USING LOW-FIELD NMR TO UNDERSTAND FINES BEHAVIOUR

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ABSTRACT

An increasing amount of the oil recovered today originates from oil sands. The recovery efficiency from oil sands is dependent on the fines content, however the relationship between fines content and oil recovery is not well understood. This paper proposes using low field nuclear magnetic resonance (NMR) as a tool to understand the correlation between the grade of the ore (i.e., the degree of ease to separate oil from the sand) and the fines content. First, synthetic samples with different clay, sand, brine and heavy oil contents were created to simulate ores of varying recovery grades. The NMR results showed that brine in montmorillonite is distinct from brine in illite or kaolinite, a fact that could be exploited in using NMR for clay identification. The results from the synthetic samples were used to create nomographs correlating NMR parameters and clay content. Second, field ore samples underwent Dean-Stark extraction and the solids were re-saturated and re-measured in the NMR. The results were compared to those obtained from synthetic samples with similar compositions and it appears the results from the two types of samples are similar, despite having significantly different particle size distributions. This work shows that it is possible to determine fines content from NMR. Upon creating a more general model, these findings may be used towards determining clay type and content from NMR logs. This information may also be used towards refining the cut-off method for more accurately determining oil reserves.

INTRODUCTION

Nuclear magnetic resonance (NMR) logging tools have been used in numerous applications within the petroleum industry for reservoir characterization. In addition to helping to determine porosity, permeability and producible fluids [1], NMR has been used to characterize heavy oil and bitumen [2-4], determine compositions of oil/brine emulsions [5-7] and determine the viscosity of heavy oil and bitumen [8]. NMR logging tools obtain information regarding fluids in porous media by using magnetic fields to polarize the protons in the fluid and monitoring the time it takes the protons to return to equilibrium. This time is commonly termed the transverse relaxation time (T_2). Low-field NMR, operating at magnetic field strengths on the order of 0.01 T, is more appropriate for analysis of porous than high field NMR, which operate on the order of 1 T and higher. This is because the lower field gradient minimizes the possibility of data degradation during a measurement [9]. Protons in bulk brine have a T_2 value of

approximately two seconds, but the T_2 values for heavy oils and bitumen are much faster (i.e., between 0.1 and 10 ms). The reason for this is the protons in heavy oil and bitumen are restricted due to the viscous environment [1, 10]. In fact, the viscosities of some heavy oils are so high that present NMR logging tools are incapable of detecting the complete spectrum from heavy oil and bitumen formations. As a result, attempts to quantify heavy oil and bitumen contents are problematic. Restriction of proton movement can occur also because the fluid has adsorbed onto clays or organic matter in the sample [11]. Consequently, clay bound brine has low T_2 values (i.e., less than 10 ms) compared to brine residing in the larger pores of the samples, which has T_2 values that are approximately 100 ms. The fact that the amplitude peaks for heavy oil and clay bound brine appear at similar T_2 values makes it difficult to differentiate between the oil and brine signals from a sample that contains both fluids. This makes the T_2 cut-off method [1], implemented to calculate volumes of free and bound fluid, impossible to use because the assumption that the signal below the T_2 cut-off value is due to oil alone is incorrect. There have been attempts to separate oil and brine signals in NMR spectra [1, 12] but these methods were not developed for samples containing heavy oil or bitumen with clay bound brine.

The purpose of this work is two-fold with the first goal being to determine the effect of clay content and type on NMR spectra of brine and heavy oil in clay/sand mixtures. Empirical correlations between NMR parameters and clay content were formulated. These results were then tested on field samples, which is the second goal. The ability to do so would enable one to determine the clay content, thus provide an idea regarding recovery efficiency. Improved oil sands characterization is a consequence of this work.

EXPERIMENTAL
Materials
Preferentially brine-wet sand was obtained in Calgary, AB and characteristics about this sand can be found elsewhere [13]. The illite clay is mined from Ravenscrag, SK while the kaolinite clay is mined from Troy, Idaho [14]. The montmorillonite clay is a calcium-substituted source clay from Apache County, Arizona obtained from the Source Clay Repository at Purdue University, IN [15]. The brine used in this experimental program consists of 2wt% NaCl. The oils used with the clay/sand mixtures were both heavy crude from Cold Lake, AB. The viscosities of these oils were 29,000 and 50,000 mPa·s, respectively and the densities of these oils were 0.9893 and 0.9886 g/cm^3, respectively, at 30°C [8].

Eight field ores from Fort McMurray, AB were collected for this experimental program. Three of the samples were representatives of ores with varying recovery grades (labelled as Ore A, Ore B and Ore C). The other five samples were obtained from one well at different depths, thus four zones of varying recovery grades were represented. The samples were labelled as Ore 1 to Ore 5.

The sample holders were glass vials with polyethylene caps and additional sealing was achieved using Teflon tape to minimize the amount of evaporation. These materials were chosen because they do not affect the NMR signal.

Instrumentation
All NMR measurements were performed at ambient temperature and pressure using a relaxometer with a frequency of 1 MHz and a field strength of 0.024 T. There are no field gradients in this machine, so relaxation due to diffusion is negligible in this program. Independent measurements with this machine have shown that this instrument can detect 0.25 g of bulk water accurately. The NMR parameters used for the samples are shown in Table 1. These conditions were chosen to capture the maximum amount of information from each set of samples in the range of T_2 values where heavy oil and clay bound brine will relax. The machine was tuned daily to ensure that differences in NMR spectra over time were indeed due to changes in the solids/oil/brine system and not due to differing degrees of proton excitation over different days. Duplicate runs were performed during each measurement in order to ensure that the results obtained were genuine and not due to experimental artifacts. The data was processed using a software package that employs the non-negative least squares method to create the NMR spectra.

Procedure
Creating And Measuring Samples With Clay Alone As The Solids Phase
Three sets of clay samples, with each set consisting of nineteen samples containing illite, kaolinite or montmorillonite, were tested (i.e., there were 57 samples tested where the solids phase consisted of clay alone). Each suspension weighed fifteen grams and the clay content in each set of suspensions varied between 5 and 95wt% by adding water to the "dry" clays. This differs from the approach of starting with a dilute slurry of clays and concentrating them. Once the sample components were added into the sample holders, the lids were placed and Teflon tape was wrapped around the lid and vial to minimize brine loss due to evaporation. The samples were then shaken for one minute and allowed to settle for 24 h. An NMR measurement was taken at that time.

Creating And Measuring Samples With Clay And Sand As The Solids Phase
Three batches of samples, each consisting of eight sets (where one set is composed of six samples), were created containing one of the three clays (i.e., there were 144 clay/sand samples tested). Each set consisted of six samples and each sample consisted of fifteen grams of clay and sand. NMR measurements vials contained 5, 10, 20, 30, 40 or 50wt% (dry weight) of each clay compound in the following manner:
• Sets 1 and 5 contained clay dispersed throughout the solids phase.
• Sets 2 and 6 contained clay added as a layer at the top of the solids column.
• Sets 3 and 7 contained clay introduced as a layer in the middle of the solids column.
• Sets 4 and 8 contained clay as a layer at the bottom of the solids column.

The system used to identify the samples is summarized in Table 2. Samples with no clay (labelled "Sand") were also tested. For Sets 1 and 5, the samples were shaken vigorously for one minute to ensure that the clays were dispersed throughout the solids. Care was

taken during sample preparation and testing of the other six sets of samples to ensure that the clay and sand layers did not mix together.

Sets 1 to 4 were exposed to brine via a syringe at a rate of approximately 1 drop per second. The purpose of adding brine in this manner was to minimize disturbance on the top of the sample column. The brine was added under atmospheric conditions to allow for spontaneous imbibition to occur and the amount of added brine was not sufficient to fully saturate the solids. The vials were then sealed and wrapped with Teflon tape to minimize fluid loss as a result of evaporation. Measurements were taken at 0, 1, 4 and 24 h after bringing the brine content to 9wt%, 17wt%, 23% and 29wt% (based on total sample weight – saturation values were not provided since the porosities for these unconsolidated samples could change depending on the fluid content). These times were determined from previous independent tests which showed that fluid uptake predominantly takes place in the first day and relatively little occurs afterwards [16]. Sample masses were recorded prior to each NMR measurement to verify that minimal evaporation has occurred.

Sets 5 to 8 were exposed to 5wt% (based on total sample weight) brine. The brine was introduced in the same manner described above and the samples were measured using the same schedule described above. The following day, heavy oil was added to bring the oil content to 5wt% (based on total sample weight). NMR measurements were obtained using the same schedule, then a new set of measurements were taken after bringing the oil content to 9, 16 and 22wt% (based on total sample weight).

Testing Field Samples
NMR measurements were taken of the ore samples and the ores underwent Dean-Stark extraction afterwards to determine the oil, water and solids content of each sample. A small sub sample of extracted solids underwent particle size analysis (PSA) in a Mastersizer 2000 laser diffractor to determine the fines content (i.e., the percentage of a sample where the particle size is less than 10 μm).

RESULTS AND DISCUSSION
Daily measurements with the standard showed the standard deviation in total amplitude during this program to be less than 1%, which is considered acceptable.

Clay/Brine Samples
The total amplitude from an NMR measurement is directly proportional to the number of protons, thus the amount of fluid, present in the sample. Figure 1 clearly shows that this relationship holds true for the clay/brine samples. The slope of the trendline is known as the amplitude index (AI), which is a characteristic value for different fluids. It has been shown that this value can be used towards determining sample composition [5]. AI for illite and kaolinite are similar to each other (16.7 vs. 16.8). AI for montmorillonite is at 15.59, which is lower than for the other clays, but one can see that a linear trendline does not fit this data set as well as the others. The AI was determined for each sample and this number was plotted against clay content. The results, shown in Figure 2, indicate that AI

values stay relatively constant for illite and kaolinite, but they monotonically increase for montmorillonite. This would suggest that mechanisms other than surface relaxation contribute to the NMR results from samples with montmorillonite.

The NMR data spectra for samples with 90wt% clay are shown in Figure 3. The relative amplitude, which is the amplitude as a percentage of the total amplitude produced by the sample, is plotted on the y-axis to remove the effect of the amount of brine in the sample. By plotting relative amplitude, one can gain a better idea of the relative amounts of brine found in each state within the sample. This figure shows that the brine relaxed at 4 ms when in illite, at 2 ms when contained within kaolinite and 0.5 ms when bound to montmorillonite. This agrees closely with other reported values [11] despite being tested in completely different NMR machines.

Figure 4 shows suspensions with increasing kaolinite content after 24 h of exposure to brine. This figure shows the dominant amplitude peak at approximately 2500 ms for a sample with 10wt% clay. However, the relaxation time of the dominant amplitude peak decreases as the clay concentration increases. This is expected as a greater fraction of the water become clay bound and less of the water remains in the bulk phase. In fact, the samples with higher clay contents are only partially saturated. These characteristics were apparent in the other sets of clay/brine samples.

Another NMR parameter that is used extensively in data analysis is the geometric mean of T_2 (T_{2gm}), or the weighted average of T_2. This parameter is plotted against clay content for the clay/brine samples and the results for the three sets are included in Figure 5. It is apparent from this figure that T_{2gm} values increase as clay content decreases, which is expected due to the increased contribution of bulk relaxation. This figure also shows that the results from illite and kaolinite are very similar whereas the results from montmorillonite are noticeably lower. For example, the T_{2gm} of a mixture with 50wt% kaolinite is approximately 30 ms whereas the T_{2gm} of a mixture with 50wt% montmorillonite is closer to 4 ms. Should T_{2gm} be used as a parameter in an algorithm correlating NMR results and clay content, this feature would likely be used to identify montmorillonite in a sample.

Clay/Sand/Brine Samples
The NMR spectra from the samples containing illite and kaolinite showed only noise. Samples containing montmorillonite did produce some signal before adding brine, but this did not translate to a significant amount of water. This implies that the clays and sand do not contain any appreciable amount of materials that would contribute to the NMR spectra. The resulting spectra obtained after adding brine would be due to the addition of this fluid.
As with the clay/brine samples, the total amplitude was plotted against water mass and the results look similar to Figure 1. The presence of sand caused a decrease in AI values for two of the clays (16.7 vs. 15.9 for illite; 16.8 vs. 16.0 for kaolinite). The sand/montmorillonite/brine samples produced an AI value of 17.64 and there was

significantly more scatter in this data set, which would undoubtedly affect the slope of the trendline. This was observed despite measuring this set of samples numerous times.

Figure 6 shows the NMR spectra for ID30 after exposure to 9wt% brine over the course of 24 h. This partially saturated sample produces two amplitude peaks at 0 h – the one that peaks at approximately 150 ms and one that is located between 3 and 80 ms. The amplitude peak between 3 and 80 ms is attributed to brine in small pores or brine in clay because brine restricted in smaller pores or bound to solids will exhibit faster relaxation than brine in larger pores [1]. This peak is broader than the analogous one in Figure 4 because NMR spectra are affected by pore size distribution [10] and clay/sand mixtures have a wider range of pore sizes than the clay alone. In the larger pores of ID30, a lower percentage of the brine is in contact with the pore wall and the brine residing in the center of the pore will relax more slowly as it is not as restricted as the protons in the bound brine. Thus, the peak at approximately 150 ms is attributed to brine in larger pores.

The shift in the amplitude peaks towards faster T_2 values shows that brine migration took place. This is expected because previous independent experiments have shown similar characteristics during brine migration into other dry unconsolidated samples [18]. The amplitude peak initially located between 3 and 80 ms not only shifts towards faster relaxation times but also grows significantly in height as more brine imbibes into the clay particles dispersed throughout the sand. The same trends were evident in samples containing kaolinite, but not montmorillonite and there are several possible reasons for this. Montmorillonite clay swells considerably, providing a greater surface area per unit mass than non-swelling minerals such as kaolin. A second theory to explain the relatively fast T_2 relaxation times of montmorillonite is the presence of paramagnetic centers, which are metal ions that exhibit a magnetic effect [19]. This theory may apply to the samples tested here due to the variance in iron content (0.9% for illite, 1.1% for kaolinite and 1.5% for montmorillonite [14, 15]).

Figure 7 plots the NMR spectra of ID30, IT30, IM30 and IB30 24 h after exposure to 9wt% brine. The location of the clay appears to affect the location of the dominant amplitude peak since it varies with the location of the clay. This is due to the relative fractions of water bound to clay and present between sand grains. When comparing ID30 to IT30, one notices that the amplitude peak is at faster relaxation times when the clay is dispersed compared to residing as a layer at the top of the column. This occurred in most of the other sets of samples. The most likely reason for this occurrence is that the samples with the clay dispersed throughout the solids phase has a broader particle size distribution and the clay particles easily filled the spaces between the sand grains.

Figure 8 shows NMR spectra obtained 24 h after the addition of 23wt% brine to KB05, KB10, KB20, KB30, KB40 and KB50. Unlike the clay/brine samples whose results are plotted in Figure 4, none of these samples were fully saturated. Consequently, the location of the amplitude peaks remain constant while the relative height of the peak from the faster relaxing component (i.e., approximately 10 ms) changes with clay content to

indicate a greater fraction of clay bound brine. The width of this peak increases with increasing clay content also, because more of the brine may be residing in the sand above the clay. In other words, the brine resides in pores that span over a wider range of sizes. Inversely, the amplitude peak at 400 ms decreases in height and width as the clay content increases. This would be due to the decreased amount of water present that is not bound to clay. These trends were apparent in other analogous sets of samples.

Figure 9 shows clay content as a function of T_{2gm} for the samples with illite dispersed within the solids phase after 24 h of exposure to all brine contents. The y-axis is labelled as "Clay content" and is expressed as weight percent based on the weight of the total sample. Due to the particle size distribution of the clays analysed here and the sand, the entire component that would be considered as fines, defined here as particles less than 10 μm in size, would be due to the clays in the synthetic samples. This figure clearly shows that T_2 values decrease as clay content increases although trendlines for lower brine contents are not linear similar to the data from higher brine contents of clay/brine samples. Analogous plots for the other sample sets show that illite and kaolinite samples exhibited similar curved trend lines and similar T_{2gm} values. However, the samples containing montmorillonite consistently had T_{2gm} values that were lower than illite and kaolinite at any given clay content. A comparison of Figures 5 and 9 shows that the presence of sand lowers the T_{2gm} values at lower clay contents as clay/sand/brine samples have T_{2gm} values lower than 350 ms whereas clay/brine samples have T_{2gm} values greater than 1000 ms at similar clay contents. The addition of sand increases the contribution due to surface relaxation but more importantly, there was less free water in the clay/sand/brine samples.

Clay/Sand/Brine/Oil Samples
Heavy oil was slowly added and the viscosity of the oil prevented this fluid from entering the solids phase even after several weeks of exposure. Figure 10 shows the NMR spectra obtained from IT50 after exposure to 5wt% brine and after the addition of 5wt% oil. The absence of notable changes in the amplitude peak for the fastest relaxing component in Figure 10 indicates that significant fluid migration does not occur. At this point the brine had already reached equilibrium and bulk relaxation is the dominant mechanism for the heavy oil. The predominant amplitude peak for this sample remains at T_2 values less than 20 ms, although this is now partially due to the bulk relaxation of the heavy oil in addition to the clay bound brine. This figure clearly shows that the contribution from clay bound brine to the amplitude peak of the faster relaxing component would lead to an incorrect calculation of oil content based on the conventional cut-off analysis [1].

Figure 11 depicts the correlation between T_{2gm} and clay content for the samples with containing varying concentrations of clay (when it is layered on top of the sand column), 5wt% brine and various concentrations of heavy oil. This figure shows that there is a strong correlation between T_{2gm} and the clay content in a sample, regardless of oil concentration. The scatter in the data from the samples containing montmorillonite is possibly due to the theories mentioned previously. It is worth noting, though, that the

T_{2gm} values from these samples are much lower (i.e., 0.7 – 2.7 ms for montmorillonite vs. 1.3 – 19 ms for illite as oil concentration increases from 5 to 22wt%). Consequently, small changes in T_{2gm} values, such as from 1.1 ms to 1.3 ms, can be significant.

Using Clay/Sand/Brine/Oil Nomographs On Field Ore Samples
The Dean-Stark extraction on the field ores provided the oil contents for these samples. The oil content was used to determine which correlation similar to one shown in Figure 11 should be used on each of the eight samples. The T_{2gm} values from these samples, which were typically between 1 and 4 ms, were inserted into the correlations from each of the three sets of data and the fines content as predicted by NMR and measures by PSA can be found in Table 3. One can see that for most of the field ore samples, one of the numbers predicted from NMR data matched more closely to the PSA data than the other two. This may be a way to determine the clay type in the sample. The solids will undergo x-ray diffraction to identify the clays present.

These results are encouraging as it appears the nomographs created based on results from synthetic samples may be used directly on field samples to determine fines content. The results between NMR predictions and PSA analysis closely match despite the fact that the field samples and clays have vastly different particle size distributions, as shown in Figure 12. However, it is acknowledged that more work is required to make a more general model since parameters such as oil and brine content are necessary at this stage to predict fines content. Additional work is necessary to also understand the effect of parameters such as temperature, brine salinity and brine composition on clay-containing samples.

CONCLUSIONS
Through measuring clay-containing suspensions and packings, NMR provides data that can be used towards determining the presence of montmorillonite because samples with this clay typically produce lower T_{2gm} values than those with equal amounts of illite or kaolinite. Furthermore, correlations found between NMR parameters and clay content for synthetic samples appear to apply well on field samples. Matches between fines content as predicted by NMR and as measured by PSA are strong, even through the particle size distribution of different samples are vastly different. At the present time, oil and brine contents must be known before determining fines content in a field sample, but work will continue on these nomographs to determine a general empirical correlation. Once the effect of parameters such as brine composition and salinity and temperature on NMR spectra are understood, it is the goal to create a general model so that clay content and type can be determined from NMR logs.

ACKNOWLEDGEMENTS

NOMENCLATURE
AI   amplitude index
NMR   nuclear magnetic resonance
PSA   particle size analysis
T   tesla, measure of magnetic field strength
T_2 transverse relaxation time (ms)
T_{2gm} geometric mean of transverse relaxation time (ms)

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Table 1. NMR parameters used.

Samples	Time-to-echo (ms)	Number of echoes)	Number of trains	Wait-time (ms)	Gain
Clay/brine	0.3	5000	25	15,000	4
Clay/sand/brine(/oil	0.3	5000	16	5500	8
Field ores	0.3	5000	25	7500	4

Table 2. System used to label synthetic samples tested.

	Clay content (wt%, dry weight)					
Clay in solids phase	**5**	**10**	**20**	**30**	**40**	**50**
Dispersed	*D05	*D10	*D20	*D30	*D40	*D50
Layered at top	*T05	*T10	*T20	*T30	*T40	*T50
Layered in middle	*M05	*M10	*M20	*M30	*M40	*M50
Layered at bottom	*B05	*B10	*B20	*B30	*B40	*B50

The '*' refers to the clay type: "I" is illite, "K" is kaolinite and "M" is montmorillonite.

Table 3. Fines content from NMR predictions and particle size analysis (PSA).

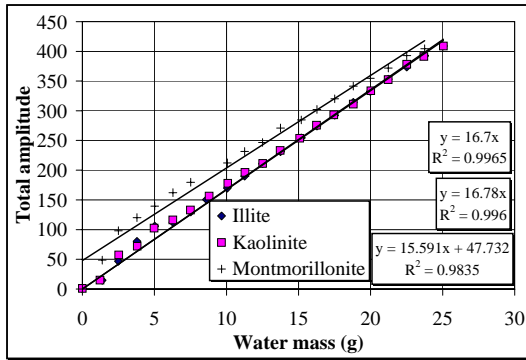| Solids Sample	Ore A	Ore B	Ore C	Ore 1	Ore 2	Ore 3	Ore 4	Ore 5
Fines content (illite) (wt%)	**7.52**	25.52	26.26	34.42	66.24	41.38	34.41	4.93
Fines content (kaolinite) (wt%)	8.22	23.02	23.58	25.68	43.57	26.56	29.60	4.38
Fines content (montmorillonite)	0.51	**2.42**	**2.51**	**1.38**	**3.39**	**5.25**	**3.54**	**0.44**
Fines (< 10 μm) from PSA (%)	7.03	1.53	1.34	1.35	4.88	2.88	3.66	1.01

Figure 1. Correlation between water mass and amplitude in clay/brine samples.
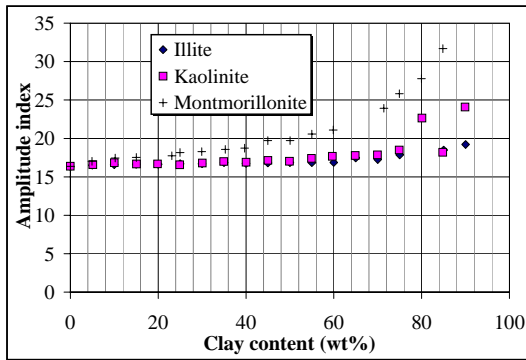

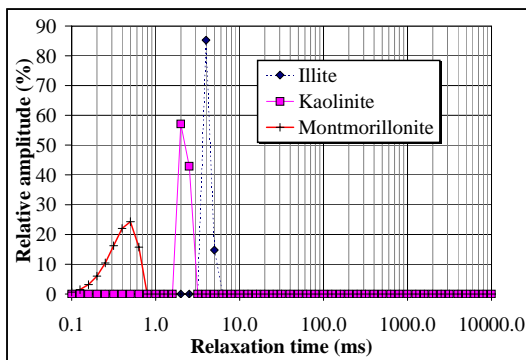Figure 2. AI as a function of clay content for clay/brine samples.

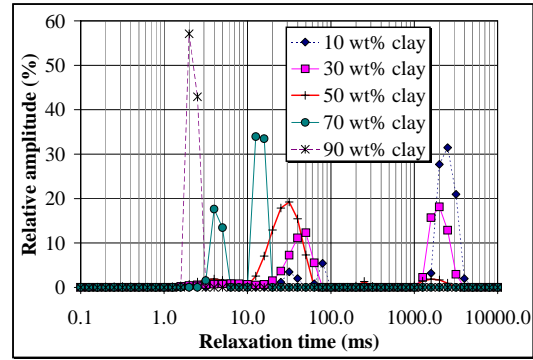Figure 3. NMR spectra of isolated clays. Each clay appears to have a characteristic relaxation time.

Figure 4. NMR spectra of kaolinite/brine samples. As clay content increases, the amplitude peaks appear at faster relaxation times and increase in size.
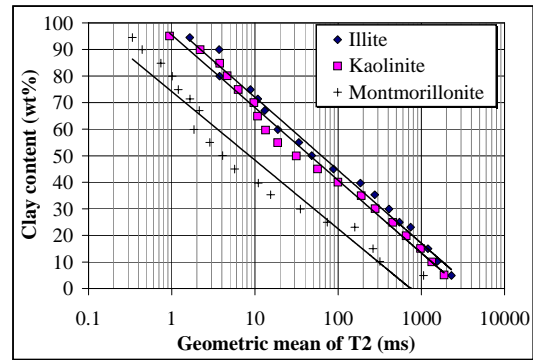

Figure 5. Correlation between T_{2gm} in clay/brine samples. Note that illite and kaolinite are similar, but montmorillonite consistently has lower T_{2gm} values at any clay content.
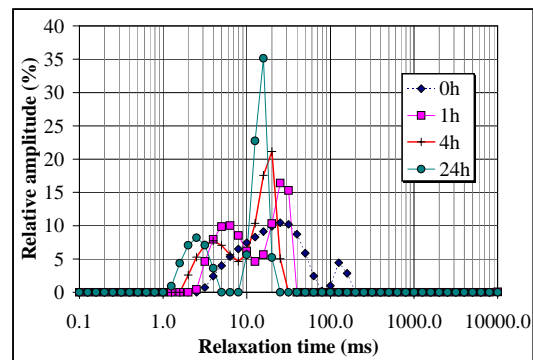
Figure 6. NMR spectra from ID30 after exposure to 9wt% brine.
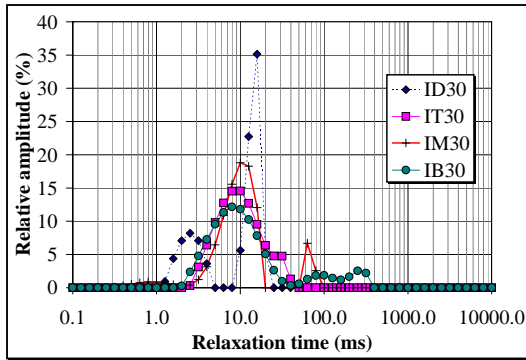
Figure 7. NMR spectra from samples with 30wt% illite after exposure to 9wt% brine.

Figure 8. NMR spectra from kaolinite/sand/brine samples after 24 h of exposure to 23wt% brine.

Figure 9. Clay content as a function of T_{2gm} for samples with illite dispersed in the solids phase.

Figure 10. NMR spectra from IT50 before and after the addition of 5wt% oil. One can see that clay bound brine relaxes at the same times as heavy oil.
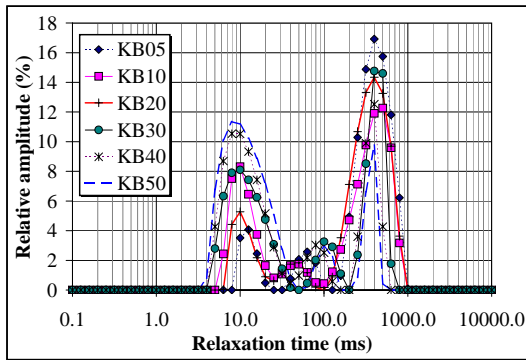

Figure 11. Relationship between T_{2gm} and clay content for samples with illite (IRC) and montmorillonite (MRC) at the top of the solids phase.
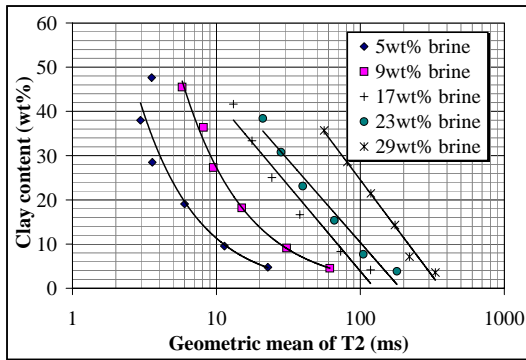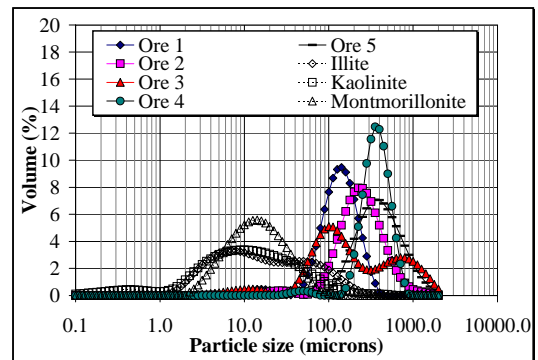

Figure 12. Particle size distribution for clays and solids extracted from Dean-Stark cleaning.