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Monolithic silica gels prepared by sol-gel method

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Monolithic materials are characterized by a continuous three-dimensional structure which provides access to the surface for a variety of applications. Monoliths are used in different processes, including the emission control of industrial and automobile gases, catalysis, and high-performance liquid chromatography [1, 2].

These materials should possess a selection of characteristics, i.e. mechanical strength, permeability, meso- and macropore ratio, heat and mass transfer efficiency, specific surface area, etc. It is possible to produce materials with necessary properties via careful control of conditions and chemical composition. Organic polymers and silica monoliths offer the possibility of surface modification. However, silica monolith is more resistant to a wider range of solvents, and possesses higher thermal and mechanical stability. The homogeneity of both macroporous space and skeleton thickness of monolithic silica, offering the opportunity to make efficient and selective catalysts with narrow residence time distributions, is an advantage over packed beds.

Porous SiO_2 monoliths with different compositions were prepared by sol-gel method with the use of TEOS as a SiO_2 precursor, CTAB and/or PEG as porogens [3, 4]. The monoliths were characterized using BET, SEM, and water permeability using Darcy's law. The porosity was determined by weighing the dried and water-saturated monoliths.

Silica monoliths with two types of structure were prepared with a careful control of chemical composition and concentration range of porogen/SiO_2. Silica monoliths of type A1 were prepared in the concentration range of porogen/SiO_2 from 0.35 to 0.56 and were found to have bicontinuous structure. These silica monoliths A1 are bimodal having both interconnected flow-through pores and those located in silica skeleton. In this range, as the polymer concentration increases, the size of macropores and the thickness of skeleton decrease from 41 and 24 μm to 7 and 3 μm, respectively. This leads to a decrease in strength of the materials from 2 to 1.2 MPa. The presence of large macropores promotes high values of permeability of monoliths ($3.5 \cdot 10^{-12}$ m^2).

The second type of the silica monoliths (A2) was prepared in the concentration range of porogen/SiO_2 from 0.24 to 0.54. These monoliths had the globular structure with interconnected macropores in the concentration range of porogen/SiO_2 from 0.24 to 0.42. Monoliths of the A2 type possessed high porosity and low skeleton strength which resulted in high permeability (up to 7·10–12 m2). Silica A2 with more strength was formed due to an increased amount of porogen. These monoliths had mesoporous structure and were proven to have low permeability ($8 \cdot 10^{-16}$ m^2). All monoliths had high porosity (85-92 %) and specific surface area (220-350 m^2/g).

Monolithic SiO_2 having bicontinuous structures were discussed in this thesis. The monoliths of A2 type are weaker than the ones of A1 type which may lead to destruction of their skeleton. Low strength of A2 type makes it possible to use these only in processes with low resistance of the medium. Properties of A1 silica monoliths allow to consider it as a catalyst support for flow-through reactor in liquid-solid reactions.

References

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