NMR PETROPHYSICS FOR TIGHT OIL SHALE ENABLED BY CORE RESATURATION

Ravinath Kausik, Kamilla Fellah, Erik Rylander, Philip M. Singer, Richard E. Lewis, Schlumberger, Steven M. Sinclair, Matador Resources Company

This paper was prepared for presentation at the International Symposium of the Society of Core Analysts held in Avignon, France, 8-11 September, 2014

ABSTRACT

Economic production from tight (organic) shale reservoirs is governed by the ability to position horizontal wells using the log and core data acquired from the vertical wells. The major factors that drive successful production from these wells include petrophysical properties such as porosity, permeability, wettability, hydrocarbon saturation, and pore pressure. Other factors include geo-mechanical attributes such as hydraulic fracture surface area plus fracture conductivity. Accurate measurements of these petrophysical properties are fundamental for improved performance from tight oil reservoirs. In this paper, we describe a novel methodology based on NMR relaxometry of hydrocarbon and water resaturated cores to determine the hydrocarbon saturations, wettability and porosity, and to determine accurate T_2 cut-offs.

One caveat for NMR core analysis is the loss of fluid, including producible hydrocarbon, during core retrieval to the surface. This can lead to a porosity deficit compared to logs. In this paper, we introduce a high pressure fluid resaturation methodology, and demonstrate the effectiveness in saturating extremely hydrophilic Vycor porous glass, with pore sizes comparable to the smallest pores in shale (4 nm diameter), using hydrocarbons as the saturating fluid. Applying the same core resaturation methodology to tight-oil shale samples, we demonstrate how 2D NMR (Nuclear Magnetic Resonance) relaxometry experiments can then segregate the T_2 distribution into 3 separate components. The longest T_2 component—generally above 10 ms—can be clearly identified as fluids in intra-particle or inter-particle porosity. The fluids in this mixed-wet pore system exhibit T_1/T_2 ratios close to one, and constitute movable or producible fluid. The second component consists of the T_2 distribution from 1 ms to 10 ms. This contribution is mostly from the light oil in oil-wetting organic porosity exhibiting T_1/T_2 ratios between 3 and 6. We demonstrate that only a part of this oil is producible. The shortest T_2 component has values less than 1 ms, and constitutes the bound water and bitumen signal. Their T_1/T_2 ratios generally range from 4 to 8.

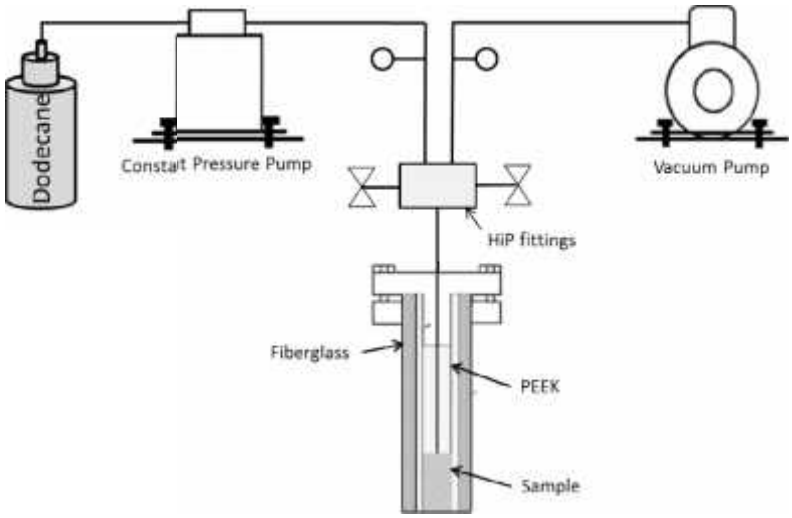

Fig.1.The high pressure shale resaturation setup used for this paper is shown. The formation fluid, either oil or brine, is re-injected back into twin shale samples after vacuum evacuation.

INTRODUCTION

NMR core-analysis applications developed recently have shown to be useful for the characterisation of fluids in organic shale plays [1]. The understanding of the NMR relaxation and diffusion properties of bound water and gas in gas-shale samples [1] has resulted in new methodologies for logging and interpretation in these formations [2,3]. Recently, the interest has shifted to tight-oil (organic) shale reservoirs because of the economics and the improvement in technology to make production from these plays successful. Laboratory NMR core-analysis experiments have been attempted in combination with other methods like Tight Rock Analysis (TRA) and mercury injection capillary pressure (MICP) to better characterise these rocks [4]. One caveat for the NMR core-analysis methodologies is the loss of fluid, including producible hydrocarbon during core retrieval to the surface. This makes the NMR measurements of the core samples unrepresentative of down-hole reservoir conditions. Nevertheless, the one direct application of these measurements is the use of the porosity difference between the core and log porosities, as a proxy for the movable fluid fraction [4,5]. To better understand the shale samples at their in-situ state, we introduce a high-pressure method for resaturation the formation fluids, followed by 2D NMR T_1-T_2 experiments, and demonstrate how new insight about wettability and fluid saturations can be obtained.

EXPERIMENTAL

The sample resaturation setup is shown in Figure 1 and consists of a Poly Ether Ether Ketone (PEEK) sample holder fitted into a fiberglass unit custom ordered from Temco, capable of handling pressure up to 5,000 psi. The sample holder is connected to a vacuum pump and a high-pressure pump through a three-way valve. The high-pressure pump is supplied with the formation oil for resaturation. The formation oil

we used had 42° API gravity, a viscosity of ~ 3 cP at ambient, and an ambient bulk NMR response peaked at T_2 ~ 300 ms and T_1/T_2 ~ 1.1.

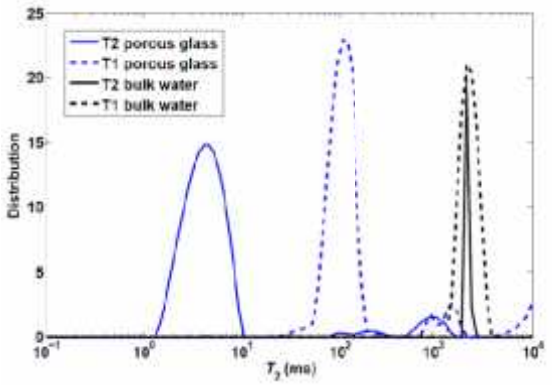

Fig.2. The T_1 and T_2 distributions of bulk water are compared with those for water filled Vycor porous glass. The reductions in the relaxation times are due to the strong hydrophilic nature of the Vycor glass.

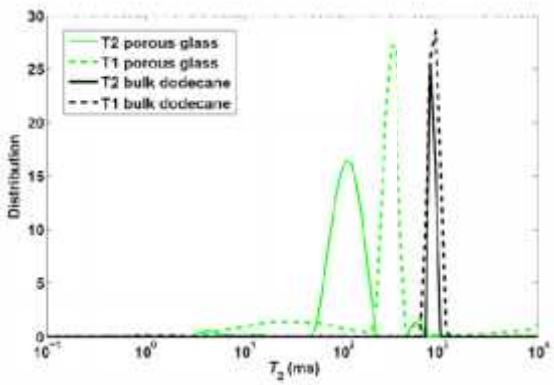

Fig.3. The T_1 and T_2 distributions of bulk dodecane are compared with those for dodecane filled Vycor porous glass. The reductions in the relaxation times are due to slow motions inside the pores of Vycor glass.

All high-pressure connections were purchased from High Pressure Instruments (HiP) and were rated to 30,000 psi. Since the sample holder is made of non-magnetic parts, NMR measurements can be made in parallel during the resaturation process. The highest pressures used in the experiments was 2,000 psi, at which almost complete resaturation of the samples was achieved. For the shale samples, this was confirmed by comparing NMR porosity with Tight Rock Analysis (TRA) data on the same samples. The NMR data were acquired on an Oxford Instruments GeoSpec2 rock-core analyzer at a resonance frequency of 2 MHz, which is the same as that of the logging tool. The measurements were made at ambient temperature (72 °F) using echo times (TE) of 100 μs. The native samples and twin samples resaturated with formation oil and brine were separately measured. The 2D NMR T_1-T_2 maps were acquired with 24 log-spaced inversion-recovery steps ranging from 0.2 ms to 1 s, and processed using the fast inverse Laplace transform [6].

Vycor porous glass cylinders with uni-modal pore size of 4 nm were cut to 3.5 cm by 0.6 cm. The shale samples from the lower Eagle Ford were chosen to avoid high clay streaks. The samples consisted of end trims of irregular shapes, and were broken to cross-sectional dimensions of ~2 mm in order to increase the surface-to-volume ratios for fluid resaturation. Bulk density measurements were made on solid cylindrical cores, from which the end trim samples were obtained. The core bulk densities did not change, indicating good core preservation over the 18-month period after they were recovered.

RESATURATION OF MODEL VYCOR POROUS GLASS

One Vycor porous glass rod was saturated with oil, while a separate twin sample was saturated with water. Both were saturated at a pressure of 2,000 psi for a period of 48

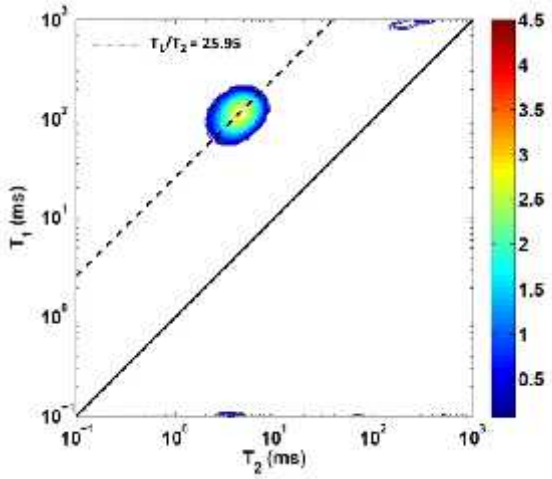

Fig.4. The T_1-T_2 ratio water in the 4nm pores of Vycor porous glass is shown to be 25.95.

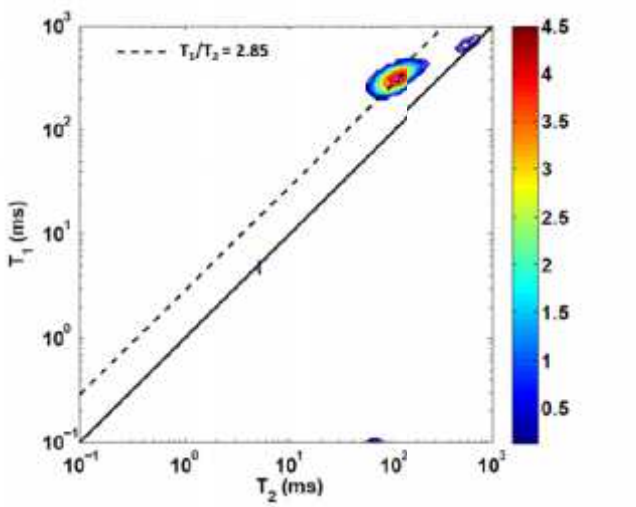

Fig.5. The T_1-T_2 ratio dodecane in the 4nm pores of Vycor porous glass is shown to be 2.85.

hours after vacuum evacuation for 2 hours. The NMR porosities measured by integrating the T_2 distributions for water and dodecane-resaturated Vycor showed a good match with conventional gas porosity (Helium injection) and Brunauer-Emmett-Teller (BET) measurements (Table 1). This indicates that all the extremely hydrophilic pores of Vycor (4 nm size) were completely saturated, thereby justifying the resaturation methodology for application on shale samples with similar sized small pores. The other challenge in shale is the low permeability and the possibility of unconnected pores, therefore shale samples of small size and high surface to volume ratios were used. The reduction in the T_1 and T_2 distributions of the oil and water resaturated Vycor in comparison to the respective bulk distributions are shown in Figures 2 and 3. In the case of water, the T_1 is reduced to a value of 10 ms and the T_2 value to 4 ms, resulting in a T_1/T_2 ratio of 25 (Figure 4). For oil, the T_1 and T_2 are reduced from their bulk value of 2 s to 280 ms and 100 ms, respectively (Figure 3), resulting in a T_1/T_2 ratio of 2.85 as shown in Figure 5. This increase in the T_1/T_2 ratios is due to the slowing down of motions at the pore surfaces in Vycor glass. The T_1/T_2 ratio is especially high for the case of the water as the porous silicon oxide Vycor glass is highly hydrophilic, and is therefore strongly water-wet. The T_1/T_2 ratio for oil is only slightly increased from the bulk value due to the moderate motional slowing down at the pore surfaces. The data shows that the T_1/T_2 ratio is potentially a good wettability indicator, where increasing T_1/T_2 ratio indicates increasing wetting.

Sample	Gas Porosity	BET Porosity	NMR Porosity
Water saturated porous glass	31.0	31.8	33.9
Dodecane saturated porous glass	30.9	31.6	32.9

Table 1: The NMR porosity determined from dodecane and water resaturation experiments is compared with Helium gas porosity and BET porosity (porosity units).

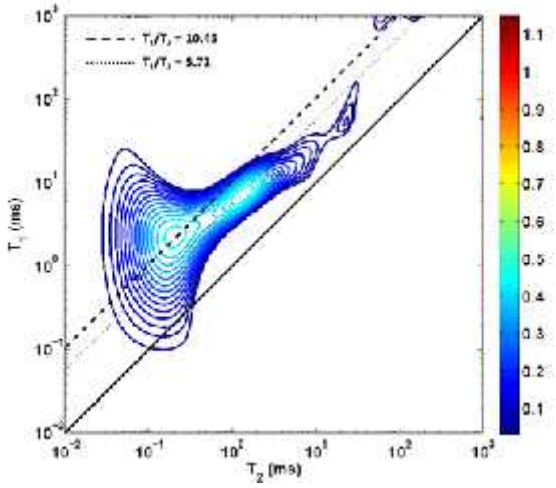

Fig.6. The T_1-T_2 map of native state shale sample consists of the Bitumen and bound water signals <1ms and the immovable oil between 1ms and 10ms.

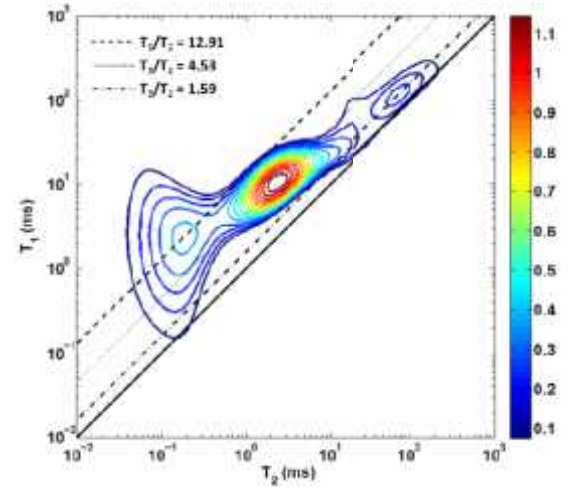

Fig.7. The T_1-T_2 map of formation oil resaturated shale sample shows the movable oil in organic porosity (kerogen) between 1ms and 10ms in addition to the native sample response.

OIL AND BRINE RESATURATION OF THE SHALE SAMPLES

The 2D NMR T_1-T_2 map of the native shale sample is presented in Figure 6, showing peaks at ratios of 10.45 and 5.72. These peaks correspond to residual fluids after the movable fluids escape during core retrieval [4,5]. The peak with $T_2 \sim 0.1$ ms and $T_1/T_2 \sim 10.45$ corresponds to bitumen, as confirmed by experiments on bitumen isolates. The clay-bound water overlaps with this signal, and is not clearly differentiated because the resolution in the maps is low at these short relaxation times. The T_1-T_2 map of the shale resaturated with formation oil shows three distinct peaks (Figure 7). The first peak at short relaxation times is similar to the native shale. The second peak with T_2 from 1 ms ⇔ 10 ms has a $T_1/T_2 \sim 5$ and shows a large increase in amplitude after oil injection, indicating that this peak is associated with re-injected oil in the organic porosity. This can be more clearly seen from the comparison of 1D T_2 distributions of the native state sample with the oil-resaturated core (Figure 8). The injected oil in the organic pores is a good measure of the movable oil porosity in the reservoir. The third peak with $T_2 > 10$ ms and $T_1/T_2 \sim 1.59$ corresponds to injected oil in inorganic porosity, which the NMR log response indicates is largely absent under down-hole conditions. Similar results were observed for 8 other samples from different depths in the lower Eagle Ford.

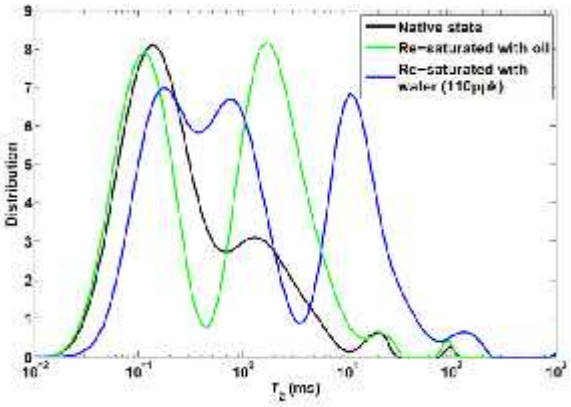

Fig. 8: The 1D T_2 distributions of the native shale sample is plotted together with the formation oil and brine resaturated shale.

	Injected oil: Organic pores	Injected oil: Inorganic pores	Injected water: Organic & Inorganic pores	Bitumen & Clay bound water
T_2	1 ms ⇔ 10 ms	> 10 ms	6 ms ⇔ 80 ms	< 1.5 ms
T_1/T_2	3 ⇔ 8	1 ⇔ 2	1.3 ⇔ 2.5	6 ⇔ 15

Table 2: Injected fluid identification using T_1-T_2 ratios of the resaturated shale samples.

The 1D T_2 distribution of the 110 ppk brine resaturated shale sample is also shown in Figure 8. The injected water shows a roughly uniform increase in signal amplitude across T_2 = 1 ms ⇔ 10 ms, with roughly uniform T_1/T_2 ratio between 1.3 ⇔ 2.5 (not shown). The data indicates that the injected oil T_1/T_2 ratio is more sensitive to organic versus inorganic pore environments than the injected water response.

CONCLUSION

A core resaturation methodology involving re-injecting of formation oil and brine back into tight-oil shale samples has been introduced. The separation of the different fluids using the T_2 and T_1/T_2 values are summarised in Table 2. These can be directly applied to determine fluid saturations and producible fluid quantities using core-log data integration. The most important conclusion is that the injected oil signal in the organic and inorganic porosities can be well separated using this technique, which paves the way forward for determining log cut-offs and oil recovery estimates.

ACKNOWLEDGEMENTS

The authors would like to thank Matador Resources Company and Schlumberger Technology Corporation for allowing the publication of this work.

REFERENCES

1. Kausik, R., Cao Minh, C., Zielinski., L., Vissapragada, B., Akkurt, R., Song, Y.-Q., Liu, C., Jones, S., and Blair, E., “Characterization of Gas Dynamics in Kerogen Nanopores by NMR,” SPE (2011), 147198.
2. Hook, P., Fairhurst, D., Rylander, E., Badry, R., Bachman, N.H., Crary, S., Chatswanich, K., and Taylor, T. “Improved Precision Magnetic Resonance, Acquisition: Application to Shale Evaluation,” SPE (2011), 146883.
3. Cao Minh, C., Crary, S., Zielinski, L., Liu, C.B., Jones, S., and Jacobsen, S., “2D-NMR Applications in Unconventional Reservoirs,” SPE (2012), 161578.
4. Rylander E., Singer P.M., Jiang T., Lewis R.E., McLin R., Sinclair S.M., “NMR T_2 Distributions in the Eagle Ford Shale: Reflections on Pore Size,” SPE (2013), 164554.
5. Singer P.M., Rylander E., Jiang T., Lewis R.E., McLin R., Sinclair S.M., “1D and 2D NMR core-Log Integration in organic shale”, SCA 2013-018, (2013) .
6. Venkataramanan L., Song Y.-Q., Hürlimann M.D., “Solving Fredholm Integrals of the First Kind with Tensor Product Structure in 2 and 2.5 Dimensions,” IEEE Transactions on Signal Processing, 50(5), 2002.