TRANSPORT IN NANOMATERIALS WITH A HIERARCHY OF PORE SIZES

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Introduction

Over the last several decades nanostructured porous materials have found applications in such areas as catalysis, mass separations, molecular storage and optics. Molecular transport inside nanoporous materials often suffers limitations imposed by a slow rate of diffusion in nanopores. Recent advancements in the fabrication processes have made possible a production of novel nanostructured materials with a dual size pore networks where functionally-active nanopores are connected with the surroundings by transport mesopores. This opens up new frontiers for applications of porous materials in separations and catalysis. In these applications diffusion through network of pores is often the rate-limiting process. The existence of a hierarchy of pore sizes in such materials requires substantial modifications of the contemporary models developed for transport in pores with a single size. Using *Pulse Field Gradient Nuclear Magnetic Resonance* (PFG NMR) technique molecular diffusion and correlations between movements of molecules can be studied on sub-micrometer and micrometer scales. The objective of this project is to develop a fundamental understanding of diffusion in such nano- materials with a hierarchy of pore sizes.

Experimental

The experiments are conducted using the 750 MHz NMR spectrometer located at the AMRIS facility, MC Knight Brain Institute, University of Florida. Diff60 Gradient coil manufactured by ***Bruker Biospin*** is used. Diffusion studies are carried out by applying very high gradients (up to 30 T/m). PFG NMR pulse sequences such as the stimulated echo with eddy current delay are used for carrying out diffusion measurements. Diffusivity can be obtained from a plot of the signal attenuation versus the amplitude of applied magnetic field gradients squared. A sample of porous material (i.e. zeolite powder) is placed inside a 5 mm NMR tube constricted at 1cm from the bottom. The constriction limits the possibility of unwanted movements of zeolite crystals inside the tube during the diffusion measurements. The zeolite sample in the tube is degassed under vacuum at elevated temperatures. Upon degassing the sample is loaded with guest molecules of choice (e.g. butane) at room temperature. The sample tube is flame-sealed after the loading.

Results and Discussion

So far a lot of developmental work towards understanding and optimizing the software and hardware for diffusion studies has been carried out. 2D diffusion measurements have been performed successfully for water and glycerin samples. The latter sample was used to test the possibility of diffusion measurements using ultra-high magnetic field gradients. The next step is the diffusion measurements on the actual samples. Figure 1 shows an example of the measured attenuation curve for glycerin

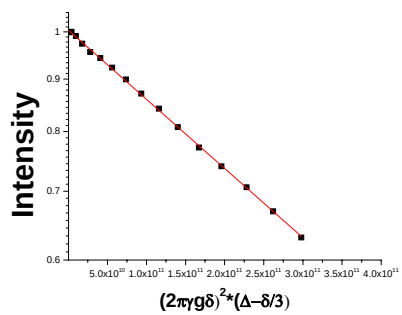

Fig.1. Example of the PFG NMR attenuation curve for glycerin measured by the stimulated echo sequence using 750 MHz spectrometer.

Conclusions

Perfect mono-exponential fit results for glycerin and water with the correct diffusivities have been achieved. We are now ready for experiments using actual samples.

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