NOVEL RESORCINOL-FORMALDEHYDE AEROGELS: SYNTHESIS, STRUCTURE AND FRACTAL PROPERTIES

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Organic aerogels belong to a very attractive class of aerogel materials possessing both the properties inherent to traditional oxide aerogels (high specific surface and porosity, low apparent density), and unique flexible chemical composition (e.g. the presence of various functional groups). Organic aerogels can be synthesized by polycondensation of resorcinol and formaldehyde producing so-called resorcinol-formaldehyde (RF) aerogels. RF aerogels possess high porosity, specific surface area and pore volume, as well as low thermal conductivity (for instance, RF aerogels are better thermal insulators than commercial fiber glass). On the other hand, RF aerogels are stiffer and stronger than inorganic aerogels. During the synthesis of RF-aerogels, pH of a reaction mixture, concentration and resorcinol/formaldehyde ratio, the type of gelation catalyst (acid or base), and resorcinol/catalyst ratio govern the structural characteristics (density, specific surface area, particle size, pore size distribution).

In the present work, the small angle neutron scattering (SANS), the small angle X-ray scattering (SAXS) and low temperature nitrogen adsorption techniques have been used to study the mesostructure and fractal properties of resorcinol-formaldehyde aerogels prepared by the reaction between resorcinol and formaldehyde using different solvents (acetonitrile or perfluoroacetone). Additionally, RF-lyogels were aged at various temperatures from 20 to 70°C to achieve fine-tuning of the structure of aerogel materials.

The synthesis of the resorcinol-formaldehyde lyogels was conducted using acetonitrile and hexafluoroacetone hydrate as solvents. Hexafluoroacetone hydrate plays not only a role of a solvent, but also a gelling agent for the synthesis of RF-lyogels, this approach for the RF-lyogels synthesis was used for the first time. Supercritical drying in CO2 allowed synthesizing monolithic (including flexible) RF aerogels possessing specific surface up to 400 m2/g, specific porosity up to 1.3 cm3/g. All the obtained RF-aerogels were hydrophilic.

Both the amount and type of the solvent used at the gelation stage allow varying the texture characteristics and the microstructure of the resultant RF-aerogels. In particular, the use of acetonitrile as a solvent resulted in aerogels with a highly developed surface; on the contrary, the use of hexafluoroacetone hydrate lead to the formation of macroporous aerogels consisting of submicron dense spherical particles with a virtually smooth surface. The rate of RF lyogel formation is governed by the type of the solvent used. In acetonitrile, the gel formed in several hours, in hexafluoroacetone hydrate – in a few seconds. The increase in the ageing temperature of RF-lyogels from 20 to 70°C resulted in

a significant decrease in the surface fractal dimension DS of aerogels (from ~2.5 to 2.0), which indicated a significant smoothing of the surface of the nanoparticle aggregates during the ageing process. On the contrary, the ageing temperature did not virtually affect the size of nanoparticle aggregates. A significant increase in the size of nanoparticle aggregates occurs when the gelling system is diluted (an increase in the amount of the solvent used).

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