In Situ Investigation of Boehmite- γAlumina Transformation

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Introduction

The dehydroxylation mechanism of boehmite has received considerable attention particularly due to their end products properties, commonly referred to as transition aluminas. The application of such materials includes catalysis, adsorption, and ceramics [1]. However, there is a wide gap in the literature in terms of understanding transition alumina structure. This factor is attributed to its poor crystallinity, which compromises any attempt to conduct a thorough characterization [2]. So far, most of the characterization studies focus on X-Ray Diffraction (XRD) and Diffuse Reflectance Infrared Fourier Transform spectroscopy (DRIFTS). Concerning the crystalline structure of boehmite, X-ray Rietveld refinements indicate that the structure is well ordered [3]. These studies report that the irreversible transformation of boehmite to γ-Al2O3 forms a defect spinel structure. To understand this transformation and the nature of the end product, it is usually considered that such defect structure results from coherent spinel domains. In our work, we provide some insight into this process by presenting some results of X-Ray diffraction and DRIFTS, both in situ.

Materials and Methods

Boehmite samples were obtained from bayerite by hydrothermal synthesis at 150ºC for 24h in an autoclave internally covered with Teflon®. The material obtained was filtered and dried in a muffle furnace at 102º C. These samples were analyzed in a Rigaku DMax X-Ray diffraction equipment (XRD) coupled with an Anton Paar XRK900 catalytic reactor. The diffractograms were obtained at room temperature and at intervals of 100ºC up to 450 ºC. The DRIFTS analysis was carried out in a Nicolet Nexus 470 spectrometer equipped with an MCT-A detector, 4cm^{-1}, and a Spectra-Tech diffuse reflectance cell. The sample was heated under helium flow, 30ml/min, and the spectra were recorded from 300º C and at intervals of 100º C up to 580º C. This temperature was kept for 90 min, and spectra were also recorded at this time.

Results and Discussion

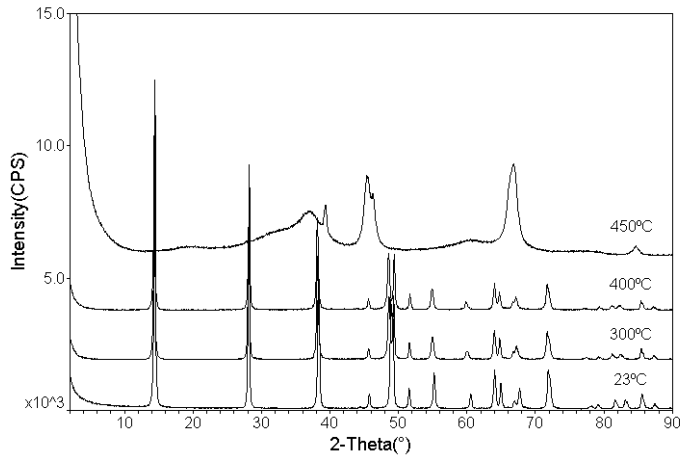

Figure 1- in situ XRD of boehmite- γAl2O3 transformation

The diffractogram at room temperature shows the characteristic peaks of well-crystallized boehmite. At 450º C there is an abrupt change in the structure, and instead of finding boehmite characteristic peaks, a new pattern is found. As can be seen in the diffractogram, the diffuse pattern indicates the formation of γAl2O3 phase. The increase in the small angle X-ray scattering points to the presence of a porous structure, typical of γAl2O3. These results are consistent with DRIFT spectra, which show boehmite transformation into γAl2O3 through the gradual disappearance of OH stretching (ν_s –3400 cm^{-1} and 3185 cm^{-1} ν_{as}) bands[4]. These hydroxyls are responsible for the cohesion between the boehmite layers. Though our research is underway, our preliminary data indicate that in situ investigations of the transformation of boehmite-derived aluminas can potentially fill an important gap in current studies.

Significance

Our study aims to contribute to a better understanding of boehmite-derived aluminas, whose transformations, though extensively studied, still point to a gap in the literature, particularly because in-situ investigations are lacking.

References

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