THERMAL ANALYSIS OF ALUMINA PRECURSORS PREPARED BY ‘PFHS’ METHODS

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Thermal analysis of alumina precursors prepared by two different PFHS (precipitation from homogeneous solution) methods and a conventional method is described. All three precursors exhibit distinct thermal behaviour patterns, marked by multiple phase transformations, to yield γ-Al₂O₃ ultimately. Thermal analysis studies, coupled with XRD, IR and elemental analysis data, indicate that the precursors obtained by the PFHS methods are monophasic in nature, and hence yield relatively small, uniform microspheroidal alumina in comparison with the alumina obtained by the conventional method.

Preparation of alumina with desired structural, textural, physical and chemical characteristics for catalyst supports can be achieved by carefully controlling the preparation procedure. Generally, the preparation involves the precipitation of hydrated alumina as precursors from salts of aluminium under well-defined conditions and the calcination of these to yield various transition aluminas. The properties of the precursors and their calcination procedures largely determine the characteristics of the final alumina samples. Depending on the precipitation conditions, one can attain single phases or a mixture of the following hydrated alumina phases as precursors, with varying degrees of crystallinity, crystallite size and morphology [1]: gibbsite, bayerite, boehmite, diaspore and nordstrandite. PFHS (precipitation from homogeneous solution) methods of preparation are known to give coarse and easily filterable precipitates with uniform and finer crystallites and, since the precipitation conditions are uniform throughout the solution, a monophasic alumina precursor can be obtained [2].

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In our studies [3] on the preparation of alumina and its role as a support for nickel (hydrogenation) and cobalt-molybdenum (hydrodesulphurization) catalysts, we attempted two methods of PFHS, namely a) neutralization of sodium aluminate by ethyl acetate (EA) hydrolysis and b) precipitation of an alumina precursor from aluminium nitrate by urea hydrolysis in the presence of succinic acid (SA), together with a conventional method, i.e. neutralization of sodium aluminate with nitric acid (NA), to obtain the alumina precursors. Since the characteristics of the aluminas derived from these precursors are also dependent on the nature of the thermal treatment during calcination, a detailed analysis of their thermal behaviour has been carried out in conjunction with other analysis, such as XRD, IR, SEM and elemental analysis. The features of the thermal analysis studies are reported in this paper.

**Experimental**

Detailed methods describing all the conditions of precipitation of the precursors have been presented elsewhere [3]. However, a brief summary of the preparation method, the designation of the samples and the calcinations are given in Table 1. All chemicals used in the preparation were of ‘AnalaR’ grade or equivalent. The precursors were filtered, thoroughly washed and dried at 373 K for 24 hours before being subjected to thermal analysis in a static atmosphere of air in the range 300–1223 K using Stanton and Redcroft DTA and TGA instruments (Model 673–4). A heating rate of 10 deg/min was used.