Preparation of Ni/Al<sub>2</sub>O<sub>3</sub> by microemulsion and sol-gel

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Introduction

Nanoparticles have been extensively studied in recent years seen their potential applications in several areas such as materials for catalysis. The main player in a catalytic process is the catalyst compound often small metal particles dispersed on an inert support. For this reason, research on the synthesis and characterization of nanoparticles has attracted considerable interest in this area.

In this work, we studied the effect of the preparation method on the physicochemical characteristics of Ni/Al<sub>2</sub>O<sub>3</sub> catalyst. (25 w%). Sol-gel (SG) and the inverse microemulsion (ME) were used in this case for the synthesis of the catalyst from the precursor salts Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O and (Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O).

Materials and Methods

Nickel catalysts supported on alumina were prepared by two different techniques: sol-gel (SG) and the other more original the reverse microemulsion (ME). The first step of microemulsion method is to set up a reverse microemulsion by mixing an aqueous solution containing 55 g of Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O source, and an appropriate amount of Ni salt, with 150 mL of double distilled water, 561 mL of cyclohexane, 116 mL of butanol and 109 g of surfactant CTAB (cetyltrimethylammonium bromide). Meanwhile, a second similar to the first microemulsion containing ammonium hydroxide (NH<sub>4</sub>OH, Panreac) as the aqueous phase is prepared. These two microemulsions are separately maintained under vigorous magnetic stirring for 1 hour, then mixed and stirred at room temperature for 24 hours. The resulting microemulsion is then filtered and washed several times with methanol. The solids obtained were dried 48 hours in an oven at 110 °C and then calcined at 900 °C (temperature rise = 3 °C.min<sup>-1</sup>) for 2 hours.

SG catalyst is prepared with a mixture containing Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O, M(NO<sub>3</sub>)<sub>2</sub>, double distilled water and aqueous solution of citric acid. The resulting solution is stirred at 80 °C until a formation of a gel and then cooled to room temperature. The gel obtained is dried for 24 hours at 110 °C before being calcined in air at 900 °C for 2 hours. Content components of the catalysts were determined by X)ray fluorescence spectrometry. The specific surface area of the catalysts was measured by adsorption / desorption of N<sub>2</sub> at -196 °C according to the method Brunauer-Emmett-Teller (BET) with a volumetric adsorption unit type N<sub>C</sub>OUlTER SA 3100. The analysis by X-ray diffraction (XRD) is carried out using a powder diffractometer and D8-ADVANCE anticathode Cu (λ = 1.54 Å). The average size of the metal particles is calculated from the width at half height of the peaks corresponding to Ni through the application of the Debye-Scherrer relationship. The morphology of the catalysts was studied by transmission electron microscopy (TEM) on a Philips CM 120 microscope (120 kV) coupled with X-ray spectroscopy energy dispersive (EDX: Energy Dispersive X-ray). EDX analyzes were performed in probe sets for qualitative and quantitative analysis and to examine the homogeneity of the sample.

Results and Discussion

The results of chemical analysis revealed that the actual content of Ni corresponds to the relatively fixed value. This shows the good conditions for preparing catalytic solids via two techniques. The BET area comparison catalysts show specific area significantly higher for the sample prepared by microemulsion. XRD spectra (Figures 1) shows, the presence of intense lines characteristic of the spinel phase NiAl<sub>2</sub>O<sub>4</sub> crystallized in a cubic system {21 °, 37 °, 45 °, 59°} structure (2 ° and 65 °). This result is confirmed by electron diffraction. An example is given in Figure 2. Similar results were obtained by Otero et al. in the presence of Ni/Al<sub>2</sub>O<sub>3</sub> catalysts prepared by sol-gel [1] and the ceramic method [2]. In addition, the diffractograms shows no characteristic line to metal oxide phase and these whatever the method of preparation. This result does not exclude their presence but can be linked to a good dispersion of these oxides on the support and / or a strong metal-support interaction promotes the formation of aluminates. The comparison of the crystallite size NiAl<sub>2</sub>O<sub>4</sub> depending on the preparation technique shows a smaller crystal size for the catalysts prepared by microemulsion compared to its counterpart prepared by sol-gel. This result allows to state that the microemulsion gives a better dispersion of the particles to the substrate surface. Direct observation of TEM images of the catalyst prepared by microemulsion shows particles with good dispersion and a relatively homogeneous distribution. While for the catalyst prepared by sol-gel reveals a larger particle size with a heterogeneous distribution in the form of two-dimensional clusters. In addition, the EDX analysis in the first case showed good uniformity on the content of nickel with an Al / Ni uniform and no nickel oxide free, which will be a confirmation of the formation of nickel aluminates.

References
