SOL-GEL PREPARATION OF In$_2$O$_3$-Al$_2$O$_3$ SUPPORTS WITH CONTROLLED TEXTURAL AND STRUCTURAL PROPERTIES

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Abstract

Alumina supports with a very narrow pore size distribution were obtained with indium-doped alumina prepared by the sol-gel method. The formation of aluminum in pentahedral coordination was identified by $^{27}$Al NMR-MAS spectroscopy. A good correlation was obtained with the $^{27}$Al NMR-MAS intensity signal and the activity in isopropanol dehydration. The insertion of In$^{3+}$ substituting some Al$^{3+}$ in the alumina network was suggested.

Keywords: Indium-doped alumina, sol-gel alumina, indium-alumina $^{27}$Al NMR-MAS, indium-alumina XRD, indium-alumina isopropanol dehydration

INTRODUCTION

Alumina preparation by the sol-gel method has been reported in a large number of papers as a suitable method to obtain alumina with important applications as adsorbent, support or catalyst. In most of the cases the textural...
and structural properties of sol-gel prepared alumina were controlled by the variation of the synthesis conditions: hydrolysis rate of alkoxides (according to the hydrolysis pH), solvent, water/alkoxide ratio, activation conditions and aging of the gels [1-7]. Additionally, the doping of alumina gel with metallic cations has been reported as an alternative method to obtain alumina supports with controlled textural and structural properties. An example of that is the tin-doped alumina [8] used as support for Pt-Sn/Al₂O₃ catalysts which have wide application in the naphtha reforming process [9]. With this in mind, in the present study we report the synthesis of indium-doped alumina by the sol-gel method. The doping metal was chosen because In/Al₂O₃ has been reported as a suitable support for the preparation of Pt-In/Al₂O₃ bifunctional reforming catalysts [10], as well as for important applications in the NO reduction in gas exhaust converter [11, 12]. The In/Al₂O₃ synthesis was made by adding indium acetylacetonate to an alumina gel. Nitrogen adsorption, X-ray diffraction, ²⁷Al NMR-MAS spectroscopy and NH₃-TPD were used for the characterization of the solids. The acidity was evaluated in the acid-base isopropanol dehydration reaction.

**EXPERIMENTAL**

**Preparation of supports**

Alumina reference xerogels were prepared from a starting solution containing 238 mL of ethanol (J. T. Baker 96%) and 14 mL of distilled water, adjusting the pH value to 3 and 5 with nitric acid (Baker 65% Vol) and glacial acetic acid (J.T. Baker 99.7%), respectively. After that, the solution was maintained under reflux and stirring for 72 h at 70°C. Then, the gel was dried in an oven at 80°C for 12 h and annealed at 600°C for 4 h, using a heating program rate of 2°C/min. For the preparation of the indium-doped alumina, there were added simultaneously 70 mL of an ethanol-water solution (3:1 vol) containing the appropriate amount of indium acetylacetonate (Chemat 98%) and 48 g of aluminum tri-sec-butoxide (Aldrich to 97%) to the starting solutions (pH 3 and pH 5). The amount of indium acetylacetonate added was calculated to obtain a concentration of 1 and 3 wt.% of indium in the support. After that, the doped gels were dried in an oven at 80°C for 12 h and then annealed at 600°C for 4 h, with a heating program rate of 2°C/min. For identification, the reference alumina and indium-doped alumina supports were labeled as InX-Al₂O₃-Y, where X denotes the indium content and Y the pH used for the hydrolysis.