

Nuclear Magnetic Resonance Dipolar Cross-Relaxation Interaction between Nanoconfined Fluids and Matrix Solids

Jin-Hong Chen,* Chao Liu, Stacey M Althaus, and Mohammed Boudjatit



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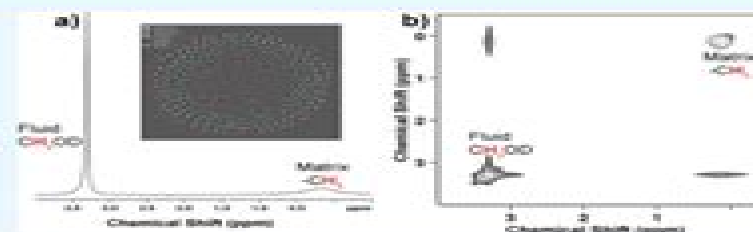


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ABSTRACT: Many different methods have been developed to investigate fluid–solid interactions in nanoporous systems. These methods either only work in the liquid phase or provide an indirect measurement by probing the fluid–solid interaction based on a measured property change of the fluid or solid under different sample conditions. Here, we report a direct measurement technique using NMR dipolar cross-relaxation between the nanoconfined fluids and the matrix solids. The method was tested using a methyl-functionalized mesostructured silica saturated with methanol as a model sample. A formal theory was established to describe the enhanced dipolar cross-relaxation interaction between the nanoconfined fluids and the matrix solids. Both the experiment and theory showed that nanoconfinement of the fluids enhances the dipolar cross-relaxation interaction between the fluid and the matrix solids, which can be applied to investigate the fluid–solid interaction for various materials of a similar nanostructure.



INTRODUCTION

Fluid–solid interactions are ubiquitous in natural and manmade porous materials. The interfacial interaction between a fluid and a solid significantly influences the properties of the solid as well as the storage and transport of the fluid.^{1–3} This interaction is of special importance in nanoporous materials because of the large interfacial areas and high surface-to-volume ratios.⁴ Methods to quantify the fluid–solid interaction thus have broad applications in many different nanomaterials including synthesized mesoporous silica nanoparticles, carbon nanotubes, metal–organic frameworks, and natural nanomaterials, such as zeolites, shale, etc. Moreover, materials with significant fluid–solid interactions have major economic impacts on hydrocarbon extraction,⁵ energy storage,⁶ and radioactive waste disposal.⁷ In shales and coalbeds, the capacity for natural gas extraction^{8,9} and greenhouse gas sequestration¹⁰ is determined by the gas adsorption and transportation through the nanopores embedded in their matrices. Thus, there is a great need to understand and quantify the interfacial interaction in many of these nanoporous materials.

Many different methods have been developed to investigate the fluid–solid interaction, such as isothermal titration calorimetry (ITC),^{11,12} surface adsorption,^{13,14} neutron or X-ray scattering,^{15,16} and zeta potential analysis.¹⁷ ITC is a very useful measurement of the thermodynamic parameters of interactions, but the method only works in solution. Most other measurement methods are indirect and probe the fluid–solid interaction based on the measured property change of the fluid or solid under different sample conditions. Analytical and

computational methods are also widely used to study the fluid–solid interaction.¹⁸

Nuclear magnetic resonance (NMR) can directly measure the fluid–solid interfacial interaction using the information from nuclear spin dipolar coupling. Dipolar coupling of nuclear spins is particularly important in magnetic resonance spectroscopy and imaging because it depends on distance, orientation, and dynamics.¹⁹ The net dipolar coupling in solid-state NMR experiments provides a direct approach to extract information about structures and dynamics²⁰ and has been used to study interaction of fluid molecules on the solid surface.^{20,21} Dipolar coupling in isotropic fluids averages to zero as a result of molecular Brownian diffusion and becomes a key relaxation mechanism.²² The dipolar cross-relaxation, also called the nuclear Overhauser effect (NOE), has been the most important and extensively used NMR method in determining structure with molecular dynamics, especially for macromolecules such as proteins and nucleic acids.²³ NMR has also been widely applied to study porous materials, mainly by measuring nuclear spin–lattice (longitudinal) relaxation time T_1 ^{24,25} and/or spin–spin (transverse) relaxation time

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