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Hierarchical Zeolites as Catalysts for Cyclohexene Epoxidation Reactions

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Abstract : A catalyst-assisted oxidation reaction is one of the key reactions exploited by various industries. Their conductivity yields essential compounds and intermediates, such as alcohols, epoxides, aldehydes, ketones, and organic acids. Researchers are devoting more and more attention to developing active and selective materials that find application in many catalytic reactions, such as cyclohexene epoxidation. This reaction yields 1,2-epoxycyclohexane and 1,2-diols as the main products. These compounds are widely used as intermediates in the perfume industry and synthesizing drugs and lubricants. Hence, our research aimed to use hierarchical zeolites modified with transition metal ions, e.g., Nb, V, and Ta, in the epoxidation reaction of cyclohexene using microwaveheating. Hierarchical zeolites are materials with secondary porosity, mainly in the mesoporous range, compared to microporous zeolites. In the course of the research, materials based on two commercial zeolites, with Faujasite (FAU) and Zeolite Socony Mobil-5 (ZSM-5) structures, were synthesized and characterized by various techniques, such as X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and low-temperature nitrogen adsorption/desorption isotherms. The materials obtained were then used in a cyclohexene epoxidation reaction, which was carried out as follows: catalyst (0.02 g), cyclohexene (0.1 cm3), acetonitrile (5 cm3) and dihydrogen peroxide (0.085 cm3) were placed in a suitable glass reaction vessel with a magnetic stirrer inside in a microwave reactor. Reactions were carried out at 45° C for 6 h (samples were taken every 1 h). The reaction mixtures were filtered to separate the liquid products from the solid catalyst and then transferred to 1.5 cm3 vials for chromatographic analysis. The test techniques confirmed the acquisition of additional secondary porosity while preserving the structure of the commercial zeolite (XRD and low-temperature nitrogen adsorption/desorption isotherms). The results of the activity of the hierarchical catalyst modified with niobium in the cyclohexene epoxidation reaction indicate that the conversion of cyclohexene, after 6 h of running the process, is about 70%. As the main product of the reaction, 2-cyclohexanediol was obtained (selectivity > 80%). In addition to the mentioned product, adipic acid, cyclohexanol, cyclohex-2-en-1-one, and 1,2-epoxycyclohexane were also obtained. Furthermore, in a blank test, no cyclohexene conversion was obtained after 6 h of reaction. Acknowledgments The work was carried out within the project “Advanced biocomposites for tomorrow’s economy BIOG-NET,” funded by the Foundation for Polish Science from the European Regional Development Fund (POIR.04.04.00-00-1792/18-00.

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