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Synthesis, Characterization, and Catalytic Application of Modified Hierarchical Zeolites

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Abstract : Zeolites, classified as microporous materials, are a large group of crystalline aluminosilicate materials commonly used in the chemical industry. These materials are characterized by large specific surface area, high adsorption capacity, hydrothermal and thermal stability. However, the micropores present in them impose strong mass transfer limitations, resulting in low catalytic performance. Consequently, mesoporous (hierarchical) zeolites have attracted considerable attention from researchers. These materials possess additional porosity in the mesopore size region (2-50 nm according to IUPAC). Mesoporous zeolites, based on commercial MFI-type zeolites modified with silver, were synthesized as follows: 0.5 g of zeolite was dispersed in a mixture containing CTABr (template), water, ethanol, and ammonia under ultrasound for 30 min at 65°C. The silicon source, which was tetraethyl orthosilicate, was then added and stirred for 4 h. After this time, silver(I) nitrate was added. In a further step, the whole mixture was filtered and washed with water: ethanol mixture. The template was removed by calcination at 550°C for 5h. All the materials obtained were characterized by the following techniques: X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), nitrogen adsorption/desorption isotherms, FTIR spectroscopy. X-ray diffraction and low-temperature nitrogen adsorption/desorption isotherms revealed additional secondary porosity. Moreover, the structure of the commercial zeolite was preserved during most of the material syntheses. The aforementioned materials were used in the epoxidation reaction of cyclohexene using conventional heating and microwave radiation heating. The composition of the reaction mixture was analyzed every 1 h by gas chromatography. As a result, about 60% conversion of cyclohexene and high selectivity to the desired reaction products i.e., 1,2-epoxy cyclohexane and 1,2-cyclohexane diol, were obtained.

Keywords : catalytic application, characterization, epoxidation, hierarchical zeolites, synthesis
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