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Influence of Kneading Conditions on the Textural Properties of Alumina Catalysts Supports for Hydrotreating

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Abstract : Mesoporous alumina is commonly used as a catalyst support for the hydrotreating of heavy petroleum cuts. The process of fabrication usually involves: the synthesis of the boehmite AlOOH precursor, a kneading-extrusion step, and a calcination in order to obtain the final alumina extrudates. Alumina is described as a complex porous medium, generally agglomerates constituted of aggregated nanocrystallites. Its porous texture directly influences the active phase deposition and mass transfer, and the catalytic properties. Then, it is easy to figure out that each step of the fabrication of the supports has a role on the building of their porous network, and has to be well understood to optimize the process. The synthesis of boehmite by precipitation of aluminum salts was extensively studied in the literature and the effect of various parameters, such as temperature or pH, are known to influence the size and shape of the crystallites and the specific surface area of the support. The calcination step, through the topotactic transition from boehmite to alumina, determines the final properties of the support and can tune the surface area, pore volume and pore diameters from those of boehmite. However, the kneading extrusion step has been subject to a very few studies. It generally consists in two steps: an acid, then a basic kneading, where the boehmite powder is introduced in a mixer and successively added with an acid and a base solution to form an extrudable paste. During the acid kneading, the induced positive charges on the hydroxyl surface groups of boehmite create an electrostatic repulsion which tends to separate the aggregates and even, following the conditions, the crystallites. The basic kneading, by reducing the surface charges, leads to a flocculation phenomenon and can control the reforming of the overall structure. The separation and reassembling of the particles constituting the boehmite paste have a quite obvious influence on the textural properties of the material. In this work, we are focused on the influence of the kneading step on the alumina catalysts supports. Starting from an industrial boehmite, extrudates are prepared using various kneading conditions. The samples are studied by nitrogen physisorption in order to analyze the evolution of the textural properties, and by synchrotron small-angle X-ray scattering (SAXS), a more original method which brings information about agglomeration and aggregation of the samples. The coupling of physisorption and SAXS enables a precise description of the samples, as same as an accurate monitoring of their evolution as a function of the kneading conditions. These ones are found to have a strong influence of the pore volume and pore size distribution of the supports. A mechanism of evolution of the texture during the kneading step is proposed and could be attractive in order to optimize the texture of the supports and then, their catalytic performances.

Keywords : alumina catalyst support, kneading, nitrogen physisorption, small-angle X-ray scattering
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