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Parallel NMR cryoporometry, relaxometry and diffusiometry studies of porous carbon aerogels

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NMR cryoporometry, relaxometry and diffusiometry are widely used for the characterization of porous materials.[1,2,3] In this work we investigated resorcinol-formaldehyde (RF) based carbon aerogels, prepared in different ways, with all the three NMR techniques parallel in aqueous medium. This made possible the comprehensive characterization of the aerogels concerning pore size and shape, pore volume, hydrophilic/hydrophobic properties, saturation mechanism.

$T2$ relaxation times and diffusion coefficients for water in the aerogels were determined at different water contents by low field 1H-NMR methodology [1,2]. Cryoporometric measurements were carried out at several saturation states by high field 1H-NMR. The results show that the hydrophobic carbon aerogels contain hydrophilic sites, which bind the moisture of the air resulting in short $T2$ values. Spherical pores with 45 nm diameter get saturated at about 4 g/g water content according to the cryoporometry. First, pores get filled with water, and then the number of filled pores increases. At higher water contents cylindrical channels appear.

From the cryoporometric results, combining with the low-field relaxometry, we could deduce the surface relaxation time and surface layer thickness of these materials. The mechanism of pore saturation was confirmed by NMR diffusiometry showing restricted diffusion due to the pore walls and diffusion through gas phase in partially filled samples.

Different ratios of water and ionic liquid were used during the preparation process of RF hydrogels. The different compositions of solvents changed the pore size and the hydrophilic/hydrophobic character of the prepared carbon aerogels. The characterization, beyond NMR, was also carried out by means of water and nitrogen adsorption methods. The comparative analysis of the adsorption and NMR techniques could give a detailed picture for the morphology of the carbon aerogels, the effect of preparation medium and the mechanism of water loading.

References

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