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Epoxidation of Cycloalkenes Using Bead Shape Ti-Al-Beta Zeolite

Authors : Zahra Asgar Pour

Abstract : Two types of Ti-Al-containing zeolitic beads with an average diameter of 450 to 750 µm and hierarchical porosity were synthesized using a hard template method and tested as heterogeneous catalysts in the epoxidation of cycloalkenes (i.e. cyclohexene and cis-cyclooctene) with aqueous hydrogen peroxide (H_2O_2) or tert-butyl hydroperoxide(TBHP) as the oxidant agent. The first type of zeolitic beads was prepared by hydrothermal treatment of a primarygel (containing the Si, Ti, and Al precursors) in the presence of porous anion-exchange resin beads as the hard shaping template. After calcination, these beads (Ti-Al-Beta-HDT-B) consisted of both crystalline zeolite Beta and an amorphous silicate phase. The second type of zeolitic beads (Ti-Beta-PS-deAl-14.4-B) was obtained by post-synthesis dealumination of Al-containing zeolite Beta beads using 14.4 M HNO_3, followed by Ti grafting (3 wt% per gram of zeolite). The prepared materials were characterised by means of XRD, N2-physisorption, UV-vis, XRF, SEM, and TEM and tested as heterogeneous epoxidation catalysts. This post-synthetically prepared catalyst demonstrated higher activity (cyclohexene conversion of 22.7 % and epoxide selectivity of 33.5 %) after 5 h at60 °C, which emanates from the crystalline structure and higher degrees of hydrophobicity. In addition, the post-synthetically prepared beads were prone to partial Ti leaching in the presence of H_2O_2, whereas they showed to be resistant against Ti leaching using tert-butyl hydroperoxide as the oxidant agent.

Keywords : epoxidation, structured catalysts, hierarchical porosity, bead-shape catalysts
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