

Thermal Treatment of Aluminium Trihydroxide and its Effect on the Reactivity

¹F. SHAHEEN, ¹W. A. SHAH, ¹M. P. I. QAZI AND ²M. L. MIRZA

¹Glass and Ceramics Research Centre PCSIR Laboratories, Complex Lahore Pakistan

²Department of Chemistry, Islamia University Bahawalpur Pakistan.

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Summary: Aluminum trihydroxide $\text{Al}(\text{OH})_3$ was heat treated at temperatures 250, 350 and 550°C and the ultimate effect of water of crystallization on its reactivity was studied. The reactivity was found reduced to half on heating. The process described thermal transformation of crystalline phase from gibbsite to boehmite and to alumina.

Introduction

Alumina in the form of amorphous or crystalline phases with micro/nano porous structure is of great interest now a days due to its remarkably versatile applications [1]. Nano alumina is synthesized by employing aluminium salts [2], aluminium alkoxides [3], aluminium hydroxides [4] etc. Thermal transformation of aluminium hydroxide and its subsequent conversion to transitional aluminas depends upon the morphology and crystalline size [5-7]. Aluminum hydroxide exists in several modifications which include hydrargillite or gibbsite, bayerite, boehmite, diaspore and nordstrandite. The structures of all the aluminium hydroxide consist of stacked double layers of oxygen atoms in which aluminium ions are located in octahedral coordination in interstices [4]. The physical and chemical aspects of aluminas and aluminas hydrate have been studied [8, 9].

In the present study aluminium trihydrate was subjected to various specific heat treatments for identical time intervals. The structural changes were interpreted and chemical reactivity towards nitric acid was monitored.

Results and Discussion

Thermal analyses of aluminum trihydroxide before and after ignitions are shown in Fig. 1 and their XRD diffractograms in Fig. 2 respectively. The DTA curve of sample 1a shows two small endothermic peaks at 223, 550 and a large endothermic peak at 288°C. The curve is typical of dehydration of aluminum hydroxide [10]. The total weight loss corresponding these peaks is 33 %. The theoretical weight loss for the reaction is 34 %, which is comparable with the experimental results. DTA and TG results are given in Table-1. TG depicts that

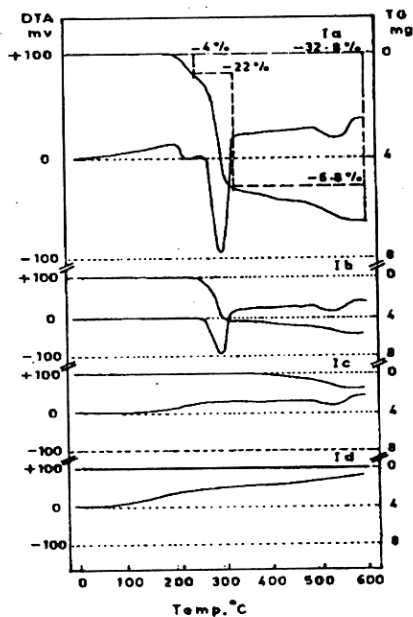


Fig. 1: DTA and TG Graphs of Aluminum Trihydroxide with 1a) with zero ignition loss, 1b) with 4% ignition loss, 1c) with 26% ignition loss, 1d) with 33% ignition loss.

water of crystallization is released at three different temperatures. In temperature range 25-250°C a 4 % weight loss was recorded. The major weight loss occurred between 250-350°C. Further 7 % loss was observed above 350°C. XRD pattern obtained for sample 1a is identified as pure aluminum hydroxide. Heat treatment of sample 1b at 250°C resulted in 4 % weight loss. DTA curve shows the elimination of low temperature endothermic peak. Heating of sample 1c

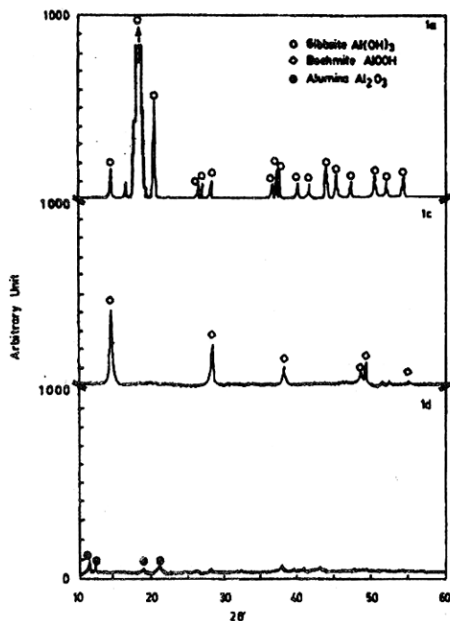


Fig. 2: XRD Patterns of 1a) Aluminum Trihydroxide without ignition loss, 1c) with 26% ignition loss and 1d) with 33% ignition loss.

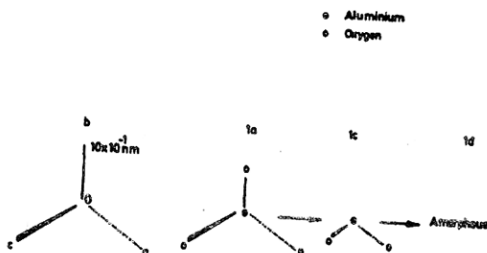


Fig. 3: Unit Cell of Crystal System 1a) Gibbsite $\text{Al}(\text{OH})_3$ Monoclinic, 1c) Boehmite $\text{AlO}(\text{OH})$ – orthorhombic and 1d) Amorphous Al_2O_3 ⁽⁸⁾

at higher temperature i.e. 350°C resulted in 22 % weight loss. The principal endothermic was eliminated and crystalline phase transformed to aluminum hydroxyoxide $\text{AlO}(\text{OH})$. Further heat treatment at 550°C resulted in 7 % weight loss. The DTA trace of sample 1d is a straight line showing none of the peaks but change in crystalline phase is obvious in XRD pattern. Aluminum hydroxyoxide was converted into amorphous aluminum oxide Al_2O_3 . Schematically the thermal event of $\text{Al}(\text{OH})_3 \rightarrow$

$2\text{AlO}(\text{OH}) \rightarrow 2\text{Al}_2\text{O}_3 + 3\text{H}_2\text{O}\uparrow$. The ignition of aluminum trihydroxide resulted in the transformation of crystalline phase from aluminum hydroxide (gibbsite) to aluminum hydroxyoxide (boehmite) and finally to amorphous aluminum oxide (alumina). The conversion of crystal structure [11] is shown in Fig 3.

Table-1: DTA and TG data of aluminum trihydroxide

Temp (°C)	DTA		TG Weight loss (%)
	Exo/Endothermic	Temp range (°C)	
223	Small Endo	30-250	4
228	Large Endo	250-350	22
550	Medium Endo	350-550	7

The chemical reaction between aluminum trihydroxide and nitric acid to give aluminum nitrate nonahydrate $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ may be written as $\text{Al}(\text{OH})_3 + 3\text{HNO}_3 + 6\text{H}_2\text{O} \rightarrow \text{Al}(\text{NO}_3)_3 + 9\text{H}_2\text{O}$. The effect of the ignition of aluminum trihydroxide on the yield of aluminum nitrate crystals is shown in Fig 4. The % age yield was calculated by weighing aluminum nitrate crystals and comparing with the theoretical value. Aluminum trihydroxide with out ignition reacted with nitric acid and the % age yield of aluminum nitrate nonahydrate crystals achieved was 60 %. Ignition up to 250°C has shown a slight increase in the yield i.e. by 2 %. However; heat treatment at 350°C had drastic effect and reduced the reactivity of aluminum hydroxyoxide to 33 %. On further heat treatment at 550°C the reactivity of amorphous aluminum oxide towards nitric acid almost remained the same.

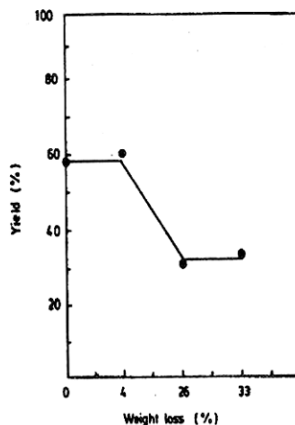


Fig. 4: The effect of ignition of Aluminum Trihydroxide on %age yield of Aluminum Nitrate Nonahydrate Crystals.

Experimental

Aluminum trihydroxide $\text{Al}(\text{OH})_3$ (China) was used as starting reagent. Three samples of aluminum trihydroxide ignited at 250, 350 and 550°C at the rate of 20°C per minute separately. The heating temperature was maintained for 2 hours in each case. Heating was carried out in platinum crucible in high temperature rapid heating electric resistance furnace, RHF/3 Carbolite, UK. The samples were characterized using differential thermal analyzer (DTA), thermogravimetric analyzer (TG) and powder X-ray diffraction analyzer (XRD). DTA/TG studies were carried out on DT-40 thermal analyzer, Shimadzu Corporation, Kyoto, Japan, by heating 20 mg of sample @ 10°C/min up to 600 °C and the data was auto recorded. XRD studies were carried out using Seimen's Diffractometer D 5000, at 10-60° 2 θ value. For convenience, sample with zero ignition loss was denoted as Ia and three ignited at previously described temperatures as Ib, Ic and Id respectively. 2 gm of each sample were reacted with 100ml of 30 % HNO_3 in an autoclave at 550 KPa pressure for 2 hrs. The resulting solution was filtered using distilled water. The filtrate was concentrated on water bath at 100°C temperature. To the concentrate were added a few crystals of aluminum nitrate as seedlings and crystals were developed at room temperature.

Conclusions

It was concluded that the reactivity of aluminum trihydroxide depends critically upon the heat treatment and subsequently to other factors. Without ignition, the reactivity with nitric acid was maximum whereas 34 % ignition loss made it chemically inert and reduced the reactivity to half. As

revealed by XRD the reduced reactivity is related to the phase transformation during heat treatment i.e. the conversion of gibbsite or α aluminum hydroxide $\text{Al}(\text{OH})_3$ to boehmite or aluminum hydroxyoxide $\text{AlO}(\text{OH})$ and to alumina or amorphous aluminum oxide Al_2O_3 .

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