

# NMR INVESTIGATION OF INVASION PROCESS OF FORMATE MUD IN SANDSTONE CORES

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## ABSTRACT

This paper reports the results of using Nuclear Magnetic Resonance (NMR) to examine the invasion process of formate mud into sandstone core plugs, with emphasis on the timing of formate mud entering the Bulk Volume Irreducible (BVI) and further movement of the formate mud inside the samples. Two sets of experiments emulated the formate mud invasion occurring in water-bearing and oil-bearing formations, to gain insight on formate mud redistribution in the core samples. In both experiments, D<sub>2</sub>O brine was initially used to saturate the core plugs. In the first set of experiments, formate mud was injected into the samples under pressure. The T<sub>2</sub> spectra were measured immediately after the experiment to identify the pores in which the formate mud initially replaces /mixes with the original fluids. Subsequent NMR measurements monitor formate mud movement inside the core samples. The second set of experiments considered the fact that formate fluids are immiscible with oil and oil in the pore system would complicate the invasion process. To emulate the oil-zone invasion, D<sub>2</sub>O-brine-saturated core plugs were injected with mineral oil and subsequently injected with formate mud. NMR measurements were made during each step of the process to monitor the fluid movement.

Both sets of experiments show that formate invasion occurs at a very early stage, with formate mud replacing D<sub>2</sub>O inside the BVI fraction and D<sub>2</sub>O or/and oil in Bulk Volume Movable (BVM) fraction. This suggests that diffusion of formate mud among differently sized pores occurs rapidly after invasion starts. It is also noticed that formate mud moves into BVI preferentially many days after the invasion experiments in water zone, suggesting that it takes long time to reach a full equilibrium. It is anticipated that results of these experiments can help to develop porosity correction algorithm for NMR logs for wells drilled with formate mud.

## INTRODUCTION

Despite many engineering advantages of the solid-free formate mud, it can invade into the formation to a large extent. This poses problems for formation evaluation, as wells drilled with the solid-free formate mud do not have mud cake on the wellbore walls, and formate mud and water are miscible [1]. The invasion of formate mud into the formation alters the formation fluid composition and affects the NMR porosity measurements, in

addition to causing a possible shift of the NMR  $T_2$  spectrum and changes to BVI cutoff. Therefore, a detailed investigation of formate mud invasion is warranted.

The invasion process includes the drilling fluid entering into the formation and further redistribution to different pore sizes inside the formation. The process is affected by several factors including the pore size distribution, the original formation fluid constituents, and the physical properties of the formate mud. NMR provides a valuable technique to investigate the invasion process, as it accurately measures the fluid quantity in the formation. In addition, the NMR  $T_2$  spectrum reflects the pore size distribution. NMR, therefore, can monitor the fluid movement among pores with different sizes. This paper summarizes NMR results on core samples with formate mud invasion.

## **SAMPLES AND METHODS**

Four conventional core plugs, Z2, Z11, Z13 and Z26, were used in the study, all one inch in diameter and one inch long, from Tarbert and Ness formation in a well on the Norwegian Continental Shelf. The core plugs are medium-to-coarse grain, well-cemented sandstones. Three samples are uniform and Z13 is laminated. Examination under binocular microscope shows that quartz and feldspar are the main constituents of sand grains with minor or no clay minerals. In the laminated sample Z13, there is pyrite in the black lamination. Before the experiments begin, the samples are cleaned in a Soxhlet system with toluene and chloroform by a commercial laboratory.

The following fluids are used to saturate the samples or to replace fluid in the pore space of the samples: Saline  $D_2O$ ; saline water; formate mud of 1.85 g/cc and 2.15 g/cc, and mineral oil ET3000. The NMR  $T_2$  distribution spectra of the pure fluids are shown in Figure 1. The hydrogen indices (HI) for both formate muds are 0.59 and for ET3000 is 1.07.

Saturation of the samples is achieved with a vacuum method. The samples are inserted into a vacuum chamber and a degassed fluid is added to the chamber after full vacuum is reached. Invasion simulation tests are performed with a RCH-Hassler Type Core Holder at the Baker Hughes Fluid laboratory. The net weight gain after fluid saturation of a single fluid is used to calculate the porosity for the sample, after excessive fluid is carefully dabbed off with a wet Kimwipe.

### **NMR measurements**

CPMG sequence on a Maran 2-MHz spectrometer from Oxford Instruments was used to perform the NMR measurements at room temperature. The data are processed by software developed by Baker Hughes. NMR porosity values are in volumetric percentages (p.u.) after calibration to a cylindrical volume of one inch in diameter and one inch in length ( $12.87 \text{ cm}^3$ ). A fixed  $T_2$  cutoff at 33ms is used through this paper to separate BVI and BVM and is shown as a vertical line in cyan color in the figures.

## RESULTS

### Injection simulation in a water-saturated zone

Two core plug samples, Z11 and Z13, are initially saturated with D<sub>2</sub>O using the vacuum saturation method. Each core plug is then placed into the Hassler core holder and 1.85 g/cc formate mud is flushed through for 90 minutes at a pressure of 300 psi. No fluid is produced at the exit end of the core holder at the end of each injection experiment. After the injection test for each sample is stopped, NMR measurements are performed continually. Figure 2 shows the T<sub>2</sub> spectra of 3 hours and 144 hours after the injection of 1.85g/cc mud into the D<sub>2</sub>O-saturated samples Z11 and Z13, respectively. Please note that only a few spectra are shown in the graph, and there are many more NMR measurements in between.

### Injection simulation in an oil-saturated zone

Two core plug samples, Z2 and Z26, are first saturated with D<sub>2</sub>O using the vacuum saturation method. Each sample is then placed into the Hassler core holder and a mineral oil, ET3000, is flushed through for 90 minutes at a pressure of approximately 300 psi. This step simulates the replacement of water by oil in an oil zone. At this point, an NMR measurement is performed. The sample is replaced in the Hassler core holder and the 2.15 g/cc mud is injected. At the end of each experiment, NMR measurements are performed.

The NMR T<sub>2</sub> spectra of the two samples, Z2 and Z26, at various conditions are shown in Figure 3. The red lines show the T<sub>2</sub> spectra after oil injection and the blue lines show the T<sub>2</sub> spectra after formate mud injection subsequent to oil injection.

## DISCUSSION

During the injection experiments, saline D<sub>2</sub>O is used instead of saline water to view the NMR signals of the formate mud. Repeated saturation experiments were performed to show that the weight porosity values of saline water and saline D<sub>2</sub>O for the same sample remain essentially the same.

### Invasion in a water-saturated zone

Figure 2 clearly shows an obvious signal in the BVI for both samples, indicating that the formate mud enters BVI shortly after the injection before the first NMR measurement (3 hours after injection) is completed. It is likely caused by molecular movement in the form of diffusion. Since the diffusion coefficient of water at room temperature in free space is  $2.3 \times 10^{-9} \text{ m}^2/\text{s}$ , water molecules can move at a distance of 70  $\mu\text{m}$  in a second. The formate molecules are larger, thus would diffuse more slowly, but the diffusion speed is estimated to be within one order of magnitude relative to water.

For water zone simulation, both Z11 and Z13 show similar features and only Z11 is discussed here. Figure 4 shows cumulative T<sub>2</sub> spectrum of full water saturation (red line) and spectra of partial saturation by 1.85g/cc formate mud (3 and 144 hours after injection

in green and black lines, respectively), with the BVI/BVM cut-off line overlaid. All the cumulative spectra intersect the cut-off line, and the intersecting points, A, B, and C, show the volume of the measurable fluid inside the BVI. The Y value of Point A representing the BVI of the sample, and points B and C represent the volume of formate mud in BVI 3-hours (point B) and 144-hours (point C) after injection. The percentage (or saturation) of formate mud inside BVI after injection tests can be calculated from the Y values of points 'B' and 'C' divided by the Y value of point 'A'. Similarly, the fraction of formate mud in BVM can be calculated using the BVM of formate mud in the invasion experiments divided by the BVM of the saline water of the same sample.

Figure 5 shows the change with time of the percentage of formate mud inside BVI and BVM after the injection experiments. There is a discernible increasing trend of formate in BVI and a decreasing trend of the formate fraction in BVM of the sample. It should be noted that the percentage of formate mud in BVI increases from approximately 50% to 60%, but the percentage of formate mud in BVM decreases from approximately 45% to 25%. This is due to the fact that the BVI (about 8%) is double the BVM (4.4%) in this sample. The total volume of the formate mud remains unchanged as the sample is seated in a tightly sealed container with no visible loss of liquid during the measurements. This is confirmed by the weights of the sample before and after the measurements.

The increasing trend of formate mud in BVI and decreasing trend of formate mud in BVM of the sample Z11 seem linear as shown in Figure 4R. The correlation coefficients of the line fitting are around 0.6 as indicated by the wide scattering of the points along the line. This is likely due to NMR instrument error. Despite the scatter of the points, it appears that formate mud tends to concentrate in the irreducible volume by moving from the bulk volume. The movement of formate mud into the BVI results in a separation of the fluid inside the sample; i.e., the composition of the fluid in BVI becomes different from that in BVM. It is not quite clear what mechanism caused this separation of formate mud and water, more work is needed to determine the mechanism.

### **Invasion in an oil-saturated zone**

For oil-zone simulation, the mud invasion experiments show that the formate mud entered both the BVI and BVM pore space in an early stage as shown in Figure 3, indicated by the peaks around 8 ms on the blue lines. The formate mud movement from BVM into BVI can also be explained by the diffusion process.

However, the formate mud invasion in an oil zone seems complicated. Oil stays in BVM only in sample Z2, whereas oil stays in BVI and BVM in sample Z26. More work is needed to fully understand the cause of this.

## **CONCLUSIONS**

Our experiments show that formate mud invades into both BVM and BVI in a very early stage in the brine-saturated cores (i.e. water zone) and brine-oil saturated cores (i.e. oil

zone). The diffusion process of molecules seems to explain the fast mixing of formate mud and the formation water.

It appears the formate mud tends to move to small pores even many days after the injection experiments in brine-saturated cores. The exact cause of the separation of the fluid in the studied samples needs more studies to determine.

For formate mud invasion in water/oil saturated rock sample, the situation is complicated and more work is needed.

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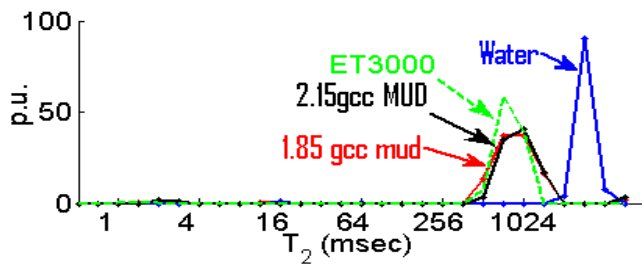


Figure 1: The NMR T<sub>2</sub> distribution of saline water (blue line), formate mud with 1.85 g/cc (red line) and 2.15 g/cc (black line), and ET3000 (green dashed line)

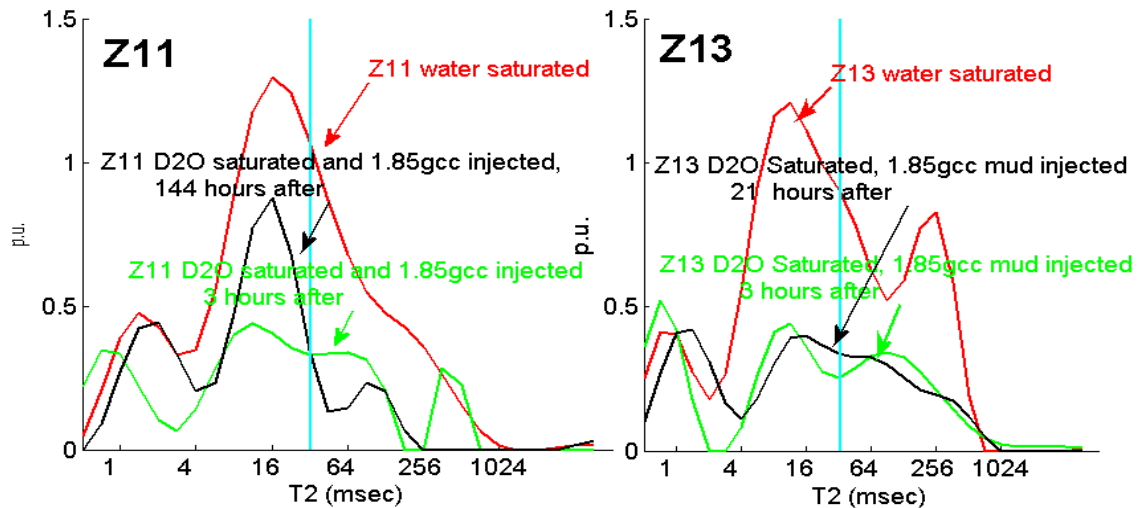


Figure 2. Incremental T<sub>2</sub> spectra acquired at different times after 1.85g/cc formate mud flooding for sample Z11 and Z13. The spectra for saline water-saturated samples are also drawn for reference. The total porosities of Z11 and Z13 are 12.4 p.u. and 13.5 p.u., respectively, from saturation tests.

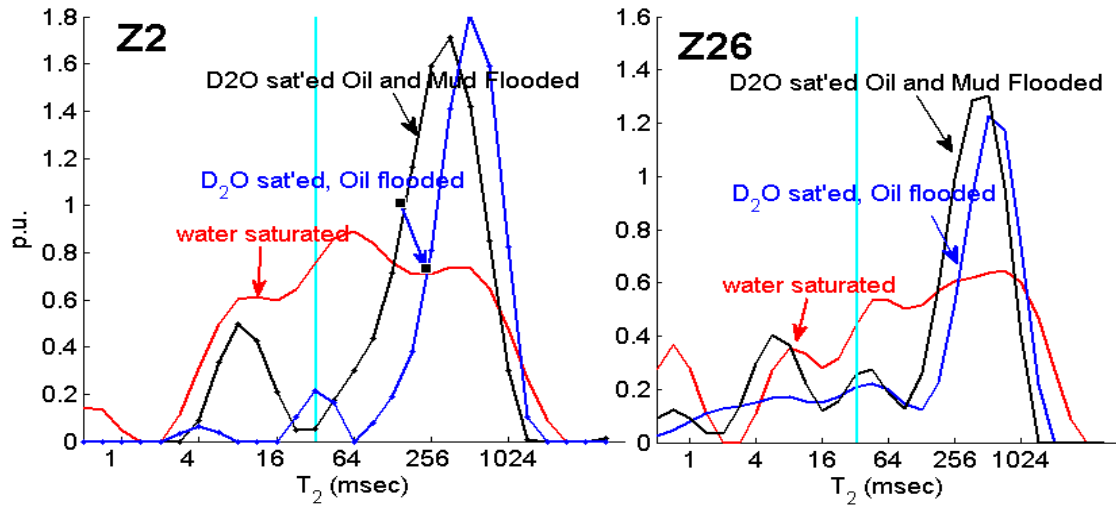


Figure 3.  $T_2$  spectra after formate mud injection into previously oil-injected samples and Z26). Both samples show that formate mud enters into the BVI and BVM. The porosity of Z2 is 12.6 (BVI 4.1 + BVM 8.5) and of Z26 is 9.7 p.u. (BVI 2.9 + BVM 6.8).

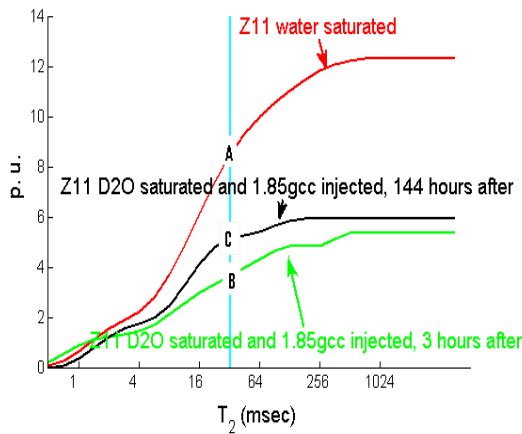


Figure 4. Cumulative  $T_2$  spectra acquired at 3 hours (green line) and 144 hours (black line) after 1.85g/cc formate mud injection for sample Z1.

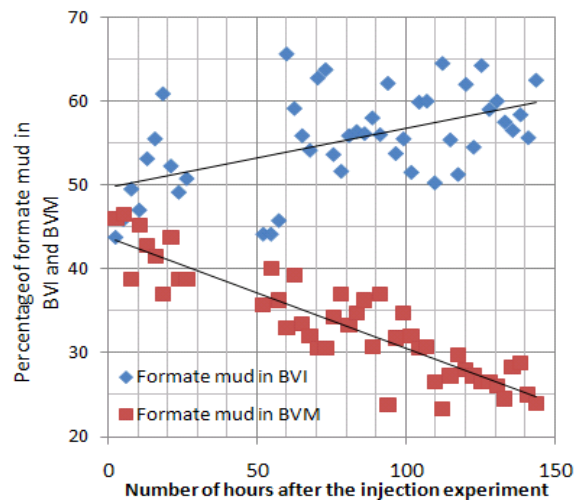


Figure 5. Percentage of formate mud in BVI and BVM versus time.

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