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Communication

Imaging of multiphase fluid saturation within a porous material via sodium NMR

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ABSTRACT

We present in this paper a method to monitor multiphase fluid core saturation through measurement of the sodium NMR signal. In a rock core saturated with water and oil, sodium will be present only in the water phase, and therefore can be used to separate the two fluids. Two dimensional sodium images were taken to monitor the movement of brine into oil saturated rock cores. The measured fluid exchange agrees well with expected behavior from traditional core analysis methods. Indications of damage to the rock structure can be seen from the patterns of fluid imbibition.

1. Introduction

Laboratory characterization of rock cores is important in making decisions regarding development and utilization of hydrocarbon reservoirs. The methods used to calculate rock properties, such as capillary pressure or relative permeability, require accurate quantification of the fluids present in the core at the end of experimentation. The amount of different fluids within the core is usually determined by monitoring the amount of expelled fluid during experimentation; however this method is prone to error. Fluids can be lost during the handling of cores, particularly in very permeable samples, or strongly wetting samples can unknowingly imbibe fluid during experimental setup. Two distillation techniques, the Dean Stark and the Karl Fischer methods, are often used to confirm the amount of oil and water within the cores. As these techniques remove the fluids present within the core, if multiple experiments are planned, these techniques can only be performed at the end of all experimentation and not at intermediate steps.

Proton NMR is commonly used in porous media characterization [1–6] as the technique is noninvasive and can be used on opaque samples. While this method is useful when only a single fluid phase occupies the rock core, it is less straightforward when multiple phases are present. The internal magnetic fields present within porous materials makes separating the signals of the oil and water phases nontrivial [7]. Due to the usefulness of separating the two signals, many techniques have been developed to attempt to individually monitor the oil and water within a porous material. Some methods use the diffusion coefficient to attempt to distinguish between oil and water [8–12]. While these techniques work well in some situations, depending on the fluid diffusion coefficients and material wettability, the results can be ambiguous in other situations [13]. Other techniques to separate oil and water signals, such as magic angle spinning [7,14] or the developing field cycling method [15], are not practical on large samples like the standard rock core sizes. Another common technique is the substitution of water with deuterated water [16–19]. However, the volumes of fluid often needed in core analysis make this a prohibitively expensive technique, as well as changing the water's properties. This technique also has the disadvantage than some oils will exchange hydrogen atoms with deuterium, giving rise to signal in the water phase.

We take an alternative approach to separate the two fluids. The brines found within oil reservoirs are typically very salty, containing in the range of 100–250 kppm NaCl. For comparison, sea water contains is approximately 35 kppm NaCl. These concentrations are much higher than the levels of sodium observed in biological NMR studies, which are approximately 10–50 times lower. Due to the non-polar nature of oil, the concentration of salt in the oil phase will be so small as to be considered non-existent. We develop a new application of sodium imaging in porous materials, using the sodium ion as a naturally present tracer in the brine phase. While work has been previously performed to image sodium within rock cores [20,21], we present the first example where sodium is used to separate fluid components in a two-phase system of oil and water.

As the sodium signal is relative to the amount of sodium present in the brine [20] and the concentration of salt in the reservoir brine is known, the amount of fluid can be determined from

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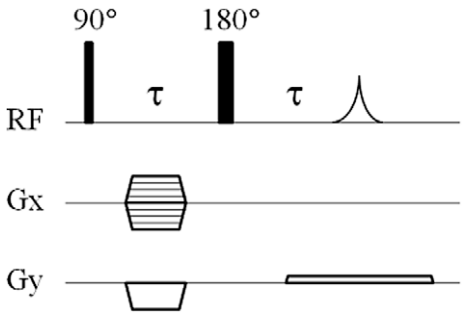

Fig. 1. 2D spin echo imaging pulse sequence.

measuring the sodium intensity. Though sodium has a lower sensitivity than hydrogen, its high abundance and short T1 means that a signal of reasonable intensity can be acquired in comparable time. In addition to providing a bulk measurement of fluid within the sample, similar to the Dean Stark and Karl Fischer methods, NMR also has the added advantage of that it can also be used to provide a spatial distribution of the different fluids throughout the core. This makes it similar to X-ray CT or gamma scan techniques [22]. These methods, however, require doping of one of the fluid phases to provide contrast for the images. Common dopants, such as cesium, can be expensive, often difficult to remove from the cores after experimentation and highly corrosive to equipment. In contrast, sodium imaging relies on a naturally present constituent in the system.

2. Materials and methods

Core plugs of 1 inch diameter and 30 mm in length were bored from Bentheimer sandstone. Bentheimer is known to be a homogenous sandstone containing only a small amount of clay and impurities. The cores were then cleaned by the immersed soxhlet method in a mix of toluene and methanol. The samples were dried to a constant weight in an oven and saturated with Isopar L mineral oil under vacuum. Pore volumes were calculated from the change in weight between the dry and saturated core and the density of Isopar L. Bentheimer, like many sandstones, is a water wet material, meaning that the surface prefers to be in contact with water over oil. When a Bentheimer core saturated with oil is placed in brine, the core will take in the brine and expel the oil, a process called spontaneous imbibition. To monitor this process, the cores were placed in 100 kppm brine and allowed to imbibe the brine for a set period of time. The cores were then removed from the brine, excess brine removed from the surfaces, wrapped in plastic film to prevent evaporation, and then imaged. Images were acquired for 10, 20, 40, 60, 120, 240, and 960 min imbibition times. The NMR experiments were performed on a Bruker 400 Avance II spectrometer with a home-built sodium saddle coil tuned to 105.8415 MHz. The images were acquired using a standard 2D spin echo imaging sequence, shown Fig. 1. The images are a projection along the length of the core as slice selection was not used. Image resolution was 256 points along the read axis with 32 points in the phase direction. The field of view was 62 mm by 47 mm. Pulse durations were 60 μs for the 90° pulse and 120 μs for the 180°. Time of repetition was 200 ms. The spin echo imaging sequence has a low duty cycle and despite the short repetition time, no sample heating was noted. Half echo time τ was 2.1 ms. Total measurement time was 25 min for 256 scans.

Due to the conductive nature of sodium, RF penetration is often a problem at high fields. The RF pulse is dissipated as it moves into the sample, such that only the spins on the outside of the sample are excited. Images taken of salt water alone show strong skin effects. At 105.84 MHz, the skin depth in 100 kppm NaCl brine is only 13 mm [23]. The RF attenuation is much less of a problem in rock cores, where the electrical conductivity of the sample is significantly less than the bulk fluid. The pores in our sample are also much smaller than 13 mm. Therefore, to calibrate the amount of sodium to the measured NMR signal, the sodium signal from a Bentheimer core of known pore volume fully saturated with 100 kppm brine was measured.

3. Results and discussion

The core fully saturated with 100 kppm brine, Fig. 2a, shows a fairly even profile, though minor skin effects are still seen. The initial image of the core saturated only with Isopar L, Fig. 2b, shows no signal as expected. The images of the spontaneous imbibition process, Fig. 3a–g, present a curious behavior. Initially, the samples show inflow of brine into the cores from both the sides of the core and the ends of the core, which was expected. However, as the cores were allowed to imbibe longer, the brine appeared to cease to travel further into the core from the sides and only enters the cores from the ends. 1D projections of the images along the *x*-axis, Fig. 4, show that the amount of brine entering the cores from the sides remains nearly constant after 40 min while the influx of brine from the ends of the core continues. Experiments on several different cores showed minor variations in the amount of fluid which entered the cores

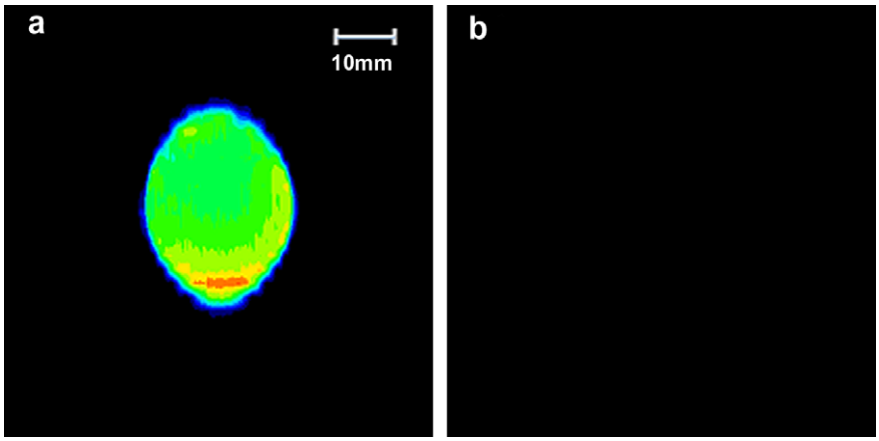

Fig. 2. 2D axial images of a Bentheimer core (a) fully saturated with 100 kppm brine (b) fully saturated with Isopar L.

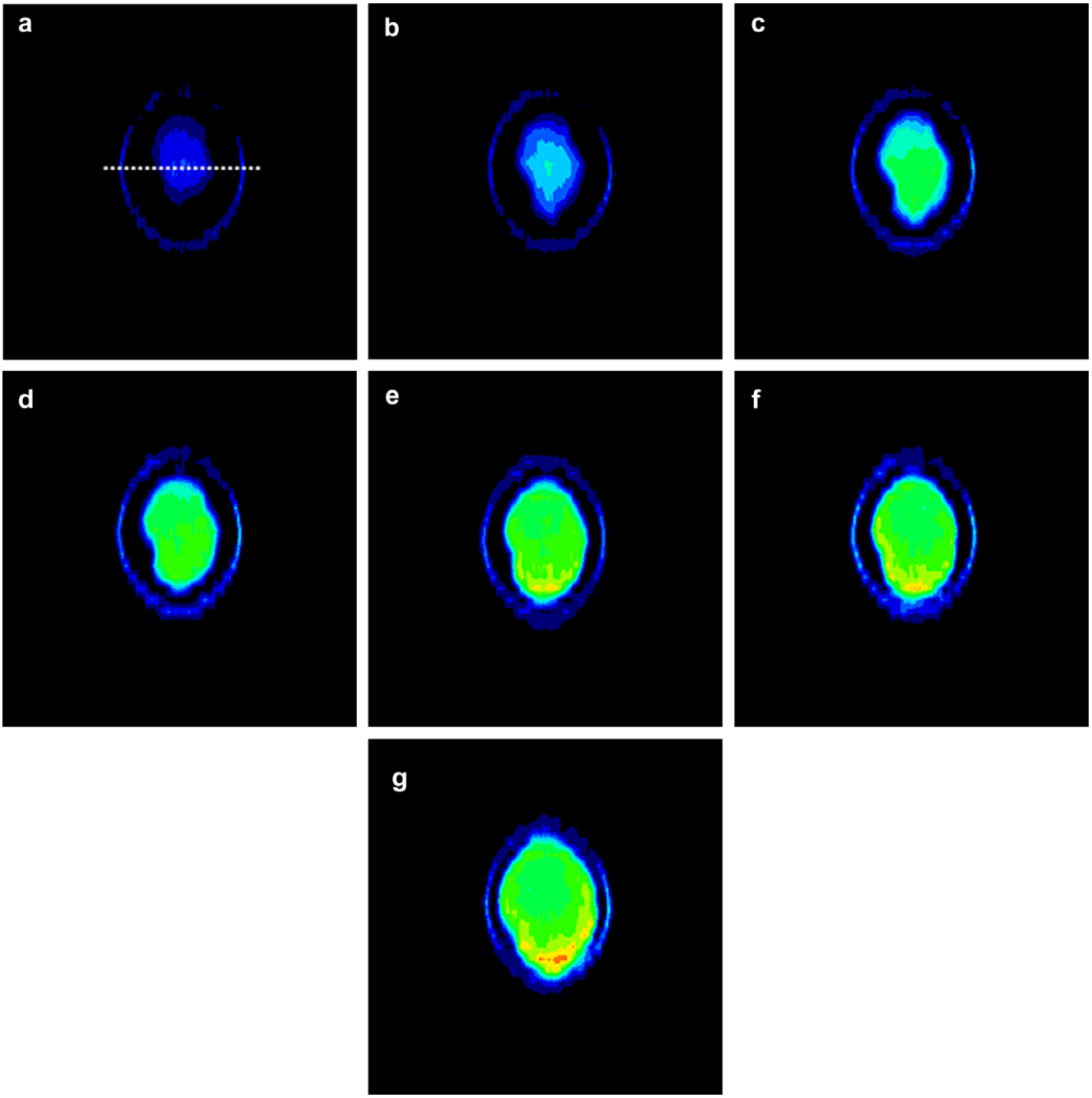

Fig. 3. 2D axial images of a Bentheimer core (a) 10 min brine imbibition (b) 20 min imbibition (c) 40 min imbibition (d) 60 min imbibition (e) 120 min imbibition (f) 240 min imbibition (g) 960 min imbibition. The dotted white line in (a) shows the location of the 1D projections in Fig. 4.

but the imbibition pattern was the same for all the cores. Average signal to noise in the images was approximately 100.

We believe this behavior is a by-product of rock formation damage caused by the coring procedure of the plugs, as other studies on spontaneous imbibition show movement of fluid into the cores from the sides [16]. The process to bore out the cylinders from the outcrop rock used a viscous mud to lubricate the drilling. In this process, if an insufficiently high oil pressure is used, a significant amount of rock dust can be pushed into the pores, clogging them and hindering fluid movement into the core. The method to cut the cores to a given length is a dry technique and less likely push dust into the pores. Preliminary results from further research show more uniform patterns of fluid uptake in cores that have been cleaned using pressurized air to remove dust from pores before saturation with Isopar L. Microfracture may also be responsible for the non-uniform fluid imbibition.

The sodium signal intensity for a core at the different imbibitions times were integrated and converted into fluid volumes. These values were plotted as a function of imbibition time, shown Fig. 5, and exhibit plateau behavior. For water wet samples, initially the core will rapidly expel oil, much of the loss often being in the first hour. The production then slows down with time. This is in agreement with behavior seen in standard core analysis methods. The final NMR measurement shows the amount of imbibed brine fills approximately 64% of the core. This is also in line with tradition core analysis measurements of water wet systems, which

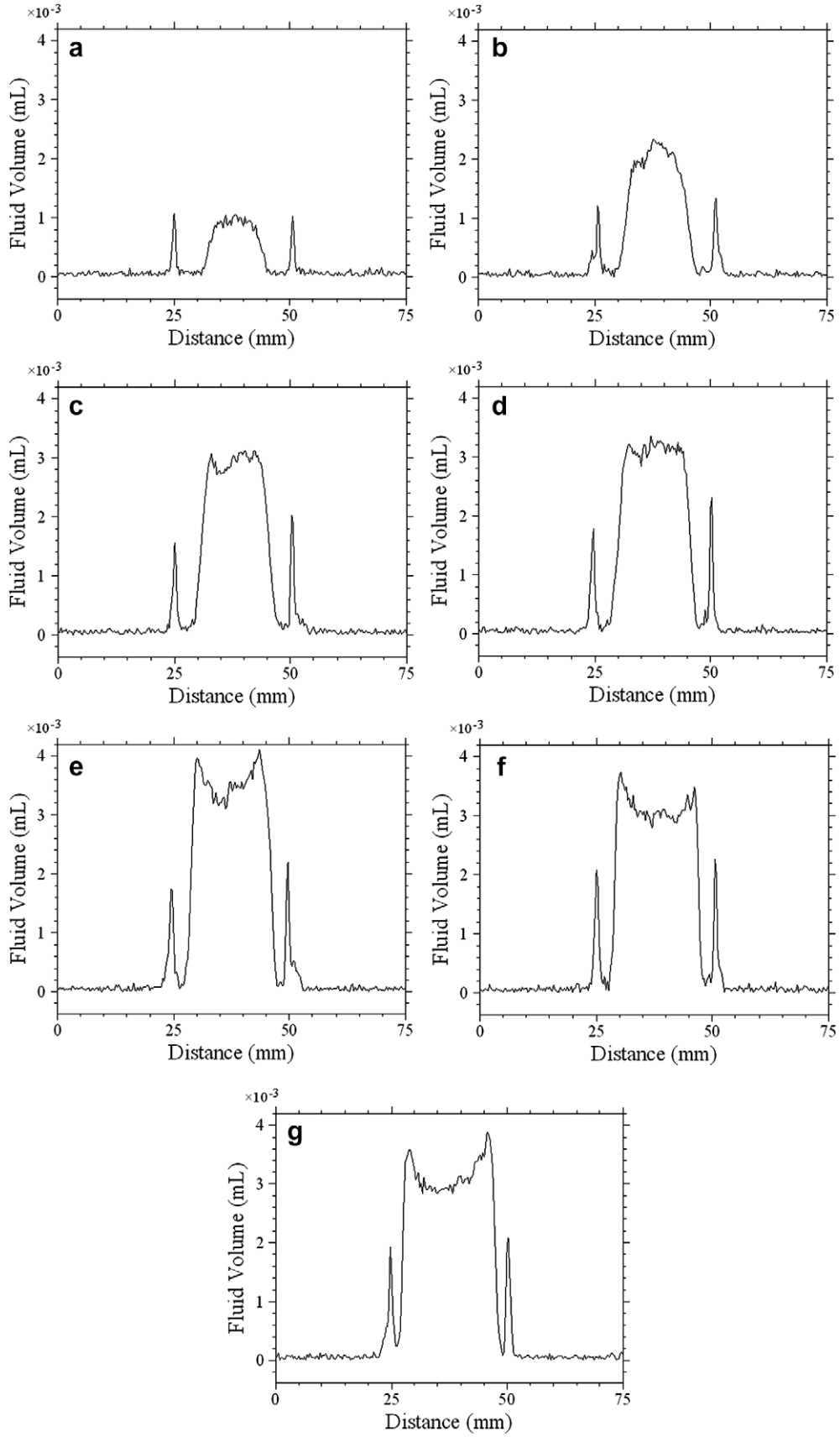

Fig. 4. 1D projections along the *x*-axis in the middle of the Bentheimer core after (a) 10 min brine imbibition (b) 20 min imbibition (c) 40 min imbibition (d) 60 min imbibition (e) 120 min imbibition (f) 240 min imbibition (g) 960 min imbibition.

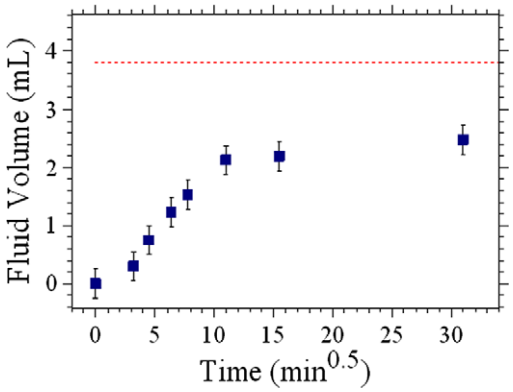

Fig. 5. Plot of brine volume in the core versus time. Note, time is plotted in the square root of minutes for viewing ease. The dashed line indicates the total pore volume of the core.

will often imbibe brine into approximately 60–80% of the pore volume. These initial results are promising for quantitative use of the technique. There is a concern, however, that some sodium ions may be absorbed at the pore surfaces or in the clays, leading to additional sodium signal present in the core not in the liquid phase. Continued investigation will look into the extent that this behavior occurs and ways the signal could be suppressed, potentially allowing the use of the method in cores with a high clay content. We are also in the process of obtaining the necessary equipment for larger scale validation of the technique upon standard 1.5 inch diameter rock cores. Future studies are planned at lower frequency to avoid the skin effects and using centrifuge logging to monitor expelled fluid as a volumetric technique is a superior method to mass balance for external validation in an oil water system.

The applications of this technique to petrophysics beyond simple saturation control are wide reaching. Using sodium as a tracer for imaging would provide an easy way to monitor the movement of the oil and water during flooding experiments. A recently developed technique to obtain capillary pressure from NMR imaging [24] would be more practical for routine use without the need to use heavy water to distinguish the two fluid phases. After a petrophysics measurement, often the core is allowed to equilibrate for a given time to allow capillary forces to evenly redistribute the fluids throughout the core before the next measurement. Imaging of the core would allow an easy check that an even fluid distribution had indeed been obtained. Additionally, imaging the sodium distribution could be used in formation damage studies to observe invasion of drilling fluid into the rock or, as the results of this study indicate, to detect how damage to the rock matrix alters fluid movement.

4. Conclusion

Measurement of the sodium signal can be used to monitor the movement of oil and water in porous materials where the internal gradients do not allow separation of the proton signals in the spectral domain. While working at 105 MHz presents the advantage of high sensitivity, due to the conductivity of sodium, future work will benefit from being performed at a slightly lower field to minimise RF skin effects. Loss of signal sensitivity at a lower field could be recouped through a more efficient, fast imaging sequence. Use of a single point imaging pulse sequence such as SPRITE [25] would allow faster image acquisition without the concern of T2 weighting seen in other fast imaging sequences. The NMR results show good agreement with expected fluid behavior seen in traditional core analysis methods, though larger scale studies with a variety of external measurements are required to thoroughly validate its use as a quantitative technique. The technique presents significant potential for saturation control in rock cores as well as a new method for separating the behavior of oil and water in two-phase NMR porous media research.

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