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International Conference on

June 27-28, 2016 New Orleans, Louisiana, USA

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The purpose of the presented studies was to develop simple and e cient methods for obtaining SBA-15 and MCM-41 materials (2D) with high degree of pore ordering, and containing sulfur atoms, as well as to investigate the in uence of synthesis method and reaction conditions on their catalytic activity. Organic sulfur-containing functional groups were introduced into the ordered siliceous matrix by two di erent procedures, namely "one-pot" synthesis or post-synthetic graing. e received formulations were evaluated in the terms of physicochemical properties. Low-angle X-ray di raction (XRD) technique, low-temperature nitrogen adsorption-desorption measurements and transmission electron microscopy were applied for the mesoporosity con rmation in the obtained materials. Moreover, selected samples were subjected to the elemental analysis in order to con rm an incorporation of heteroatoms into/onto the siliceous matrix. In addition, MCM-41 and SBA-15 materials containing sulfur atoms were tested as catalysts in the Friedel-Cra s alkylation. XRD patterns reveales re exes typical for MCM-41 or SBA-15 mesoporous structure. According to the IUPAC recommendations, nitrogen adsorption-desorption isotherms obtained for all synthesized silicas may be ascribed to type IV(a), characteristic to the mesoporous samples. Results of the elemental analysis clearly point out that synthesis methods employed for obtaining modi ed mesoporous materials allow to incorporate organic groups containing sulfur. Synthesized materials were successfully applied as acidic catalysts in the

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