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Zirconia functionalized monolithic cores with improved hierarchical porosity for continuous-flow microreactors in cascade reactions

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Authors:	Katarzyna Maresz , Julita Mrowiec-Białoń , Agnieszka Ciemięga
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This work presents a method for the preparation of zirconia-silica monoliths with improved porosity and their application in a continuous-flow system for cascade deacetalization and Knoevenagel condensation reactions carried out in two microreactors connected in series. The post-synthesis treatment of pristine micro/macroporous zirconia-silica monoliths, obtained by one step method, with the use of ammonia and sulfuric acid solutions resulted in mesoporous materials and improved macroporosity. They showed high activity in the deacetalization reaction of benzaldehyde dimethyl acetal, despite a relatively low zirconium content, ca. 0.3 wt.%, probably due to the formation of small quantities of the zirconium sulphate superacid. The Knoevenagel condensation reaction of benzaldehyde with ethyl cyanoacetate was performed in an amine-functionalised microreactor. The cascade process resulted in an 80% yield of ethyl cyanocinnamate and a very high selectivity that reached 99%. The flow resistance and residence time distribution were determined for both reactors.

Metryczka

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