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Phase Changes Under Heat Treatment During Synthesis of α-Al_2O_3 Nanoparticles by Sol Gel Method

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ABSTRACT

In the present study, α-Al_2O_3 nanoparticles synthesized using alcoholic solution of $AlCl_3$, 25% NH_3 and Polyvinyl alcohol (PVA). PVA act as a capping agent. All these chemicals were inexpensive raw materials. Stable crystalline phase of α-Al_2O_3 nanoparticles occurred at a temperature 1100^0C. During heat treatment, stableα-Al_2O_3 can be obtained through the series of phase transformations from boehmite, γ, δ, θ to α- phase of Al_2O_3. Crystalline nanoparticle formation of the dried sol was investigated using x-ray diffractometry (XRD). XRD shows crystal size increases from 25 nm to 32 nm when calcination temperatures increases from 500^0C to 1100^0C. It was shown that crystal size increases during the heat treatment. The morphology of α-Al_2O_3 nanoparticles was studied using Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), along with energy- dispersive X-ray analysis (EDAX). TGA and DTA shows, 77% weight loss and phase transformations. Synthesized α-Al_2O_3nanoparticles were applied in waste water treatment as an adsorbent.

KEYWORDS: Sol-gel method, phase change, heat treatment,calcination.

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1. INTRODUCTION

Alumina (Al_2O_3) is the most important ceramic materials used for fabrication of catalyst, catalyst supports,adsorbent materials, coatings and for industrial applications.[1]In the present work an alumina nanopowder is synthesized by sol-gel method using low cost aluminum chloride($AlCl_3$),ammonia (NH_3) and polyvinyl alcohol(PVA). Sol -gel method is most convenient method to produce ceramic nanoparticles. It consist of formation of solfrom alkoxides or organometallic precursors. In this, suspended particles polymerized at low temperatures and thus generated wet gel is then dried and heat treated.[2]However long gelation time is the drawback of sol-gel route.[3]Al_2O_3 exists nearabout fifteen distinct crystallographic phases and it can undergo a variety of transitions until the most stable corundum structure α-Al_2O_3 -forms at high temperatures.[4]During the thermal treatment, stable α-Al_2O_3 phase can be obtained through the following series of phase transformations before conversion to α- Al_2O_3 .

Hydrous alumina $\longrightarrow$ bohemite $\longrightarrow$ γ $\longrightarrow$ δ $\longrightarrow$ θ $\longrightarrow$ α[5]

The aim of the present paper is to study phase changes through TGA,DTA and XRD obtained at different temperatures.

2.EXPERIMENTAL SECTION

α- Al_2O_3 nanoparticles were synthesized by using aluminum chloride as a precursor,25% ammonia and polyvinyl alcohol (PVA). Alcoholic solution 0.5 M $AlCl_3$ was prepared,25%NH_3 was added drop by drop till resulting solution turned to a white sol, PVA was added until it becomes a transparent sticky gel. The gel was allowed to maturate for 24 hours at room temperatures and heat treated at 100^0C for 24 hours. The driedgel was divided into 4 parts and were heat treated at 500^0C,700^0C,900^0C and 1100^0C for 4 hours respectively.

3.RESULTS AND DISCUSSION

3.1. SEM/TEM and EDAX of Al_2O_3 nanoparticles

SEM image of the Al_2O_3 NPs gives the distributionpattern and size of the nanoparticles(Fig. 1A,B). The TEM micrographsshows slight agglomeration with spherical morphology and their average particle size were 77.7 nm (Fig. 1C,D)The SAED pattern of Al_2O_3 NPsshows that the rings are composed of dots suggesting the crystalline nature of these particles(Fig. 2E).The quantitative analysis ofthe Al_2O_3 nanoparticle was done using EDAX spectroscopymeasurement and it shows Al and Oas the major components of aluminium oxide nanoparticlesin the heads as shown infigure 2(F).

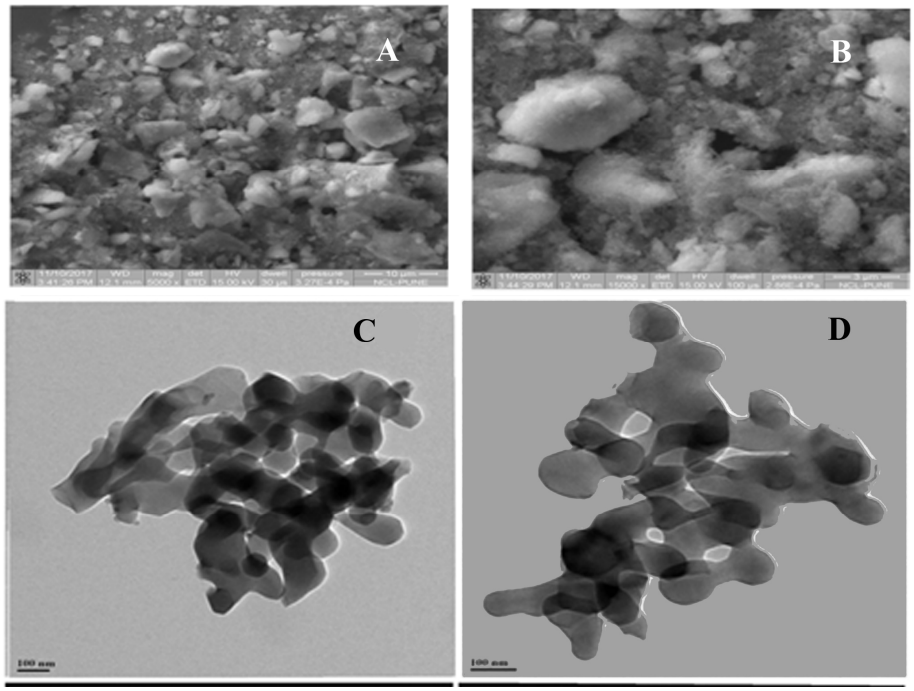


Figure 1. Scanning electron micrographs (SEM) (A, B) and Transmission electron micrographs (TEM) of Al_2O_3 nanoparticles (C, D).

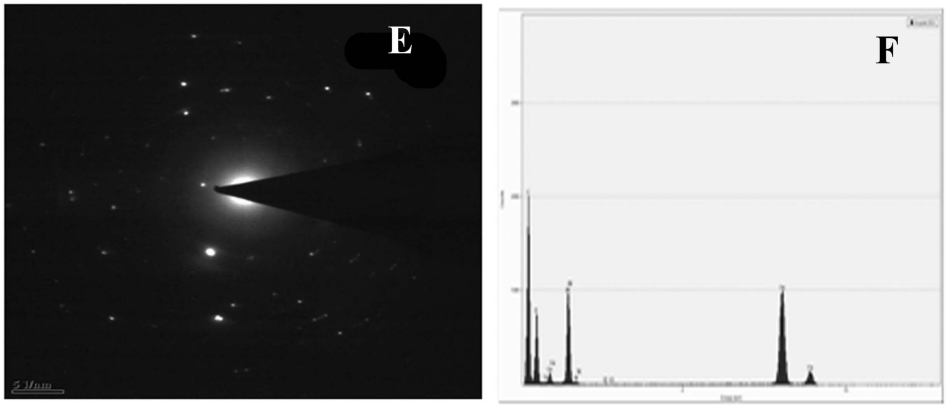
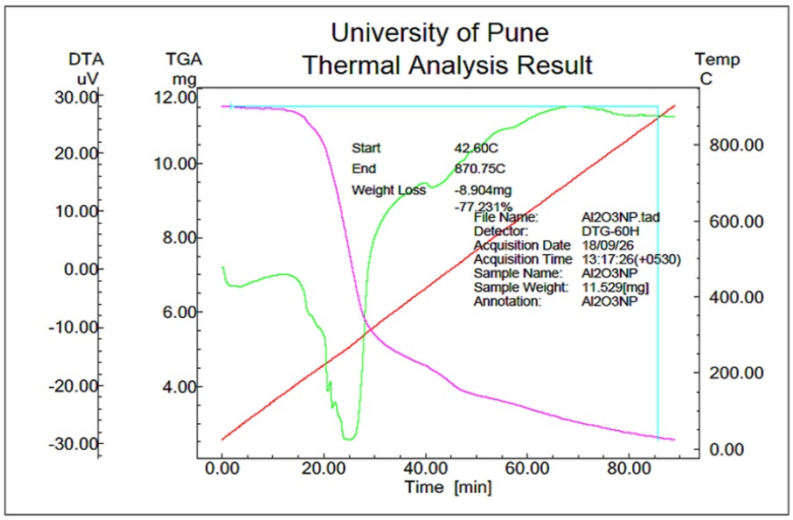


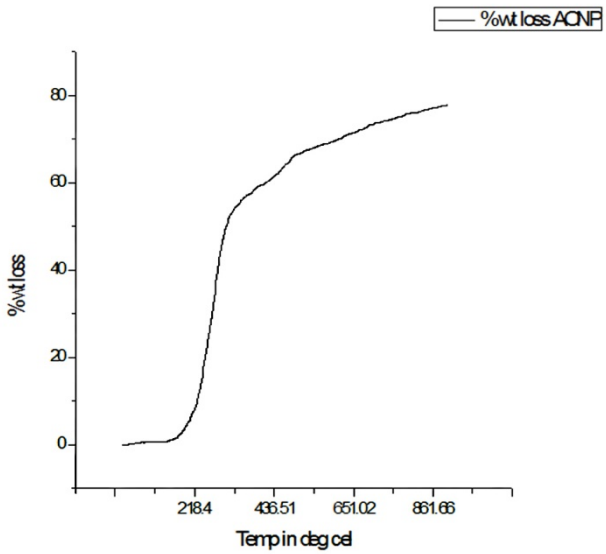
Figure2. Selected area electron diffraction pattern (E) and EDAX of Al_2O_3 nanoparticles (F).

3.2. Thermal analysis

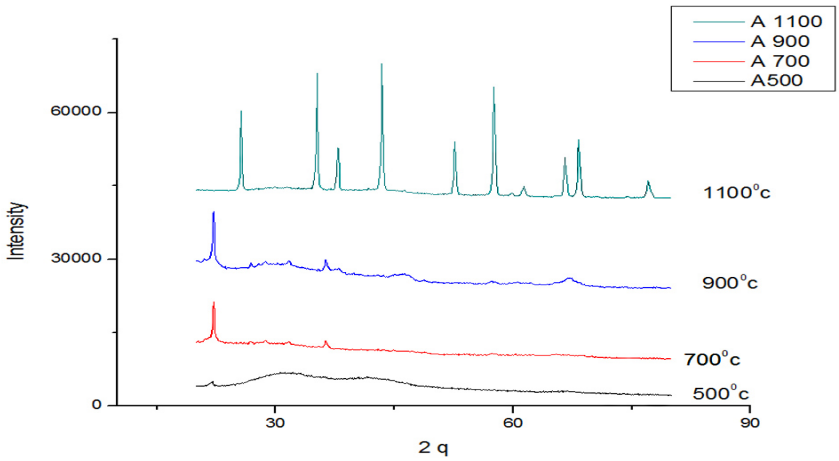
The % weight loss with temperature of TGA curve shows that nanoparticle formation temperature is 762^0C and transition temperature 800^0C with one step weight loss of 77% .The DTA curve shows endothermic reaction attributed to moisture loss and to hydroxyl loss from the decomposition of hydrated aluminum chloride and aluminum hydroxide (Graph 1a,b) The endothermic effects at about 800^0C and 900^0C may be due the transformations of polymorphous enantiotropy γ- Al_2O_3 in α- Al_2O_3. Transformation temperature γ- Al_2O_3 to α- $Al_2O_3$is less than 1000^0C shows small crystallite size and degree of crystallinity characteristic of the nanopowder.[2]



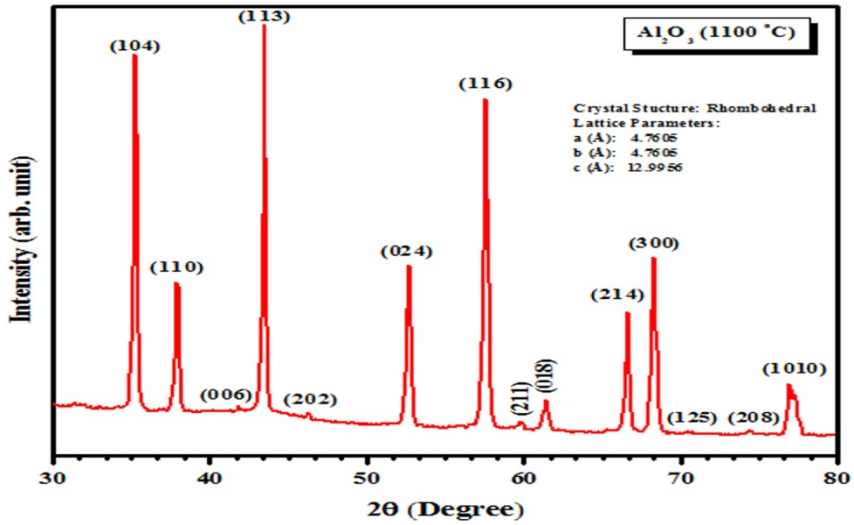
Graph 1(a).Thermal analysis (DTA and TGA)of α- Al_2O_3 nanoparticles obtained by Sol-gel method, calcined at 1100^{0}C temperature for 4h



Graph 1(b). The %weight loss of α- Al_2O_3 nanoparticles



Graph 2.XRD Patterns of dry gel calcined for 4 h at different temperatures



Graph 3. X-ray Diffraction pattern of α- Al_2O_3 nanoparticles

3.3. XRD analysis

Graph 2 shows the comparable XRD patterns of powders prepared by different calcination temperatures from 500^0C to1100^0C.X-ray diffraction images for the powders obtained from precursors $AlCl_3$ and dried at 24 hours after maturation of 24 hours,heat treated for four hours from 500^0C to1100^0C separately. It shows that thermal treatment leads to its decomposition with the formation of a mixture of γ- Al_2O_3phase(JCPDS - 47-1308) and α- Al_2O_3 phase (JCPDS file-71-1678).[6]

XRD at temperatures 500^0C and 700^0C, shows there are 4-5 broad peaks in the pattern for the sample sol- gel sample which are difficult to index according to the JCPDS data indicating that the powder is possibly amorphous. Powder at this temperature shows the existence of organic materials and confirms XRD pattern, where these compounds have prevented particles from forming crystal structure. However, the diffraction peaks are a little sharper and attributed to γ- Al_2O_3 for the sol-gel sample.[7] All diffraction peaks exhibit high degree of broadness due to formation of nanocrystals. The characteristic peaks of γ- Al_2O_3 is improved for the sol-gel sample with increasing calcination temperature upto 1000^0C.[8]The diffraction peaks of δ and γ are very close to each other, in fact overlapping in some positions. It can indicate that γ- Al_2O_3 co-exists with δ - Al_2O_3.[9] Increasing temperature of heat treatment upto1100^0Cfor four hours results in the formation of only α- Al_2O_3 (JCPDS file-71-1683) (Graph 3). At1100^0C single phase α- Al_2O_3 is completely formed. The diffraction pattern is extremely sharp indicating the existence of highly crystalline material which is shown by the curve at temperature 1100^0C.Average crystal size calculated from Debye scherrer formula was found to be increasing from 25 nm to 32 nm of dried gel when calcination temperature increases from 500^0C to1100^0C.

4. CONCLUSION

The sol-gel synthesis of α- Al_2O_3wasrelatively simple and easy method. The resulting α- Al_2O_3 powder were characterized by X-ray diffraction, differential thermal analysis and thermo gravimetric analysis (DTA,TGA). Applying heat treatment at temperatures up to 1100^0C for 4 hours α- Al_2O_3 powder was obtained at nanometric scale having rhombohedral structure. Its crystal size ranges from 25 nm to 32 nm after calcinations of dried gel from 500^0C to1100^0C. α- Al_2O_3nanopowder have superior properties as compared to the powder obtained in larger particle size and it can be used as a effective adsorbent in waste water treatment.

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