

APPLICATION OF NMR- AND CT-ANALYTICAL METHODS TO ASSESS THE FORMATION DAMAGE POTENTIAL OF DRILLING FLUIDS

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ABSTRACT

The residues of a drilling fluid in a near-wellbore environment can reduce the hydrocarbon delivery potential of a reservoir substantially, if they are not easily removed. In this case a fluid is formation damaging. The assessment of the formation damage potential of drilling fluids prior to field application is an important task for petroleum engineers.

To gain comprehensive information on the damage potential of drilling fluids, an analytical methodology was applied that combines modern, non-destructive, petrophysical analysis methods and drilling fluid exposure experiments under close to reservoir conditions.

Flooding experiments using aqueous solutions of polyanionic cellulose, xanthan and polyglycol were performed on hollow-cylinder sandstone samples (Berea, $k_a = 80 \text{ md}$). The analytical methods applied were Nuclear Magnetic Resonance Relaxation time measurements (NMRR), NMR-Imaging (NMRI) and X-Ray Computed Tomography (CT). Core samples were analyzed prior to the experiments, after exposure to the solutions of the drilling fluid additives, as well as after backflooding with crude oil.

Relaxation time distributions show that the solutions of the drilling fluid additives have substantially different NMR-relaxation time characteristics than the fluids in the rock prior to exposure. The solutions occupy pore spaces or may even accumulate in the rock. Measurements after the backflooding experiments show that changes are in some cases permanent.

The tomographic methods, especially the NMRI, show the spatial distribution of fluids in the rock samples. Two-dimensional images and "relaxation time weighted porosity profiles" quantify aspects of the filtration characteristics of the different additives. NMRI, furthermore, allows visualizing the formation damage potential of the additives. Local distributions of polymer residues are identified by the measurements after the backflooding experiments.

The information gained in this study is considered to be highly important for the evaluation of formation damage mechanisms and drilling mud design, as well as the evaluation of petrophysical rock properties by well logging. Also, applications to gain a better understanding of EOR-operations (gel-injections, polymerfloods, etc.) are conceivable.

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INTRODUCTION

Polyanionic cellulose (PAC), xanthan and polyglycol are common components of non-dispersed water-based drilling fluids (Reid, 1992). The primary tasks of these additives are, respectively, filtration control, viscosification and shale inhibition.

Filtrate losses to a formation during drilling cause fluid components, such as polymers or glycol, to penetrate a near-wellbore zone. When producing from the formation, residues of the additives may be retained in the porous network and may impair production. A fluid component that reduces the delivery potential of a reservoir is “formation damaging”.

To improve evaluations of formation damage in the laboratory a methodology based on the application of non-destructive, petrophysical analysis methods was presented in an earlier work (van der Zwaag et al., 1997). Essentially, an integrated approach to evaluate rock samples exposed to drilling fluids was suggested. The methodology includes two NMR-techniques (NMR-relaxation time measurements or NMRR and NMR-Imaging or NMRI) and X-Ray Computed Tomography (CT) in combination with drilling fluid exposure experiments. It was shown, among other things, that a mud exposure experiment performed on a high permeability ($k_a \approx 1500$ md) Berea sandstone with a water-based KCl/Polymer-fluid introduced a polymer-containing filtrate and mud solids (barite) into the water-saturated rock sample. Backflooding with brine removed the polymers completely, while a 2-mm thick internal filtercake consisting largely of barite was retained in the rock. Solids invasion and the creation of a permanent internal filtercake were identified as the formation damage mechanisms.

However, when drilling into a reservoir zone, drilling fluids encounter partially oil-saturated rocks. Also, during production, an invasion zone created by the mud components will be back-produced with oil. The test conditions described in van der Zwaag et al. (1997) are therefore only of a limited value since brine was used as the medium to saturate and backflood rock samples. Also, brine probably had a major impact on the complete removal of the water-soluble polymers.

The objective of the present study was to characterize the formation damage potential of the three drilling fluid additives mentioned above and to demonstrate the potential of the analytical methods on oil-saturated rock.

The experiments set a focus on investigations of drilling fluids. Yet, the methodology can be adapted to investigations for example of stimulation or frac-fluids and of EOR-fluids.

MUD EXPOSURE EXPERIMENTS AND NON-DESTRUCTIVE ANALYSES

The experimental plan consisted of:

1. Baseline measurements on brine-saturated rock samples, pore fluids (brine and crude oil) and the aqueous polymer solutions.
2. Immiscible displacement of brine by crude oil at room temperature;
3. Baseline permeability measurements with crude oil;

4. Polymerfloods;
5. Backfloods with crude oil;
6. Return permeability measurements with crude oil.

Steps 3 through 6 were performed at a test temperature of 80°C. NMRR measurements, NMR Images and CT were performed in four different saturation states under ambient conditions after dismounting the core sample from the holder:

1. at water saturation,
2. at crude oil saturation and initial water saturation,
3. after the polymerfloods,
4. after backflooding with crude oil.

Measures were taken to prevent any evaporation of the pore fluids during the different analyses.

Characterization of Fluids and Rock Samples

In the present study we investigated the formation damage characteristics of the following three drilling fluid additives as individual components in brine solution:

1. Polyanionic cellulose
2. Xanthan
3. Polyglycol

The solutions were prepared in a KCl-brine using concentrations typical of those used in field muds (see Tab. 1).

A formation brine composition representative of a North Sea sandstone reservoir was selected to saturate core samples (see Tab. 2). The crude used in the investigations was untreated separator oil from the Oseberg C platform.

A low permeability Berea sandstone ($k_a=80$ md) was selected for the experiments. Hollow cylinder core plugs (60 mm outer diameter, 12 mm inner diameter) were drilled, using the formation brine as a drilling fluid. After drilling, samples were dried at 60°C to constant weight and re-saturated with the filtered and degassed formation brine under vacuum.

Polymer Floods and Permeability Measurements

For fluid exposure experiments, an apparatus as described in van der Zwaag et al. (1997) was used. The set-up is characterized by a radial drainage geometry and the option to perform exposure experiments under dynamic filtration conditions. Exposure experiments were performed as in polymer floods at constant injection rates, and not under constant differential pressure conditions as described earlier.

After mounting the core plug sample in the core holder, an immiscible displacement experiment to establish initial oil saturation was run in the backflooding direction (from the outer boundaries to the inner hole of the hollow cylinder sample) at a flow rate of 10 ml/min for 1 hour. A total volume of approximately 20 PV crude oil was then flooded at low rate (0.5 ml/min) and at test temperature (80°C) over night. Baseline permeability, $k_o(S_{wi})_{80^\circ\text{C}}$, was calculated based on differential pressure measurements for four flow rates in the backflooding direction and a correction for dynamic offset was made.

The polymer floods were performed at 80°C at a constant injection rate (1 ml/min). A confinement pressure of 125 bar was selected and a pore pressure of 100 bar was adjusted. Polymer solutions were injected for 2 hours at static conditions and for 10 hours at dynamic filtration conditions (circulation rate: 1.84 l/min).

For the PAC and the polyglycol solutions a total of 50 PV was flooded through the core samples. While flooding the solution containing xanthan caused problems with the injection pump after 2 hours. The experiment had to be stopped after injecting a total of 9 PV of polymer solution.

The backflooding experiment was performed under the same conditions as the immiscible displacement experiment except that a temperature of 80°C was selected. Again, approximately 20 PV of crude oil were flooded at low rate (0.5 ml/min). The return permeability was calculated from measurements equivalent to the baseline measurements.

Non-Destructive Petrophysical Analytical Techniques

Nuclear magnetic resonance (NMR) and X-Ray Computed Tomography (CT) provide information on petrophysical properties non-destructively. The methods are sensitive to different rock or fluid properties. A comparison of the measurements on a rock sample in different saturation states may help to identify changes in the petrophysical properties introduced by the fluid additives. This allows an explanation of the formation damage characteristics of an additive based on its influence on petrophysical properties.

NMR Relaxometry (NMRR): NMR studies on core samples are used to characterize rock properties as porosity, pore size distributions and wettability (e.g. Howard et al., 1993; Howard and Spinler, 1993; Straley et al., 1994; Stallmach et al., 1996). Due to the molecular interaction of the pore fluids with the grain surfaces, the longitudinal (T_1) or transverse (T_2) relaxation times of the ^1H NMR signal originating from the pore fluids are significantly reduced in comparison to the relaxation time characteristics of the bulk fluids. Depending on the relaxation time under investigation, the time dependence of the NMR signal decay is observed. The total NMR signal is proportional to the total pore volume and can be used to determine total rock porosity. For fluids interacting with the rock surfaces, the relaxation times are proportional to the pore volume-to-surface ratio. Consequently, the RTD represent the rock pore size distribution. Non-interacting fluids, e.g. non-wetting fluids, will appear in the RTD at their bulk relaxation times.

For the NMRR-analyses the apparatus and the procedures described in van der Zwaag et al. (1997) were used. Only the (T_2)-relaxation time distributions (RTD) of the core samples in the different experimental phases were evaluated.

NMR imaging (NMRI): NMRI studies are spatially resolved measurements of NMR parameters (intensities, frequencies, relaxation times) using inhomogeneous time dependent magnetic fields superimposed to the static magnetic field of conventional NMR. Barrufet et al. (1992) and Fordham et al. (1995) presented earlier applications of NMRI in petrophysical studies. The present study investigates the spatial distribution of fluids and, hence, of porosity as a function of the saturating fluids. Two-dimensional (2d) presentations of the fluid distribution in a plane oriented parallel to the radial drainage direction of the hollow cylinder rock samples provide information on rock/fluids interaction.

A 2d non-slice selective spin echo NMRI technique was applied. The 2d map of the fluid (porosity) distribution was obtained after a 2d Fourier transformation of a set of spin echo signals. The fluid distribution maps, however, do not represent the total amount of fluids. T_2 -relaxation suffers an unavoidable reduction of signal intensities. The effect is demonstrated in Figs. 1 and 2 by a comparison of the complete relaxation time curves and the calculated relaxation time weighted RTD. The signal from small relaxation times is lost. Thus, NMRI measures the spatial distribution of fluids and porosities with large relaxation times. The quantity measured by the 2d-spin echo NMRI sequence is termed “relaxation time weighted (RTW) porosity”.

To simplify quantitative analysis the 2d RTW porosity maps were transformed into so called “1-d radial RTW porosity profile” by averaging all image points having the same radius from the center of the sample. The profiles can be compared for the different flooding states, yielding, for example, a direct measure for the penetration depth of mud components.

X-Ray Computed Tomography (CT): CT spatially resolves the X-Ray absorption coefficient $\mu(r)$ of the sample. For equal chemical compositions, $\mu(r)$ depends only on the density $\rho(r)$ of the sample material. Sample regions differing in their chemical composition exhibit different X-Ray absorption. The quantity controlling this process is the effective atomic number Z_{eff} of the material. Absorption increases strongly with the effective atomic number (Vinegar and Wellington, 1987; Wellington and Vinegar, 1987). Density variations as well as spatial variations in the chemical composition of a rock sample are reflected in 2d CT-images by intensity contrasts. In formation damage studies (van der Zwaag et al., 1997), CT-images are used to analyse such contrasts. For example, the penetration of high density materials (as barite $BaSO_4$, $Z_{eff} \gg 48$) yields a significant image intensity enhancement in a sandstone rock matrix (quartz, SiO_2 , $Z_{eff} \gg 12$).

In this study, two CT-images (radial slices, slice thickness 2 mm, distance 20 mm) were recorded after each flooding state at exactly the same sample positions using the technique presented in van der Zwaag et al. (1997). For quantitative analysis, both images were averaged. Subsequently, radial profiles of the image intensities were calculated. These “1d CT number profiles” represent a measure of the radial dependence of the X-Ray absorption coefficient in the rock sample.

RESULTS

Baseline Investigations on the Fluids and Rock Samples

Baseline T2-NMRR measurements and Hydrogen Index measurements were performed on all fluids. Values are presented in Tab. 3. A rock description including baseline core analysis measurements and NMRR and NMRI data is presented in Tab. 4 for the three rock samples used for the experiments (B.7.1, B.8.1, B.9.1).

Crude oil has, in contrast to the aqueous solutions, no single-valued bulk relaxation time, but is characterized by a relaxation time distribution (RTD). This is because crude oil consists of components with different molecular masses.

Immiscible displacement of the brine in the pore spaces by crude oil causes changes in the RTD (Figs.1,2,5,8). Oil reduces the pore space available for brine and the relaxation times (RT) of the brine phase are smaller after displacement. At the same time, additional components at larger RT originating from the oil can be detected. However, these RT components occur at shorter RT than the RTD of the bulk oil. This points towards the fact that the oil components interact with the rock matrix. It appears that the rock is partially oil-wet after the displacement experiment.

The 2d-NMRI show spatially homogeneous distributions of the RTW-porosity (Figs.4,6,9). The 1d-profiles indicate smaller porosity values in the vicinity of the center hole especially for sample B.9.1 (Fig.9). This may be caused by air that entered the sample unintentionally through evaporation or by small differences in the local porosity distribution between sample B.7.1 (baseline water-saturated) and B.9.1. The 2d-CT images and the radial CT-intensity profiles (Fig.11) show that the differences between the water and the oil-saturated sample (at initial water saturation) are insignificant.

Return Permeabilities

Measurements of the return permeabilities show that the PAC and the xanthan solutions reduce the oil permeability significantly, while the return permeability of the sample flooded with the polyglycol-solution is higher (Tab.5).

Polyanionic Cellulose, Sample B.7.1

After the flooding with the PAC-solution, the oil saturation of the sample is reduced. It becomes evident that components at larger RT originating from the oil are removed (Fig.1). An apparent porosity loss of 1.8 % can be calculated (cf. Tab.4,5). The RTW-porosity profile (Fig.3) shows a strong porosity reduction in the vicinity of the center hole, i.e. at the inflow area. The RTW-porosity then increases with the radius, approaching the baseline value at 10-15 mm. Darker zones in the 2d-image (Fig.4) show the porosity loss.

Backflooding the sample with crude makes oil components at long RT visible in the RTD (Fig.1). Yet, the initial saturation is not re-established. The RTW-porosity profile shows that the RTW-porosity remains low in the vicinity of the center hole. It appears that the oil-saturation after backflooding is clearly below the baseline value in this area.

Glycol, Sample B.8.1

Flooding with the glycol-solution reduces the oil saturation and causes a dramatic change in the RTD (Fig.5). An apparent porosity loss of 6.5% is observed (comp. Tab.4,5). The RTW-porosity is homogeneously reduced over the whole radius of the sample (Fig.6,7).

Backflooding with crude approximately re-establishes the baseline saturation (Fig.6). The total apparent porosity reduction after glycol-flooding and backflooding is relatively small (1.7%, comp. Tab.4,5). Oil components reappear in the RTD (Fig.5). An additional RT-component appears at long RT. Alterations in the RTD at short RT may indicate that the RT behavior of the residual water phase existing prior to the experiments is influenced by the polyglycol, i.e. the residual water and glycol mix during the experiment. However, the 2d-NMR Image (Fig.7) and the RTW-porosity profile (Fig.6) show that flooding with the glycol solution creates only temporary alterations. The flood does not influence the initial saturation conditions prevailing in the larger pores.

Xanthan, Sample B.9.1

The xanthan solution causes an efficient displacement of the oil phase over the whole sample radius and an equally dramatic alteration in the RTD and the RTW-porosity as the glycol (Figs.8,9). The apparent porosity is reduced by approximately 6% compared to the baseline value (Tab. 4 and 5).

After backflooding with oil smaller alterations in the RTD become visible (Fig.8), yet, the area under the RTD curve (apparent porosity) remains nearly unchanged compared to the situation after the polymer flood. Only minor changes can be observed in the RTW-porosity profile (Fig.9) and 2d-images (Fig.10). It appears, that it is impossible to re-establish the initial oil saturation under the given backflood conditions.

CT-Measurements

The CT-measurements on sample B.7.1 shown in Fig.11 are representative of the observations made on the other two samples. The 1d-CT intensity profiles show only insignificant contrasts between the samples in the different experimental phases. Differences are within measurement error. Due to similar densities and effective atomic numbers of the test fluids, the brine and the oil, fluid displacement is much harder to detect by CT measurements than by NMR. CT was shown to be valuable for solids damage (van der Zwaag et al., 1997) and may be enhanced by adding tracers.

Discussion

Polyanionic celluloses (PAC) are additives that can be used as filtration control agents or viscosifiers in drilling fluids. The product used in this study is primarily a filtration-control agent. The task of the additive is to support the creation of an impermeable filter-cake by physical and chemical interaction with the mud solids.

The experiments and analyses show that PAC is filtered out in the vicinity of the inflow area and accumulates in the near-borehole zone. The viscosity of the water in the pores is increased and affects the T_2 -relaxation times. RTs are smaller than in the oil or brine satu-

rated case. Backflooding is ineffective in removing the polymer residues. The return permeability of 39% quantifies a significant formation damage potential. The reduction of the oil permeability is most probably caused by a combined effect of reduced oil saturation, reducing the relative permeability to oil, and blocking of flowpaths by precipitated material.

In field application PAC will interact with other mud components (barite, CaCO_3 or drill solids) and establish a low permeability filtercake that reduces filtrate losses to the formation. Invasion volumes and invasion depth of the PAC may therefore be smaller than for the polymer solution. Invasion may be limited to a mud spurt-loss phase.

Polyglycols are of low molecular weight. They have several functions in WBM and are primarily used for shale inhibition. The cloud point temperature of the polyglycol (e.g. Bland et al., 1995) used in this study is above 100°C when dissolved in the KCl-solution used for fluid preparation. Hence, the polyglycol used is under the given conditions completely water-soluble.

The measurements show that glycol can be largely removed by backproducing a formation. It is at least displaced in the larger pores that mainly affect permeability. Glycol is therefore non-damaging. This result is confirmed by the good return permeability of approximately 80%. The reduction of 20% may be related to smaller increases in the initial water saturation.

Xanthan is completely water-soluble and is predominantly used as a viscosifier in WBM. Also, xanthan-solutions are widely used in EOR-projects. The polymer gives brine a high viscosity, especially at low shear rates, and is effective at low concentrations.

The present experiments show a strong reduction in the oil relative permeability by over 50%. This reduction is related to a dramatic increase in the water saturation. The initial brine is replaced by the xanthan solution. The effective displacement of oil when injecting xanthan is demonstrated. However, the reverse displacement of xanthan by oil is much less efficient because the crude oil viscosity at the test temperature is much less than the viscosity of the xanthan solution. Xanthan is, based on the present test results, formation damaging if it enters deep into the formation. The mechanism discussed remains speculative, but if true, would apply to any water-soluble polymer that produces highly viscous filtrates. A closer investigation of the effect within field application is therefore strongly recommended.

CONCLUSIONS

The present study characterizes the potential impact of common water-based mud additives (polyanionic cellulose, xanthan and polyglycol) on the hydrocarbon delivery characteristics of a sandstone formation. Laboratory experiments performed on oil-saturated (at initial brine saturation) Berea sandstone samples create the basis for the conclusions.

It was found that:

- Flooding oil-saturated rock samples (Berea, $k_a=80$ md) with solid free, aqueous solutions of the drilling fluid additives reduces the initial oil saturation of the rock samples.
- A glycol solution injected into the rock material is removed through backflooding with crude oil. Alterations in the oil permeability are small.
- Solutions of polyanionic cellulose (PAC) and xanthan remain, to a large degree in the pore system after backflooding and cause a significant reduction in the oil saturation compared to the initial situation.
- PAC is filtered out in the vicinity of the inflow area and accumulates in a near-bore-hole zone. Two formation damage mechanisms are likely; a) a reduction of the relative permeability of oil through an increase in the saturation of brine/polymer solution, b) blocking of flow paths by precipitated material. In field application PAC will probably perform differently due to stronger interaction with mud solids.
- Xanthan displaces oil efficiently and, in contrast to the PAC, distributes evenly in the rock. However, when backflooding with oil, only an inefficient displacement of the xanthan by oil is observed. The permeability to oil is strongly reduced after the polymer flood. The formation damage mechanism behind the permeability reduction is most likely an increase in the brine saturation of the rock. The observed damage mechanism requires further investigations aiming towards field applications.
- The results of this study confirm the value of the suggested methodology and prove its application on oil-saturated rocks.

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Table 1. Composition of the Test Fluids

FLUID SYSTEM		PAC	Xanthan	Polyglykol
Commercial name		Celpol ESL	Flowzan	Glydriil MC
Supplier		Metsa Speciality Chem.	Drilling Specialities Inc.	Anchor/MI Drilling Fluids
Composition	Unit			
Fresh water	l	25	25	25
KCl	g	556	556	556
Xanthan	g		75	
PAC	g	300		
Polyglykol	ml			750

Table 2. Composition of the Formation Brine

Component	Conc. (g/l)
NaCl	26.78
CaCl ₂ *2H ₂ O	1.61
MgCl ₂ *6H ₂ O	0.50
SrCl ₂ *6H ₂ O	0.33
KCl	0.76
BaCl ₂ *2H ₂ O	0.36
FeCl ₂	0.01
NaHCO ₃	0.96

Table 3. NMRR Baseline Measurements on Fluids.

Fluid	T2 (ms)	HI
Brine	2700	1.00
Crude	<326> ¹	1.10
PAC	2620	1.04
Polyglykol	2320	1.07
Xanthan	1800	1.15

¹the symbols "< >" indicate that crude oil has no single-valued bulk relaxation time, but is characterized by a RTD. The value of 326 ms is the mean value of the RTD.

Table 4. Baseline Analyses on the Rock Material

	B7.1	B8.1	B9.1
Pore volume (ml)	21.00	20.85	20.72
Bulk volume (ml)	118.74	118.36	120.04
Porosity	17.69 %	17.62 %	17.26 %
Grain density (g/l)	2.67	2.67	2.67
<u>NMR Data / water saturated</u>			
Porosity	19.94 %		
Mean log T2 (ms)	23.20		
RTW porosity (NMR Imaging)	9.85 %		
<u>NMR Data / oil saturated</u>			
Porosity (signal intensity)	18.26 %	18.99 %	18.33 %
mean log T2 /ms	23.56	24.84	24.19
RTW porosity (NMR Imaging) / %	9.79	10.20	9.82
<u>On separate 1" samples</u>			
Air permeability (Klinkenberg corrected) (md)	84.0	80.9	

Table 5. Return Permeability Measurements and NMR-Results on the Rock Samples in Different Saturation States.

	B7.1	B8.1	B9.1
Test Fluid	PAC	Glycol	Xanthan
Baseline oil perm., $k_o(S_{wi})_{80^\circ C}$ (md)	44.3	65.9	59.5
Return oil perm., $k_r(S_{wi})_{80^\circ C}$ (md)	17.5	50.9	25.9
Return oil perm. (%)	39 %	77 %	44 %
<u>NMR Data / post flood</u>			
porosity (signal intensity)	16.51 %	12.52 %	13.75 %
mean log T2 / ms	16.88	12.97	14.12
RTW porosity (NMR Imaging) / %	9.8	5.21	5.98
<u>NMR Data / backflow</u>			
porosity (signal intensity)	17.84	17.29	13.28
mean log T2 / ms	22.91	26.52	16.47
RTW porosity (NMR Imaging) / %	9.38	9.82	6.04

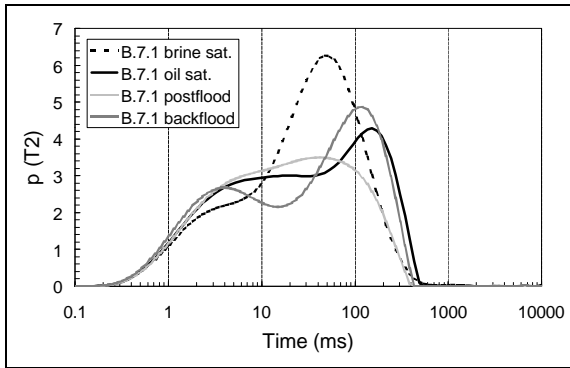


Fig.1. NMR Relaxation Time Distributions (RTD) of Sample B.7.1 (PAC).

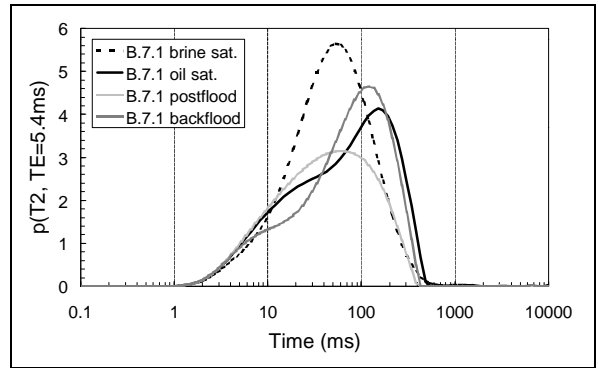


Fig.2. Relaxation Time Weighted (RTW) RTD on Sample B.7.1.

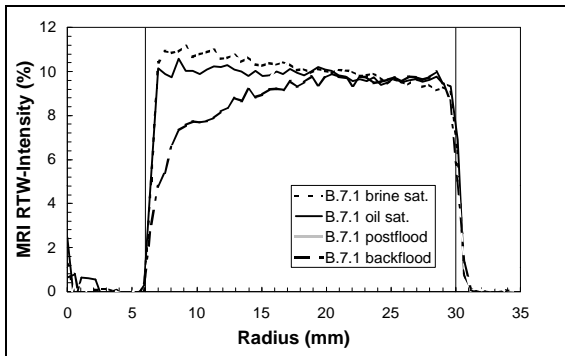


Fig.3. 1d RTW-Porosity Profiles of Sample B.7.1 (PAC).

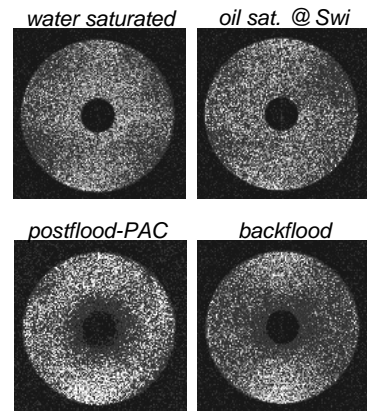


Fig.4. RTW-2d NMR Images on Sample B.7.1 (PAC).

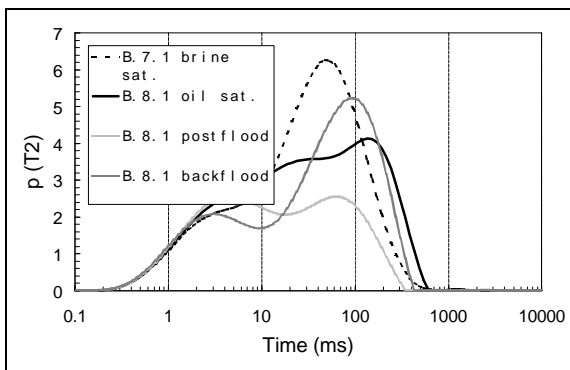


Fig.5. NMR RTD on Sample B.8.1 (Glycol).

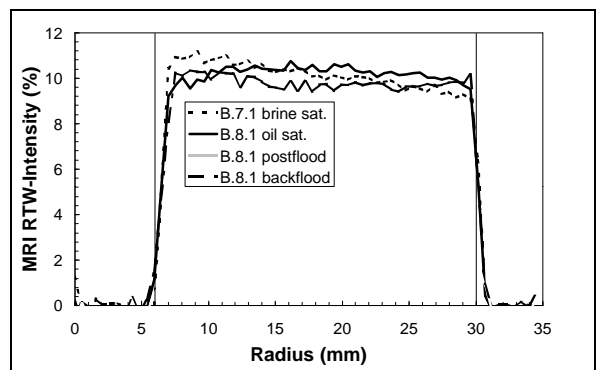


Fig.6. 1d RTW-Porosity Profiles of Sample B.8.1 (Glycol).

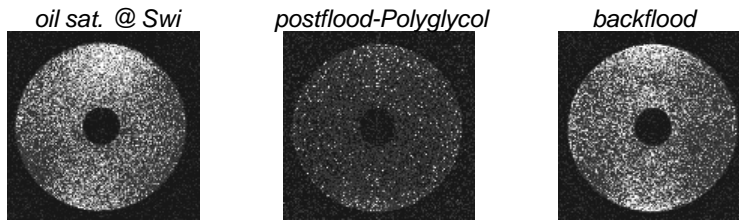


Fig.7. RTW-2d NMR Images on Sample B.8.1 (Glycol).

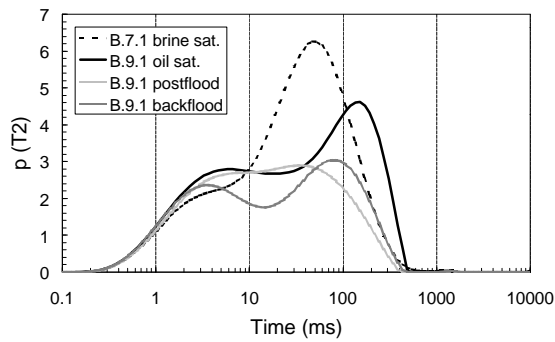


Fig.8. NMR RTD on Sample B.9.1 (Xanthan).

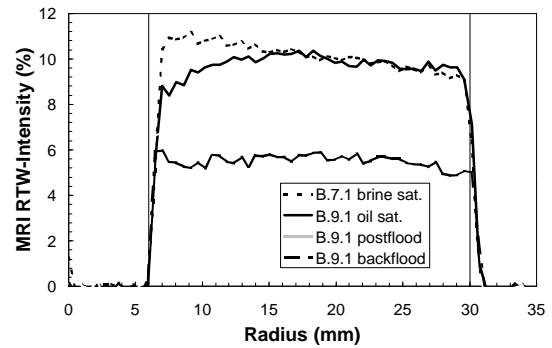


Fig.9. 1d RTW-Porosity Profiles of Sample B.9.1 (Xanthan).

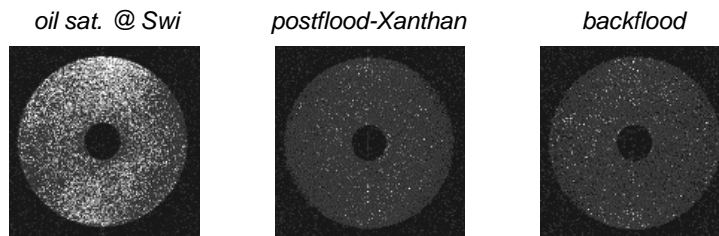


Fig.10. RTW-2d NMR Images on Sample B.9.1 (Xanthan).

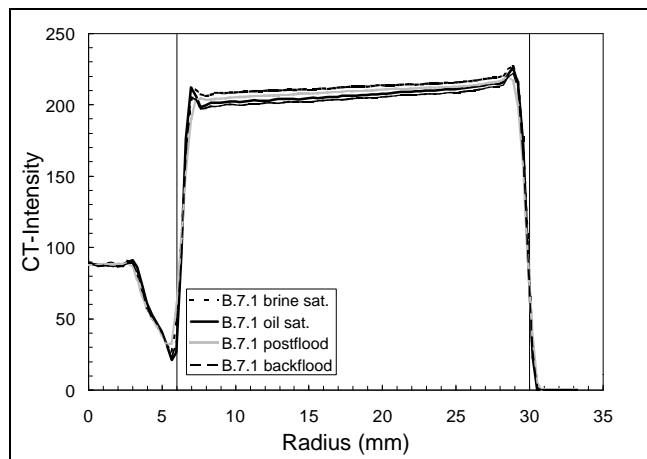


Fig.11. 1d-CT Intensity Profiles of Sample B.7.1 (PAC) in Different Saturation States