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Mini Review

NMR Spin-Lock Determination of Viscosities of Oils in Rock Pores

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Abstract

We have made the first steps in validating the application of the NMR spin-lock T1ρ sequence for the determination of the calibrated viscosities of oils in geological porous rocks. As an initial stage, we have measured the T1ρ relaxation times for four oils from Brookfield Engineering, of given calibrated viscosity. That work will be published in detail elsewhere. Here we are applying these calibrated measurements to measure the T1ρ values of the same oils in a porous sandstone rock. We have measured the T1ρ spin-lock relaxation time for one Brookfield Engineering calibrated viscosity oil, both in the bulk and when it is adsorbed into a sandstone rock. We intend to extend this work to a number of carbonate rocks and shales in the future, and also to 10mm diameter samples, in a compact specially designed compact NMR Spectrometer (Figure 1) to which we are adding an NMR Probe with a useful variable-temperature capability, to give information on the rock pore-dimensions, when there are liquids in the pores.

Introduction

NMR is a powerful non-invasive method for studying the properties of materials. In particular, it can study liquids and tar properties in porous materials such as geological rocks. Common NMR measurement sequences measure the T1 longitudinal relaxation and T2 transverse relaxation times, which are used, among other methods to study rocks.

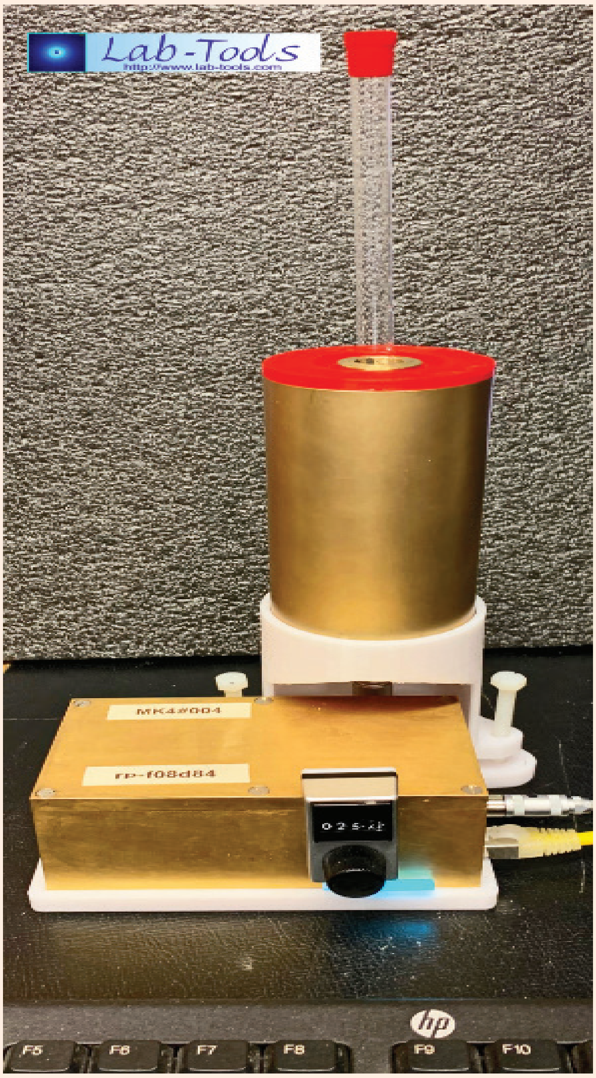

Figure 1: Lab-Tools MK4 NMR Time-Domain NMR Spectrometer with a 10 mm OD NMR sample. The NMR Probe is being converted to have a variable-temperature capability, suitable for this work.

NMR Fast Field Cycling probes the molecular motion at the T1 rotational frequency [1,2], and typically covers a frequency range from 10 kHz to some 10 s of MHz. The NMR spin lock sequence T1ρ probes the molecular dynamical motions in a solid or liquid, at twice the T1ρ rotational frequency ω_1

$$\frac{1}{T_{1\rho,DD}} = \left(\frac{\mu_0}{4\pi}\right)^2 \frac{3\gamma^4\hbar^2}{10\pi^2 r^6}\left(\frac{3}{2}J(2\omega_1)+\frac{5}{2}J(\omega_0)+J(2\omega_0)\right)$$

We typically cover a frequency range of 1 kHz to 30 kHz. Here we are applying T1ρ to determine the spectral densities, and hence give information on the viscosity of viscous liquids. In particular, in this work, we are starting to probe the calibrated viscosity of oils and possibly tars in geological rocks. Previously we have measured the T1ρ relaxation times for four oils from Brookfield Engineering, of given calibrated viscosity. Here we have measured the T1ρ spin-lock relaxation time for a Brookfield Engineering calibrated viscosity oil in a sandstone rock. There are a range of reasons why the viscosity might not be the same as in the bulk liquid including pore-surface relaxation, and tortuosity effects.

So, the question is, how closely can we determine the true viscosity (which we know fairly well in the bulk) for this oil in this sandstone rock, using the NMR spin lock T1ρ sequence? The measurement procedure has been to take a weighed dry sandstone chip (British Geological Society source), diamond sand it to be of a suitable size to go into a 5 mm OD NMR tube (112 mg), and measure the 1H proton signal the NMR Spectrometer so as to confirm that there is negligible NMR signal. Brookfield Engineering viscosity standard B1060 was used for this measurement, which has a calibrated viscosity of 1042 cP at 25C (1% accuracy). Then the sample was placed in the calibrated viscosity oil, overnight. In the morning, the chip was rolled on a non-adsorbent surface, to remove excess surface liquid, and re-weighed, to determine the oil uptake: 6.9 mg.

The T1ρ relaxation time at a W_1 rotation frequency of 10 kHz, and at a sample temperature close to +25C was measured in the bulk as 12.6 ms, and in the pore as 14.7 ms. The temperature variation of the viscosity in the pore needs to be determined – this can be done, as the NMR Spectrometer that was used has a wide-range variable-temperature probe This rotational rate gives information on the relevant molecular motion in the oil in the pores. It is too early to reliably say how different the measured viscosity in the pore is, but it is not significantly different to the value we measured in the bulk, with our derived calibration.

There are a range of good reasons why the viscosity might not be the same as in the bulk liquid including pore-surface relaxation, and tortuousity effects. In particular it is well known that the relaxation rate for liquids at an interface measured as a function of NMR rotational frequency exhibits a slope as a function of frequency. The evidence is that this depends on the oil/water wetting state of the surface and whether it is an oil or water-based liquid (Figure 2). The data we measure exhibits this effect; however, we note that the decay rate in the pore (red line) converges to the bulk value (blue line) as the measurement frequency goes down:

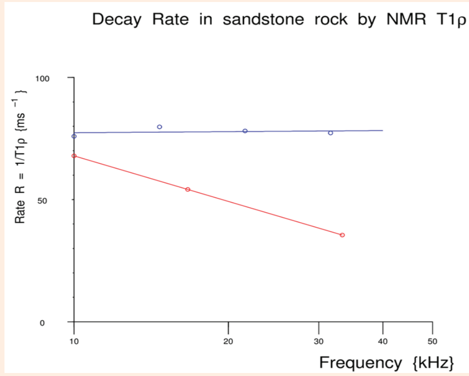

Figure 2: The decay rate R of the NMR T1ρ signal in the pore (red line) converges to the bulk value (blue line) as the measurement frequency decreases.

So, as we said earlier, the question is, how closely can we determine the true viscosity (which we know fairly well in the bulk) for this oil in this sandstone rock, using the NMR spin lock T1rho sequence? It is far too early to say whether it is significant that the bulk relaxation rate and the pore T1ρ relaxation rate are similar as a frequency of a few kilohertz. Further measurements have to be completed on the sandstone with other the calibration viscosity liquids, and then extend this work to carbonate, and shales.

Future Work

Such measurements are possible not only in the laboratory, and also in the field using the Lab-Tools compact NMR Spectrometer since it fits in a shoulder bag [3], but should also be possible down-borehole, with a suitable well logging tool.

References

These first two references discuss NMR Fast Field Cycling studies on liquids in pores:

1. Korb JP (2011) Nuclear magnetic relaxation of liquids in porous media. New Journal of Physics 13: 26.
2. Singer PM, Parambathu AV, Wang X, Asthagiri D, Chapman WG, et al. (2020) Elucidating the ^{1}H NMR relaxation mechanism in polydisperse polymers and bitumen using measurements, MD simulations, and models. J Phys Chem B 124(20): 4222-4233.
3. J Beau W Webber (2024) A rock! Is it porous, does it contain oil or water - a tool for the hiker. Journal of Mineral and Material Science (JMMS) 5(5): 1-2.