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Small-angle X-ray scattering (SAXS) studies of the structure of mesoporous silicas

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Mesoporous silica materials are mesostructured objects with structural units on the nanometer dimension. A hallmark is significantly developed pore structure and excellent textural properties like high surface area and high pore volume. The unique properties remain function of nature of the material, their porosity, structure and geometry of pores. Due to the exceptional properties they are object of interest and a real challenge in catalysis, adsorption methods, and many other areas of modern chemistry. Porous silica materials are willingly used as adsorbents, catalyst support and drug delivery system due to its high thermal stability, tunable properties, possibility of modification and inactive behavior in relation to active phases and solvents [1-5]. Depending on type of precursors and synthesis conditions silicate structured mesoporous materials may have ordered or disordered pore structures. In a large family of mesopores silica materials, the most valuable and useful are SBA-15 (Santa Barbara Amorphous) [6], MCM-41 (Mobile Crystalline Material) [7,8] and MCF (Mesostructured Cellular Foam) [9]. MCM-41 has a hexagonal arrangement of one-dimensional mesopores with diameters ranging from 2 to 10 nm. SBA-15 exhibit 2D hexagonal network with high degree of structural ordering and presence of two type of porosity (micropores (below 2 nm) and mesopores in the range from 5 to 10 nm). Mesostructured Cellular Foams are three-dimensional (3D) structures with ultra large mesopores with size up to 50 nm. It is composed of uniform sphere-shaped sections interconnected by window pores with anarow size distribution.

Small-angle X-ray scattering (SAXS) measurements of mesoporous silica materials provides information on the distribution of electron density in the mesoporous material. In particular, structure and size of the unit cell as well as type of ordered structure. SAXS can be used to characterize the nanoscale structure both of ordered materials (analisys of position of diffraction peaks) as well as disorded materials for example MCFs. In the case of materials from mesostructured cellular foam family, SAXS measurements, the pore size distribution function and many other derivatives parameters can be obtained.

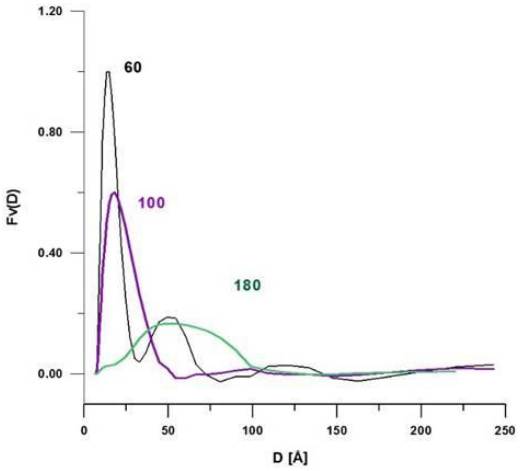

Figure 1. Pore volume distribution function of micropores in SBA-15 materials after hydrothermal treatment in 60°C, 100°C and 180°C respectively.

In this work, the SAXS measurements as well as analysis of SAXS spectra were performed for SBA-15, MCM-41 and MCF structures. Presented materials contained various proportional content of micropores and mesopores. It was found that SAXS technique allows to define parameters for describing micro and mesoporosity regardless of the the ordering degree.

[1] N. K. K. Raj, S. S. Deshpandale, R. H. Ingle, T. Raja, P. Manikandan, *Catal. Lett.* **98** (2004) 217.
[2] X. Zhang, Y. Yue, Z. Gao, *Catal. Lett.* **83** (2002) 19.
[3] S. Wang, *Microporous Mesoporous Mater.* **117** (2009) 1.
[4] M. Vallet-Regí, F. Balas, D. Arcos, *Angew. Chem. Int. Ed.* **46** (2007) 7548.
[5] W. K Cheah, Y. L. Sim, F. Y. Yeoh, *Mater. Chem. Phys.* **175** (2016) 151.
[6] D. Zhao, Q. Huo, J. Feng, B. F. Chmelka, G.D. Stucky, *J. Am. Chem. Soc.* **120** (1998) 6024.
[7] D. Zhao, J. Feng, Q. Huo, N. Melosh, G. H. Fredrickson, B. F. Chmelka, G. D. Stucky, *Science* **279** (1998) 548.
[8] D. Zhao, Q. Huo, J. Feng, F. Chmelka, G. D. Stucky, *J. Am. Chem. Soc*. **120** (1998) 6024.
[9] P. Schmidt-Winkel, *J. Am. Chem. Soc*. **21** (1999) 254.