indicate a single-crystal structure in the platelet and proof Rhombohedral crystalline structure obtained from XRD pattern for both samples.

Thermal gravimetric and differential thermal analysis (TG/DTA) thermograph (Fig.6.) with soft slop and without sharp peaks proofs single phase and no impurity in sample.

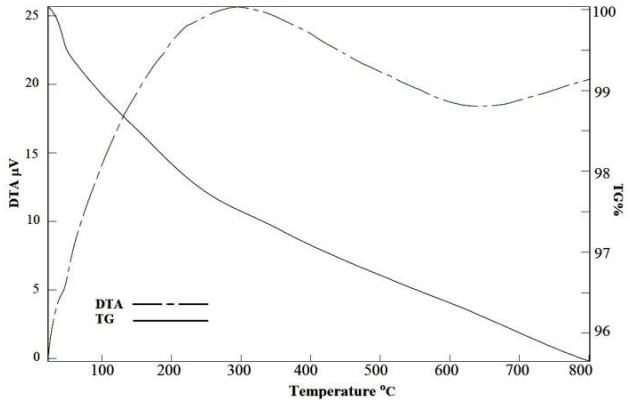


Fig.6. TG/DTA graph of α-Al_2O_3.

4 CONCOLUSION

Crystalline α-Al_2O_3 platelets were synthesized by combustion of aluminum nitrate and urea mixture in aqueous media. High purity of α-Al_2O_3 nanostructure obtained from combustion, illustrated in XRD and TG/DTA graph. TEM and SEM characterizations show platelet structure of product without requirement of annealing at high temperature.

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