

Characterization of Water-in-Bitumen Emulsions in Model Porous Media by NMR Microscopic Imaging Techniques

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1. Introduction

Production from thermal (steam enhanced) oil recovery processes is complicated by the presence of water-in-oil emulsions. [1], [2] Critical to monitoring the in-situ formation and flow of these water-in-oil emulsions is the ability to distinguish between the various components (bitumen, water/steam and emulsified water) present in a core flood experiment. In principle, NMR imaging is ideally suited to monitor the spatial distribution of absorbed fluids, and in this regard, the viability of the NMR imaging technique to examine the distribution of fluids in reservoir rock samples has recently been demonstrated. [3] - [15]. In general, several fluids or phases may be present and the ability to distinguish these components is of prime interest.

In the majority of these studies, the NMR imaging technique has been applied to samples which contain a low viscosity crude oil and water/brine. In the case of heavy oil production, the fluids have a high and varying viscosity, (ie: bitumen, water and emulsions of bitumen and water). Under these circumstances, it is possible that these differences in viscosity will lead to the ability to discriminate between phases via differences in NMR relaxation behaviour. In addition, the influence that the solid matrix has on the relaxation behaviour of water dispersed as an emulsion may be quite different from water absorbed into the porous medium. In an effort to evaluate the feasibility of characterizing water-in-bitumen emulsions by NMR microimaging techniques,

the one-dimensional NMR spectra, the relaxation time constants and the spin-echo images for a series of samples consisting of water, bitumen and water-in-bitumen emulsions absorbed into glass beads were examined and are presented herein.

2. Experimental

All water-in-oil (bitumen) emulsions were prepared from Cold Lake bitumen which has been ultracentrifuged to remove solid particles. The emulsion samples were created by passing a heated mixture of bitumen and water through an auxiliary sand column at a suitable flow rate. The emulsions were checked under an optical microscope to ensure that the water phase was well dispersed prior to packing. Eight samples were prepared each consisting of a different fluid/porous medium mixture. Sample 1 contained 10 mL of Cold Lake Bitumen. Sample 2 contained 10 mL of a 20 % (w/w) water-in-bitumen. The remaining samples contained fluid absorbed into a glass bead matrix. Two different sizes of glass beads were used. The small glass beads were determined to be 88 - 104 μm in diameter which represents fine sand. The large glass beads were 0.8 - 1.0 mm in diameter. Sample 3 contained approximately 10 g of the large glass beads with 5 mL of distilled water. Sample 4 had a similar composition to sample 3 except that the smaller glass beads were used. Sample 5 contained 10 g of the large glass beads with 5 mL of a water-in-oil emulsion (20% w/w water). Sample 6 had a similar composition except that the smaller glass beads were used. To ensure that the composition of the water-in-bitumen emulsion was maintained in the glass bead matrix, samples 5 and 6 were prepared by mixing the emulsion with the glass beads by hand.

and then placing the appropriate amount of the mixture at the bottom of a 10 mm NMR tube. The samples were then spun at low speeds on a bench-top centrifuge for 10 minutes to obtain a uniform packing. In an effort to compare the NMR behaviour of the two types of fluids directly, Sample 7 was composed of two regions, the bottom layer contained emulsion in large glass beads and the top layer contained distilled water in large glass beads. Sample 8 was identical in composition to sample 7 except that the smaller glass beads were used. The contents of the samples are summarized in Table 1.

Table 1. Composition of Samples

Sample	Fluid	Matrix
1	Bitumen	None
2	Emulsion	None
3	Distilled Water	88 - 104 μm
4	Distilled Water	0.8 - 1.0 mm
5	Emulsion	0.8 - 1.0 mm
6	Emulsion	88 - 104 μm
7	Emulsion/Water	0.8 - 1.0 mm
8	Emulsion/Water	88 - 104 μm

All NMR measurements were made on a Bruker MSL 400 spectrometer equipped with a micro-imaging system using the proton microimaging probe equipped with a vertical 12 mm saddle coil. The nonselective 90° rf pulse length was 14.5 μs . Quadrature phase cycling was used in all the spectroscopic measurements. 1D ^1H NMR spectra and Carr-Purcell spin-echo (90-tau-180) NMR spectra [16] were obtained to characterize the samples. The spin-echo sequence was also used to determine the average spin-spin relaxation times (T_2). The inversion-recovery sequence [17] was used to determine the average T_1 spin-lattice relaxation times.

After evaluating the relaxation time constants and the NMR lineshapes for several of the samples, it was determined that the spin-echo imaging sequence [18] was an appropriate choice. The spin-echo imaging pulse sequence employs a 90-tau-180 rf pulse sequence in which a hard 180° pulse sequence is used to refocus the effects of field inhomogeneity. Slice selection was performed by using a selective 90° pulse with an appropriate G_z gradient. Echo times for the spin-echo imaging experiments varied

from 4.5 ms to over 100 ms and the actual echo times are indicated in the text. The slice thickness was typically 2.1 mm. The phase encoding gradient was incremented through 256 experiments. The frequency encode gradient was 5.8 G/cm resulting in an in-plane resolution of 95 μm . The samples used in this study had a porosity of approximately 30 - 35 % and the imaging experiments required 1-4 hours to acquire. Images presented in this paper follow the convention in which an inverse gray scale is used to indicate relative intensity. The darker the region on the image, the higher the concentration of water.

3. Results and Discussion

The relaxation behaviour and linewidths were investigated (Table 2) to determine the appropriate imaging sequence for the bitumen and water-in-bitumen emulsions. A 1D ^1H NMR spectrum of Cold Lake Bitumen consisted of a large peak ($\nu_{1/2} = 690$ Hz) which is assigned to the heavy oil component (bitumen) and a small shoulder which is attributed to trace amounts of connate water. The spin-spin relaxation time (T_2) of the oil component was determined to be 1.3 ms which indicates that quantitative spin-echo imaging of the bitumen component in the samples will not be possible.

The linewidth determined for distilled water placed in the small glass beads (Sample 3) is approximately 1000 Hz, an increase of more than 2 orders of magnitude over that observed for a distilled water phantom. Similar line broadening was observed for the other samples (Table 2). The line broadening is a result of the magnetic susceptibility differences between the solid matrix and the absorbed fluids. These line-widths indicate that the application of the gradient echo sequence [18] would be difficult due to the short T_2^* values ($T_2^* = 300 - 800$ μs). The spin-spin relaxation parameter for the distilled water absorbed into either glass bead matrix (Table 2) is markedly reduced from that observed for bulk water. An NMR image of Sample 4 with an echo time of 4.5 ms demonstrates that the distribution of water in these types of samples are possible. (Figure 1a) The image with an echo time of 104 ms (Figure 1b) is more interesting. It shows a large decrease in signal intensity, particularly where the distilled water is in contact with the glass beads. Due to the non-uniform packing of the large glass beads, small pockets of water on the order of 100 - 400 μm in diameter can be observed.

An examination of the NMR relaxation parameters of the 20% (w/w) water-in-bitumen emulsion (Sample 5) absorbed into the large glass bead matrix reveals that the T_2 relaxation time constant of the water phase is unaffected by placing the emulsion into the glass bead matrix. The image obtained with a 104 ms echo time (Figure 2) displays a strong uniform signal intensity. The bitumen component of the sample has a T_2 relaxation time constant on the order of a millisecond, and thus does not contribute to the intensity of the image.

Figure 1 (a) Spin echo images of sample 4.
a) Echo time = 4.5 ms. (b) Echo time = 104 ms

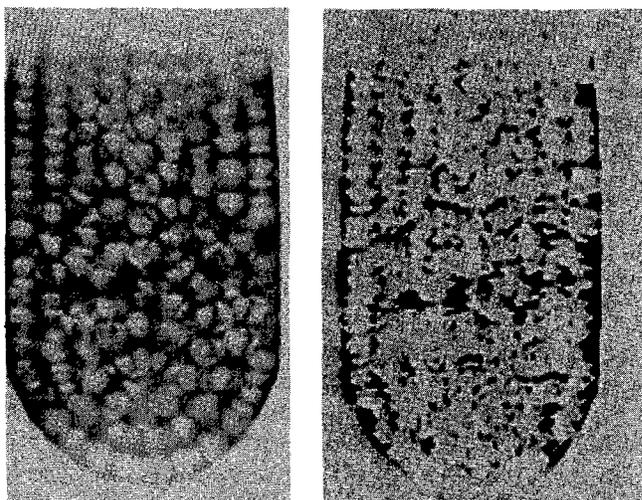
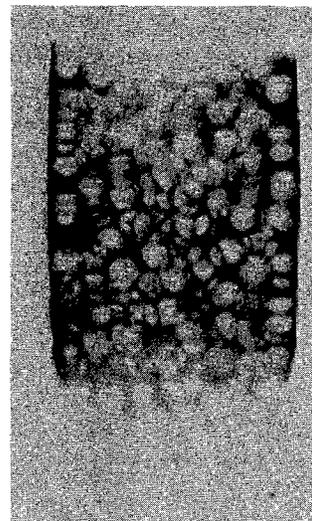


Figure 2. Spin-echo image of sample 5.
Echo time = 104 ms.

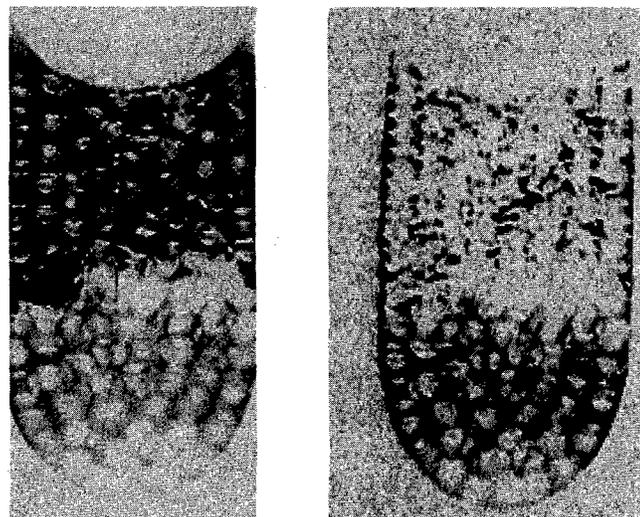


During a core-flood experiment it is likely that both water and emulsified water phases will be present. The ability to distinguish between bulk absorbed water and emulsified water is considered essential to analyzing the formation and flow of the emulsions in sand packs. To this end, a sample which contains both distilled water and water-in-bitumen emulsion in a glass bead pack was examined (Samples 7 and 8).

The conditions of the imaging experiments were chosen such that only the water component of the samples were observed. Using an echo time of 4.5 ms the distribution of water in sample 7 (Figure 3a) can be obtained. The distilled water plus large glass beads are in the top half of the NMR tube and this results in a much higher signal intensity due to the higher water concentration. To discriminate between the emulsified water phase and the absorbed bulk water, an echo time of 104 ms was used. (Figure 3b) Under these conditions, only water which has a high mobility or low surface contact will be observed (ie: water which is emulsified will be favoured). This results in an image in which small pockets of water are observed in the distilled water region, while the emulsion containing region appears nearly uniform.

The effect of the smaller grain size of the relaxation parameters was examined using sample 8. The smaller glass beads have a more uniform pore size distribution and are more representative of the matrix used in core flood experiments. In such samples, the pore size is typically on the order of 30 μm which means that all of the water will be in intimate contact with the solid matrix.

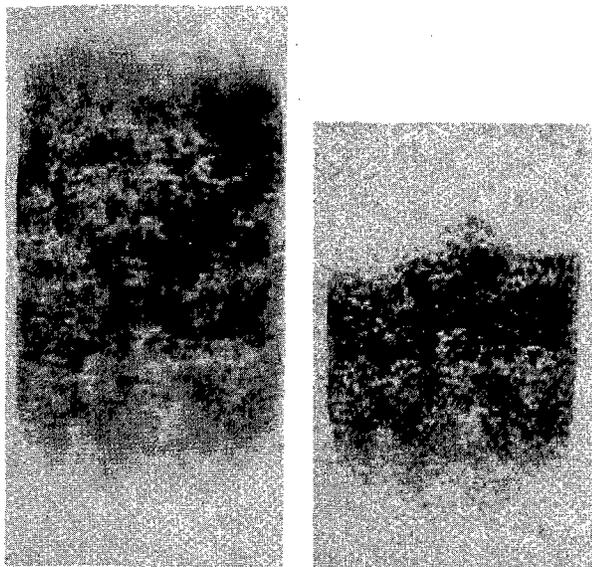
Figure 3. Spin echo images of sample 7.
a) Echo time = 4.5 ms. (b) Echo time = 104 ms



The T_2 of the distilled water component is reduced to 4.9 ms, on the same order of magnitude as the echo time (4.5 ms). This means that the intensity of the water will be strongly T_2 weighted and the region which contains the distilled water will no longer have an intensity which is much higher than the emulsion. (Figure 4a) When the echo time is increased to 34 ms, (Figure 4b) the region which contains the distilled water disappears in a uniform manner. The smaller grain size of the solid matrix has enlarged the relaxation time differences between the bulk absorbed water and the emulsified water and thus the contrast between the two physical states are enlarged.

Figure 4. Spin echo images of sample 8.

a) Echo time = 4.5 ms. (b) Echo time = 34.5 ms



3. Summary

In the present study, the feasibility of examining heavy oil emulsion samples has been demonstrated. Bitumen, water and emulsified water placed into glass beads are easily distinguished in an NMR imaging experiment on the basis of their relaxation times. The spin-spin relaxation time constant, T_2 observed for the water component of the emulsion samples placed in contact with glass beads was dramatically different than that found for distilled water. The NMR experiment is therefore sensitive to the physical state of the water, ie: whether the water is emulsified and therefore 'protected' from the solid matrix. This difference in relaxation times was exploited to provide water-selective images of the emulsified water phase in the presence of oil and non-emulsified water phases. Good S/N images can be obtained with high in-plane resolution (50-100 μm) can be obtained on a microimaging system in reasonable time periods. Saturation profiles can be obtained in a manner of seconds which would allow for the continuous monitoring of the formation of an emulsion under flow conditions. The ability to monitor the formation and flow of water emulsions will be important to understanding enhanced oil recovery processes.

Table 2: Proton Relaxation Times and Linewidths of Water and Bitumen

Sample	T_1 (s)	T_2 (ms)	$\nu_{1/2}$
1	0.71	1.3	690
2 water	2.2	200*	150
bitumen	0.62	1.8	650
3	2.4	29	350
4	2.3	4.9	1000
5 water	1.6	495	
bitumen	0.58	1.3	
6 water	1.5	484	
bitumen	0.45	1.4	

* The water component of emulsion samples show a variation in T_2 . The reasons for this variation are under investigation but are believed to be due to the size distribution of the water droplets.

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4. References

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