EDITORIAL

Focus on the physics of magnetic resonance on porous media

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Abstract. Porous media are ubiquitous in our environment and their application is extremely broad. The common connection between these diverse materials is the importance of the microstructure in determining the physical, chemical and biological functions and properties. Magnetic resonance and its imaging modality have been essential for noninvasive characterization of these materials in the development of catalysts, understanding cement hydration, fluid transport in rocks and soil, geological prospecting and characterization of tissue properties for medical diagnosis. This focus issue highlights recent NMR/MRI technical development, the underlying physics associated with the new technology, as well as novel applications of the technologies.

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1. Introduction

Porous media are ubiquitous throughout modern life, both in nature and the man-made environment, including sedimentary rocks, soil, plants and wood, construction materials, food products, many engineered materials such as paper and catalysts, and also biological tissues...
such as trabecular bone and the lung. Their application covers the broadest scope of human activities, including agriculture, mining and petroleum exploration, the food industry, earth and planetary sciences, chemistry and medicine. The functions of these materials are often determined significantly by their complex internal structure, which can span length scales of many orders of magnitude. These materials are intrinsically heterogeneous, consisting of at least two phases, often one solid and the other liquid, such as fluid-saturated rock and soil. The properties of the interstitial fluid can be very important, either because of their own value, or because of their impact on the overall performance of the materials. Magnetic resonance (MR or nuclear magnetic resonance (NMR)) has found a unique application in the study of these materials through its intrinsic sensitivity to molecular dynamics, and through the technique of imaging (MRI), to explore a larger range of length scales and phenomena in such systems. In the past two decades, there have been significant advances in many scientific disciplines, and considerable commercial development of MR instrumentation and applications for petroleum exploration.

The key insight responsible for the wide application of MR to porous media is that the surfaces of the porous space have a strong influence on the relaxation and diffusion properties of the interstitial fluids. As a result, the relaxation and diffusion can be used to obtain surface-to-volume ratio, a key structural parameter to characterize porous size, and fluid flow properties (such as permeability). The mechanism for surface relaxation has been the topic of many studies (e.g. [1–3]) and continues to be a focus of the field.

MR instrumentation is another area where the past decade has witnessed tremendous innovation. It is no longer a technique limited to the laboratory. The traditional high magnetic field and high field homogeneity system is restricted to measuring small samples inside the magnet. The fundamentally different magnet geometry using permanent magnets allows the inside-out MR system, so that MR measurement on large immovable samples becomes routine. This development has given rise to the MR well-logging industry for geological prospecting, and the quantitative study of art and historical objects, building, bridges and tunnels for non-invasive monitoring and archeology. Also, instrumentation for the technique of surface NMR (SNMR) using the Earth’s magnetic field has seen tremendous development in recent years and it is now a viable technique for exploring shallow water aquifers [4].

One significant technology shift in the past decade is the wide adoption of the two-dimensional (2D) and multi-dimensional (MD) MR methodology for relaxation and diffusion [5]. Conventional NMR techniques measure one parameter (e.g. $T_1$, $T_2$, $T_1\rho$ or diffusion constant $D$) in a single experiment. The MD-MR experiment acquires 2D or multiple dimensional data matrices and uses 2D (or MD) Laplace inversion to obtain the correlation function. This method significantly enhanced the spectral resolution of relaxation and diffusion and has been applied in many research laboratories and commercially in well-logging. Many specific techniques have been developed in recent years and several papers in this focus issue continue this trend of using 2D MR to study the pore structure.

Research in porous media is inherently multi-disciplinary and couples advances in physics, chemistry and engineering with a broad range of applications. Even though this focus issue primarily discusses the basic technical and scientific development, application is prominent in motivation, research direction and solution, as it is the purpose of this issue to connect the basic research with such diverse applications.
NMR is the study of the dynamics of the nuclear spin system and the interactions between the spins within the system and other molecules, electrons and the external environment. The spin-carrying molecules may exhibit the complex dynamics of the host materials, such as rotation, translational motion and segmental dynamics. These dynamics are some of the mechanisms for spin decoherence, relaxation and diffusion.

Conventional MR relaxation and diffusion techniques measure one parameter (e.g. $T_1$ and $T_2$, $T_{1\rho}$ or diffusion constant $D$) in a single experiment. For example, one of these parameters, $T_2$, is typically obtained by executing a series of NMR experiments with changing relaxation time period, $\tau$, and measuring signal as a function of $\tau$. For a single species of spins, the signal ($m$) decays exponentially, $m \propto \exp(-\tau/T_2)$, where $T_2$ is the relaxation time constant. For mixtures with components of different parameters, the total signal will be a superposition of the components. For example, in a fluid mixture of different molecules, the smaller molecules often exhibit faster translational and rotational diffusion, and thus longer $T_2$. Thus, the distribution of $T_2$ or $D$ can be used to quantify molecular weight distributions (e.g. [6, 7]). In porous media, $T_2$ is found to be enhanced by surface relaxation and thus it has been used to measure pore sizes (e.g. [8, 9]). However, in a more complex sample, such as a porous rock saturated with a mix of water and oil, the relaxation time constant of water and oil may overlap due to a combination of oil molecular size and surface relaxation.

The MD-MR technique improves the resolution of different molecules by measuring the correlation function of two or more parameters, such as $T_1$–$T_2$ correlation, $D$–$T_2$ correlation, $D$–$D$ correlation, etc. Compared to the conventional relaxation and diffusion techniques, MD-MR is performed by varying two or more experimental parameters (such as time periods, magnetic field gradients, RF pulses) and acquiring MR signals as 2D or higher-dimensional data matrices. Laplace inversion of such MD matrices is used to obtain the multi-parameter correlation functions. The MD correlation spectrum allows greater separation of signals from different components, and helps one to identify different phases and observe changes of the composition and physical properties.

This type of correlation experiment was first reported in the 1980s to study the multiple components of chemical and biological systems [10]. Even though the NMR experiments of this type could be performed on the NMR spectrometer at the time, data analysis was a considerable barrier due to the requirement of significant computing power [11, 12]. In early 2000, a new algorithm was developed to rapidly perform the MD Laplace inversion without the huge memory requirement so MD NMR data could be analyzed on regular desktop computers and laptops [5]. This development lead to a rapid expansion of the MD methodology in chemistry, material science, food science, and has since been used commercially in the oil and gas industry.

In addition to enhancing the spectral resolution, 2D experiments may be able to identify unusual diffusion dynamics. For example, when diffusion is not fast enough, signal decay of a uniform system can become multi-exponential. In a 1D experiment, the $T_2$ spectrum will be broadened; however, it is difficult to identify the cause of the broadening (either slow diffusion or multiple pore sizes) from a 1D experiment alone. It was proposed experimentally [13, 14] and theoretically [15] that a 2D experiment might be able to identify the slow diffusion regime and quantify diffusion flux between different compartments of the pore space. Two papers in this issue [16, 17] continue to explore the use of 2D NMR to study exchanges between different pore spaces. In [16], a packing of porous zeolite particles was used to simulate the dual-porosity...
rocks commonly found in carbonate formations. The pore size inside the grains is 0.8 nm and the grain size is 2000 nm. The double stimulated echo experiment was used to measure the $D-D$ correlation map. The diagonal signal on the map is interpreted as the population of molecules in each porosity while the off-diagonal peaks are considered to be the molecules diffused from one regime to another during the experiment.

In such a 2D experiment, the off-diagonal peaks are found to be very small, e.g. a few percents of the diagonal peaks. It is also difficult to quantify the uncertainty of such a result due to the nonlinear nature of the Laplace inversion. The paper by Fantazzini et al [18] addresses a similar issue by examining a 1D experiment of tissue in which the water dynamics exhibits an exchange between different pools. They show that under certain conditions, the $T_1$ recovery exhibits a nontrivial negative amplitude that is argued to illustrate the exchange of water molecules between two pools. Even though this method is applied to 1D data, a similar approach should be useful for 2D analysis.

3. The physics of surface interactions and applications

The idea of using spin relaxation to characterize pore size originates from the need in petroleum exploration to quantify pore sizes and rock permeability [8], and was later developed for general porous media [9, 19–21]. This idea is the basis of MR well-logging and the range of NMR applications to address complex geology and multi-phase fluids in pore space has been constantly expanding. Two papers [22, 23] in this issue are an example of the expansion of these ideas to address the specific issues in the petroleum exploration.

Even though the concept of surface relaxation is well known and has been used for many years, the physical origin and mechanism are still a topic of current research. Torrey et al [1, 2] considered dilute magnetic ions the main cause of spin relaxation. Such surface relaxation has been directly measured by drying the fluid to only a monolayer on the solid surfaces [24], showing a significant frequency dependence of the relaxation process. Systematic measurement of the field dependent $T_1(\omega_0)$ ($\omega_0$ is the Larmor frequency) of fluid saturated porous media [25–27] show anomalous diffusion dynamics on the grain surface to be characteristic of the Lévy walk. The paper by Korb [28] provides a timely review of the subject and it shows that such surface–fluid interaction measured by field cycling MR could be relevant to the fluid flow properties.

The hydration process of cement [29] has been studied by NMR for many years (e.g. [20, 30]). The spin relaxation has been used to identify water in different pores within the cement microstructure. For example, water adsorbed in the nanopores (C–S–H gel) of cement paste typically exhibit very short $T_2$ relaxation and a very large $T_1/T_2$ ratio. Such a large $T_1/T_2$ ratio is typically encountered in the NMR of solid materials, where $T_2$ is dominated by the large static dipolar coupling of spins and $T_1$ is long because of the lack of molecular dynamics at the higher Larmor frequencies. It indicates that the water in hydrated cement exhibits slow dynamics and one may apply solid state NMR techniques to identify such water species and monitor their kinetics during hydration. The paper by Rodin et al [31] applies double-quantum filter (DQF), a commonly used solid-state NMR method, to study the pore geometry of cement paste. For the water in CSH, the proton–proton dipolar interaction is not completely averaged out by the slow motion. For water in the larger pores, the proton–proton coupling is essentially zero. As a result, DQF NMR provides a new method for differentiating different water populations and their kinetics during hydration.
Hydration water surrounding biomacromolecules is essential for the function of the biomolecules, folding and secondary structures, dynamics and hydrophobic interactions. The water molecules in the hydration layers exhibit much slower dynamics, similar to the hydration water in cement, so that the spin–spin interactions can be enhanced. The paper by Ortony et al [32] discusses the use of the interaction of an electron spin label on the macromolecules and water proton in order to probe the water dynamics within the hydration layer. Similarly, Gratz et al [33] reports on a study of the diffusion of adsorbed small molecules in a metal-organic framework, which is of interest as a gas storage medium for energy application.

One area of rapid development in recent years is the numerical simulation of NMR properties in porous media from 3D structural images obtained by high-resolution x-ray tomography. This is owing in large part to the sophisticated and commercially available x-ray tomography equipment with resolution down to microns. Earlier simulations focus on transport properties and NMR relaxation and diffusion [34–36]. More recent work [37, 38] has explored simulation of other NMR experiments such as a 2D MR. Arns et al in this issue [39] focuses on both 2D NMR and the internal field distribution naturally occurring in such inhomogeneous materials. With the continuous advance in x-ray CT technology in resolution, imaging size and computation power, simulation, as well as direct solution of the diffusion problem [40] could become a commonly used method for in depth understanding of petrophysical measurements.

4. Biomedical application

Biological tissue represents another class of porous materials with complex internal structure and function. The physical structure is critical to many aspects of the biological and physiological functions of tissues in addition to their biological and chemical properties. Trabecular bone is a good example in that the geometrical properties of the porous bone structure is known to be critical to its mechanical strength and can be used as a diagnostic parameter for osteoporosis and possibly other bone metabolic diseases (for example, see osteoporosis in http://www.wikipedia.org). Another good example is the lung, where the hierarchical structure of the bronchial tree allows air to be in contact with the high surface area of alveoli in order to facilitate the exchange of carbon dioxide and oxygen. The ability of the lung tissue for gas flow and exchange is a key diagnostic for emphysema and related lung conditions. In recent years, $^{129}$Xe MRI has contributed significantly to our understanding of the gas exchange in lung tissue (e.g. [41–43]). In this issue, Patz et al [44] developed a quantitative model for gas exchange in alveoli and an MRI experiment based on optically polarized $^{129}$Xe technology to obtain key physiological parameters of lung tissue and function. The MRI development and the physical models of gas exchange represented by this paper and the related work from the xenon MRI community have significantly advanced the understanding of lung physiology and techniques for diagnosing lung diseases.

5. Surface nuclear magnetic resonance

Most NMR systems are used to examine small samples (e.g. <1 ml). In contrast, SNMR (or sometimes called magnetic resonance sounding (MRS)), developed in the 1980s [45] is a method of examining a sample up to 100 m [4, 46]. This technique uses the Earth’s magnetic field as the static field and deploys a large coil (up to 100 m) at the surface of the ground to
generate the oscillating magnetic field for spin excitation and detect the signal from the water body underneath the coil. In practice, such measurement can be very challenging because the large loop coil also receives ambient noise from many sources, such as nearby power lines, electrical machinery, ground current due to lightning and storms even miles away. These noises are typically broadband and non-stationary and difficult to filter out in the frequency domain. In recent years, it has been shown that secondary coils can be placed away from the main NMR coil in order to pick up the interfering noises. Digital post-processing combines the signals from several coils to eliminate the noise from the NMR signal [47, 48]. This technique has been shown to effectively remove the interference from such sources and allows routinely successful SNMR measurement.

Compared to the sophistication of conventional NMR/MRI, SNMR only uses a very limited set of pulse sequences due to the current limitation of its instrumentation and mobility requirement. For example, SNMR experiments mostly detect the free-induction-decay after one excitation pulse. Two-pulse sequences have been recently used in magnetically heterogeneous formations in order to measure the spin-spin relaxation time [49] or $T_1$ [50, 51]. Development of further pulse sequences will undoubtedly improve the measurement and interpretation. One important direction for SNMR is to understand and quantify the uncertainty of SNMR measurements because it is difficult to compare SNMR measurement results with the true values (e.g. porosity) of a 100 m size sample. The paper by Müller-Petke et al [52] is a good step in this direction.

6. Conclusions and outlook

Magnetic resonance of porous media has seen rapid progress in recent years in basic physical and chemical understanding, instrumentation, NMR methodology, numerical techniques, and the range and depth of applications [53, 54]. The interaction of scientists from different disciplines has contributed to the exciting development of the field. Commercial application in the oil and gas industry has been a significant stimulus to MRPM research and there is still an urgent need for novel techniques to better characterize hydrocarbon-bearing formations as well as other parts of oil production. The breakthrough in SNMR instrumentation will allow rapid adoption of more advanced NMR techniques for efficient exploration of shallow aquifers. The mobile NMR sensors have already been used to examine art and historical objects and medical diagnostics (e.g. [55]). There is much evidence that this trend will continue.

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