Diffusion in Mesoporous Materials During Melting and Freezing

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1. Introduction

Porous solids are in the focus of scientific research due to their importance in various applications such as catalysis, mass separation, pharmaceutics, and chromatography. To date, large variety of porous materials containing both micro- and mesopores have been synthesized. Such a rapid growth of various types of porous materials consisting of several structural units with different internal porosities and pore morphologies, different spatial arrangements of which may itself give rise to quite different textural polymorphs, demands an appropriate development of experimental techniques for their structural characterization on different length scales.

One of the important properties of porous materials is connectivity of the pore space. Some attempts for its quantification have earlier been undertaken based on an analysis of gas sorption and, especially, desorption scanning behavior [1]. Alternatively, some information on the interpore connectivity may be extracted from the tortuosity factor, a parameter accessible by pulsed field gradient (PFG) NMR. Because tortuosity is assessed by tracing the molecular displacements much exceeding typical pore size of the porous medium, it does provide the degree of interconnectivity averaged over the whole structure. In this respect, new experimental approaches providing more local information, e.g. a degree in which different modes of a pore size distribution function are spatially connected to each other, are highly welcome.

2. NMR cryodiffusometry

In this work, we discuss a novel approach aiming at probing the local interconnectivity known as cryodiffusometry [2]. The method is based on simultaneous measurement of the phase state of intraporous liquids at low temperatures, using NMR-cryoporometry, and their transport properties, using NMR-diffusometry. Due to pore size-dependent feature of the freezing or melting transitions of liquids in mesopores, the effective pore space available for the liquid phase can thus be traced by varying the temperature. Consequently, by studying the diffusivities of the molecules in the liquid phase at different states of freezing or melting one may obtain some information on the organization of the pore space. The relationships between the molecular diffusivities and the phase state will be demonstrated using two model porous materials, Vycor porous glass with random pore structure and nano-structured mesoporous silicon. The results obtained experimentally will further be substantiated using computer simulations.

References

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