



AREA: Synthesis and characterization of catalysts and adsorbents

Glycerol conversion by employing nano-zeolite catalyst produced by bead milling

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Abstract

Mechanochemistry has been regarded as a reemerged technique that can be particularly useful in tackling issues related to conventional synthesis routes, such as the use of huge amounts of solvents. Besides, fabrication of nanoparticles can be achieved via milling approaches. Zeolites are crystalline aluminosilicates, with well-organized porous systems, and are employed in several important industrial applications, including catalysis, gas separation, and adsorption. The production of nano-zeolites involves numerous challenges. Their synthesis is usually carried out via wet methods, known as bottom-up routes, which are both laborious and expansive. In this context, high-energy milling processes are being employed to produce nanozeolites. Herein, we present the preparation of zeolite type A via a conventional hydrothermal method, and its conversion to nanoparticles by means of processes of bead milling and recrystallization. Zeolite samples were confined in 12 mL containers together with either zirconia beads stabilized with ceria or inox steel beads and treated in a planetary mill (Retsch, model PM-100). The best milling conditions were determined by studying the following parameters: the ratio between the milling agent and the zeolite (2, 3.5, 5, 10, and 20), time (1, 3, 6, and 12 h), rotation speed (200, 400, and 600 rpm) and milling agent diameter (1, 5, and 10 mm). One particular sample underwent a further recrystallization step, employing the thermal treatment with a diluted alkaline silica and alumina source. All the samples were characterized by using X-ray diffraction, scanning electron microscopy (the images bellow clearly show the effect of the milling the zeolite particle size), infrared spectroscopy, and particle size measurements. The conventional zeolite A (A), the chosen milled zeolite (Amilled), its recrystallized form (Amilled&recryst), as well as a sample of nanozeolite, for reference purposes, obtained via a bottom-up method, with structure directing agent (Anano) were evaluated as catalysts for the dehydration of glycerol, indicating that the zeolite in form of nanocrystals produced by milling presents performance comparable to the zeolite nanocrystals obtained by bottom-up approach (93% and 97%, as indicated in the graphic).



Keywords: Nanozeolite, Bead Milling, Top-Down, Glycerol Conversion.

References

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