Hydrothermal Synthesis of Boehmite from Aluminum Sulfate

M.L. Guzmán-Castillo[1], F. Hernández-Beltrán[1], A. Rodriguez[1] and X. Bokhimi[2]

[1]Instituto Mexicano del Petróleo, Eje Central Lázaro Cárdenas 152, 07730, México, D. F., [2]UNAM, A:P: 20-364, 01000, México, D. F.

Introduction

Although known from long time transitional aluminas are still subjected to basic research with a specially renewed interest. Aluminas have enormous commercial importance as adsorbents and catalysts components in many chemical processes, including cracking, hydrodesulphurization and hydrocracking of oil cuts (1,2). Transitional aluminas are prepared by calcination of aluminum oxy-hydroxides. One of the forms is γ-Al_2O_3 which derives from boehmite. The properties of γ-Al_2O_3 materials are strongly related to the properties of the precursor boehmite and mainly depend on the crystal size (3,4). In this work we investigated the effect of the synthesis temperature of boehmites onto varying properties of their correspondent γ-Al_2O_3.

Results and discussion

Samples were precipitated in basic pH from solutions of aluminum sulfate under hydrothermal conditions. We investigated the effect of the synthesis temperature on the physicochemical properties of boehmites and their corresponding γ-Al_2O_3 which were obtained by calcination at 600°C. The samples were studied by XRD-Rietveld refinement, N_2 adsorption, TGA-DTA and FTIR-pyridine adsorption.

Solids with the boehmite structure of varying crystallinity was observed in all cases but aluminum sulfate was also observed in samples precipitated at lower temperatures (Figure 1). The crystallinity and crystallite size of boehmites increased monotonically with the synthesis temperature (Table 1). The surface area (S_{BET}) went through a maximum for γ-Al_2O_3 obtained from boehmite precipitated at 140°C. Compared to the rest of the samples, the average pore size increased notoriously for γ-Al_2O_3 obtained from the boehmite produced at 180°C.

TGA-DTA of boehmites showed a continuous weight loss up to completion of dehydoxylation. Transition of boehmite to γ-Al_2O_3 was observed between 380-550°C, through an endothermic change. The transition temperature to γ-Al_2O_3 and then to α-Al_2O_3 could be clearly related the crystallite size as previously observed with boehmites obtained from aluminium chloride (4). Small crystals produced γ-Al_2O_3 at lower temperature, thus showing lower stability while possessing a less ordered structure with smaller pores (5). However, the comparatively lower S_{BET} obtained from boehmites precipitated at lower temperature is not fully consistent with their small crystal size. Differences could be explained to the presence of some sulfate impurities that remained in γ-Al_2O_3 after calcination. The chemical analysis showed higher sulfur content in γ-Al_2O_3 from boehmites with smaller crystallite size. Therefore, synthesis at lower temperatures seemed to produce sulfate impurities that partially blocked pores in γ-Al_2O_3.

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D patterns of samples hydrothermal conditions A) ples; B) γ-Al_2O_3 sampl Sulfate; ■ Aluminum Sulfate

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	Acidity			[S]	Crystal Size (Å)		Transition T, °C	
	Bronsted	L	Å	wt. %	Boehmite	γ-Al_2O_3	γ-Al_2O_3	α-Al_2O_3
	-		35	6.48	-	-	388	1085
	52		43	5.86	16.9	19	388	1095
	12	126	28	4.70	23	18	390	1117
0		248	49	2.376	36	29	509	1161
80		136	159	0.001	102	51	520	1205

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