# ANALYZING MICROBIAL IMPROVED OIL RECOVERY PROCESSES FROM CORE FLOODS

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## ABSTRACT

Several mechanisms have been proposed to account for the increased oil recovery observed as a result of Microbial Improved Oil Recovery (MIOR) processes, both in laboratory experiments and field cases, but few of these mechanisms are verified. One of the reasons may be that these systems include living bacteria that ideally should be kept in a growing state during the investigations. This paper presents experimental results and a literature review where mechanisms are discussed, with main focus on changes in interfacial tension (IFT), wettability and permeability reduction during MIOR experiments. Special emphasis is put on wettability changes in a laboratory study of an MIOR core. The wetting conditions are studied by using an environmental scanning electron microscope (ESEM) that has been improved by modifying the cryogenic vacuum system for sample preparation. The ESEM equipment enables qualitative and semi-quantitative investigations of wettability changes after a core flood. The saturation profile in an MIOR core should show where the incremental oil is mobilized. An alternative technique for saturation determination is therefore presented, which is based on image analysis from electron images acquired in the same process as the wettability investigations. The number of cross sections with saturation estimations in this study is too few to make a reliable oil saturation profile (e.g., compared to a computer tomography scan). However, the average saturation from the images is close to the saturation calculated from the flooding process. The results of the study indicate that only small parts of the pore space have changed from their original strongly water-wet condition to a more oil-wet state.

## INTRODUCTION

MIOR has shown significant potential for improving the recovery factor and should therefore be considered for implementation in producing oil fields and also in reservoir development plans. The technology is very cost efficient in a field case due to several reasons: When a water injection system is in use, the MIOR process can be initiated without major changes, thus the investment costs are low. Further, the use of biocides to limit the growth of bacteria producing $H_2S$, and thereby causing reservoir souring, can be reduced since the MIOR process stimulates the growth of other bacteria which suppresses the growth of the $H_2S$ producing strains [1]. Portwood [2] reviewed 322 MIOR projects and calculated a cost of $0.25-0.50 per barrel of oil, again making this technology desirable in IOR projects. The process has been proven efficient in both carbonates [3] and sandstone reservoirs [4].

Beckman was the first to discover that bacterial growth can result in increased oil recovery in 1926 [5], and ZoBell performed detailed studies on this in the 1940's [6]. Many successful field trials have been reported since then [7, 8]. However, the mechanisms behind the improved recovery are still poorly understood. Many mechanisms are proposed, but few of them have been confirmed. Frequently suggested mechanisms are the reduction of IFT, changes in wettability and partial blocking of pores. Oil degrading bacteria need access to the oil phase, and therefore they produce a biosurfactant to reduce the IFT between water and oil. It is, however, necessary to significantly reduce IFT to achieve the low residual oil saturation reported in some core flooding experiments [4]. In a water-wet core a reduction of IFT by 3-4 orders of magnitude may be required, and such low measurements have recently been reported [9]. The MIOR process may also cause changes in wettability which may have an important impact on the residual oil saturation. A change from water-wet to mixed wet conditions can result in a significant reduction in the residual oil saturation in an extended water flood. This is known as film-drainage [10] where the rock becomes partly oil-wet and the oil forms continuous films which may be drained to saturations as low as 5%. Bacterial growth can also cause partial blocking of the pore throats, and such processes will be most efficient in zones with best access to nutrients; i.e. in the high permeable reservoir zones. When the permeabilities in these zones are reduced by bacterial growth, the water will be diverted to other zones. This process will enhance the sweep efficiency in the reservoir. Diversion of the flow path may also increase the velocity of the injection water, and this, in combination with a reduction in IFT, may mobilize some of the residual oil. The residual oil saturation is related to the capillary number through capillary desaturation curves (CDC), as shown in Figure 1. The capillary number is defined as the ratio between viscous and capillary forces as in Equation 1 This example of CDCs [11] shows that when the velocity increases, or the IFT is reduced, more oil may be mobilized.

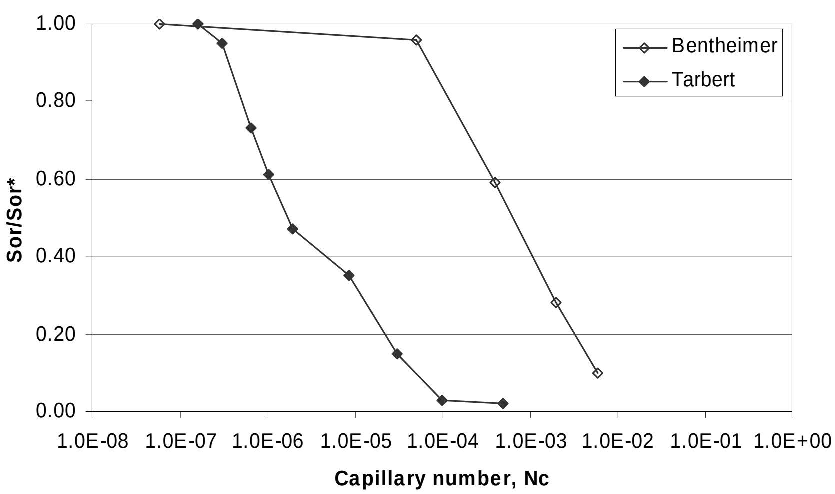

Figure 1. Example of capillary desaturation curves (from ref. 11).

$$N_c = \frac{v\mu}{\sigma} \qquad \text{[Eq. 1]}$$

## ANALYZING MIOR MECHANISMS

In addition to production and flow parameters from core flooding experiments and field tests, it is important to also include measurements of other basic parameters, such as IFT and wetting properties, in order to fully understand the mechanisms involved in MIOR. A growing bacterial system is more challenging than a chemical system, where equilibrium may be established within a few hours. The growth of bacteria is exponential and several days may be required before a representative measurement can be made. Traditional special core analysis (SCAL) methods are not designed for this, and may therefore fail when dealing with growing bacterial systems that undergo large changes in reservoir parameters during the experiment.

### Interfacial tension

Explaining the increased oil recovery reported in some laboratory experiments [4] by reduction in IFT only, requires that the bacteria have a significant impact on the water/oil interface properties. This imposes special requirements for the experimental methods to be used. Several techniques exist for measuring IFT, but few of them meet all the requirements needed for MIOR systems. The method must be able to perform continuous measurements for several weeks without making any changes to the fluid system, and the measurement range must be at least four orders of magnitude. A method such as pendant drop has been tested, but this failed to measure for a sufficiently long time [12]. Spinning drop may be a better suited method, but the volumes in such a system might be too small to support a bacterial system with nutrients for a sufficiently growth period. However, values of IFT down to 0.07 mN/m has been reported when using this method [3].

Laser light scattering is a method that is well suited for studying growing bacterial systems, in that the method itself does not affect the results when IFT is measured over a long period of time. Measurements in a growing aerobic bacterial system, showing a reduction in IFT from 35 to 0.17 mN/m over a period of approximately two weeks have been reported when using this technique [13]. There were, however, reasons to believe that these results were limited by the experimental setup, and new measurements were performed [9]. Oxygen and nutrients were continuously supplied to the bacterial system and the resulting reduction in IFT was measured from 38 to $6 \cdot 10^{-3}$ mN/m, as shown in Figure 2. This is a reduction of nearly 4 orders of magnitude, and if these effects are directly compared to a capillary desaturation curve for a strongly water-wet rock [11], it has the potential of reducing the residual oil saturation to almost zero. These measurements were performed with a single strain bacterial culture, and may not be directly comparable to a reservoir case. It still proves that IFT reduction is an important mechanism in MIOR. How this effect will expand into a reservoir from an injector is still a question to be answered since an aerobic process may probably be limited to an area not far from the injector.

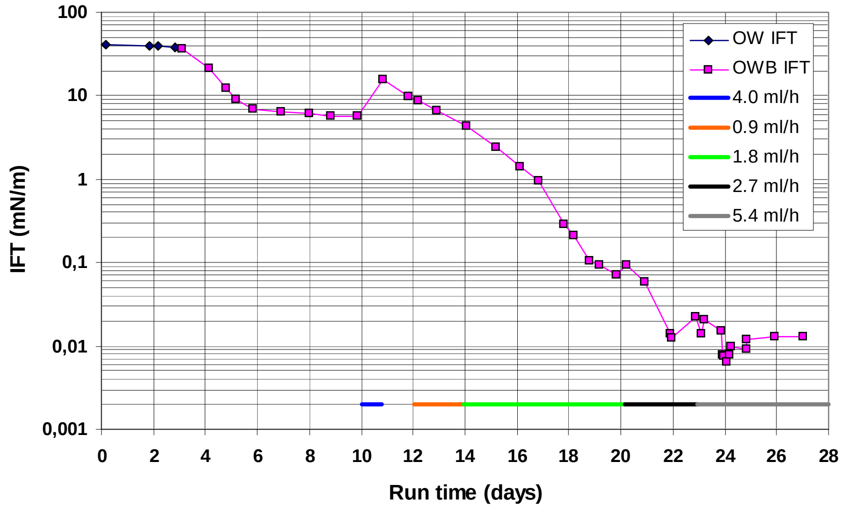


Figure 2. IFT reduction in a growing aerobic bacteria system (after [9])

## Wettability

Wettability measurement in a bacterial system is also a challenging task, since the traditional methods such as the Amott-Harvey technique [14] are difficult to perform in an active bacterial system. It is not known whether the effect on the wetting properties remains when the bacteria stop growing, and it is therefore necessary to either investigate under growing conditions or fix the liquid distribution so that no further changes occur. The Amott-Harvey index is frequently determined by a series of centrifuging or flooding processes and spontaneous imbibition of liquids. The spontaneous imbibition parts of the investigations may last for a considerable number of days, and with only minor changes in saturation. This means that the bacteria only get oxygen and nutrients through diffusion, which is a rather slow process, and it is therefore not likely that they can maintain the growth. The same will be the case if a centrifuge is used to reach the endpoint saturations, while a flooding process would continuously provide oxygen and nutrients to the bacteria. For these reasons, the effect on wettability may be reduced or not even be observed with traditional SCAL methods. Several investigations have analyzed the wetting properties of the rock material in MIOR projects with various results. One study [15] concluded that there was a significant difference in the spontaneous imbibition curves before and after bacterial stimulation in originally strongly water-wet Bentheimer sandstone cores. The results indicated that the system changed towards less water-wet conditions. However, there were no clear signs on the Amott-Harvey indices that the wetting properties had changed. It may therefore be difficult to identify which of the two mechanisms, IFT reduction or wettability alteration, caused this behaviour. In another study it was observed that the in-situ growth of bacteria changed the wetting properties in a sand pack [16]. Some bacteria were observed to give more water-wet properties while other bacteria had no impact on the wettability.

Poulson et al. [17] used an environmental scanning electron microscope (ESEM) to investigate the shape of water drops on a quartz surface both with and without a biofilm present. The results showed that without biological growth, water showed a low contact angle on the quartz grain surfaces. However, when allowing for two days of bacterial growth on the quartz, the surface changed to less water-wet with spherical water drops (high contact angle).

Fixing the liquid distribution in the pores while the bacteria are in a growing state, is a possible solution to determine the wetting properties. This may be directly visualized by studying the fluid distribution in the pores by using a Cryo-ESEM technique. However, this method is more uncertain for achieving an overall quantitative picture of the wettability situation since the distribution can only be visualized in 2D and the selected area may not be representative to the whole sample. Nevertheless, qualitative comparisons between different parts of the rock sample can give valuable information on the conditions inside the sample during the bacterial growth and oil mobilization.

### Selective blocking

When the water permeability in parts of a formation is reduced, the injection water might be diverted to areas with lower permeability. This may be observed as small increases in injection pressure during laboratory experiments [9]. Such investigations have confirmed that reduced permeability is an effect in core floods and that the magnitude of the permeability reduction is dependent on the bacteria involved [18]. Other publications have demonstrated that the selective blocking may be dependent on the core material, and that native cores are more likely to experience a significant permeability reduction than outcrop cores [19].

## EXPERIMENTAL

The wetting properties of an aerobic MIOR core were investigated by visualizing the fluid distribution in single pores using a Cryo-ESEM technique. This technique has been previously described [20], and has been further developed for analysis of larger sample sizes. In addition, the oil saturation in some of the electron images has been calculated to investigate the potential of saturation determination by image analysis of Cryo-ESEM micrographs.

### Core flood

The core sample parameters are shown in Table 1 together with the average oil saturations before and after the MIOR process. The sample was a highly permeable outcrop sandstone (Bentheimer), which was first saturated with water and then flooded to irreducible water saturation before the water flooding was initiated. The fluids used were North Sea crude oil and synthetic seawater with additional nitrate and phosphate. After reaching residual oil saturation, the core was inoculated with bacteria and the aerobic water flooding was continued. During the following flooding the residual oil saturation was reduced by 9% points due to the bacterial activity, making it a good candidate for investigating wettability as an MIOR mechanism. A complete change towards mixed wet conditions with drainage of continuous oil films is likely to result in a lower residual oil saturation than observed in this core, so a significant change in wettability was not expected. Still it would be of value to investigate if the improved technique could visualize the fluid distribution and thereby indicate any changes in the wetting conditions.

Table 1. Core properties and flooding results

| Parameter | Value |
|---|---|
| Length [cm] | 30 |
| Diameter [cm] | 5.1 |
| Porosity [fraction] | 0.23 |
| Permeability [mD] | 2400 |
| Sor [fraction] before MIOR | 0.44 |
| Sor [fraction] after MIOR | 0.34 |
| Flow [cm/day] | 30 |

**Cryo-ESEM procedures**

The technique consists of rapidly freezing slices (of a few centimetres) from selected parts of the core used in the flooding experiment. The freezing process is performed in liquid nitrogen and quickly immobilizes the fluids in the pores. A small piece of the slice (approximately 1x1x1 cm) is mounted in a cryo preparation chamber connected to the microscope. The sample is then fractured under vacuum conditions to prevent ice crystal growth on the freshly exposed surface, and then transferred into the ESEM cold stage, held at a temperature around -100 ºC. The fracturing technique has been improved by changing the construction of the tools. The identification of the fluids and minerals are performed by energy dispersive X-ray analysis (EDS) in the ESEM. Characteristic X-rays generated by the electron beam in the ESEM are used for both point analysis and elemental distribution X-ray images. By combining elemental distribution images from selected elements into a composite elemental image, the liquid distribution in the pores can be visualized. Carbon (C) will represent the oil phase and oxygen (O), together with sodium (Na) and potassium (K), will represent the brine. Silicon (Si) and aluminium (Al) are used to separate grains and clay from the fluids (Figure 3).

The equipment used is a FEI Quanta 400 ESEM with a Thermonoran Vantage EDS system. The cryo system is a modified version of Oxford instruments cryo transfer system CT1500. The modification involves a more robust fracturing device and sample holders designed for larger samples. In this study the microscope was operated at a lower pressure than in previous studies [20], which allows detector arrangements with the ability to examine larger areas. Typical microscope operation conditions are given in Table 2.

Table 2. Typical microscope parameters

| Parameter | Value |
|---|---|
| Sample chamber atmosphere | Nitrogen |
| Accelerating voltage | 15 kV |
| Sample chamber pressure | 0.3 Torr (0.4 mbar) |
| Sample holder temperature | -100 ºC |

Three areas were selected for further analyses. One very close to the inlet side of the core during the flooding process, one close to the outlet of the core, and the last area was in the middle of the core (approximately 15 cm from the outlet end).

**Saturation calculations**

In a scanning electron microscope the images are formed by detecting the electrons generated or reflected by the interaction of the primary beam and the sample surface. There are basically two types of electrons generated, secondary electrons (SE) and backscattered electrons (BSE).

SE are low energy electrons escaping from the upper nanometres of the sample surface and they visualize the topography of a sample. BSE are higher energy electrons of the primary electron beam reflected from the sample. A component with high mean atomic number will reflect more electrons than a component with low mean atomic, creating a brighter part in the image. The BSE electrons signal in the Cryo-ESEM enable good contrast between the oil, brine and mineral grains. Unlike other imaging techniques, like i.e. Computer Tomography (CT), there is no need for adding tracers or dopants to the fluids for enhanced contrast. This is an important factor in this study to avoid any unknown effects caused by the tracers or doping material on the bacteria. Mineral grains will appear as the bright areas since they have a dense atomic structure, and the oil will show up as the darkest areas. The brine has brightness in between minerals and oil. The brightness of the liquids is in some degree dependent on the composition of the salinity of the brine and trace elements in the oil.

Image analysis software has been used to separate the mineral and fluid phases by the grey level differences in the BSE image. A pixel counting routine is then used to determine the area of the total image covered by oil and water. The ratio of oil pixels to the sum of oil and water pixels will represent the oil saturation. Each individual image covers a few millimetres of core material. Several images must be analysed to achieve a representative result. The number of images required will depend on the heterogeneity of the core, in terms of such properties as pore sizes and connectivity.

## RESULTS AND DISCUSSION

The liquid distribution close to the inlet side of the core is shown in Figure 3, and a further magnified part of this area is shown in Figure 4. Carbon is coloured red, indicating the oil phase. Oxygen is coloured blue, which may be an indicator of quartz, clay minerals or water. However, the relative amount of oxygen is higher in water so the areas containing water will have a significantly higher intensity than the rock minerals. Blue is therefore used as an indicator of water. It would be preferable to use one of the elements specific to seawater, but the concentrations of these are relatively small and consequently the signals are too weak. Silicon is coloured green, indicating rock minerals, and aluminium is coloured pink to show the kaolonite and feldspar in the sample. An area closer to the centre of the core (approximately 15 cm from the inlet) is shown in Figure 5, and two areas close to the outlet are shown in Figures 6 and 7. As can be seen from all the figures the liquid distribution indicates that most of the mineral grains have remained water-wet after the MIOR process. However, in some of the images there are a few quartz grains that appear to be partially oil-wet (marked by yellow circles), indicating that the core is not completely water-wet. This may be caused by the bacterial lowering of the IFT, resulting in the rupture of the wetting water films. This will lead to the oil contacting the mineral grain and possibly making this part of the grain oil-wet. The overall impression is still that the core is water-wet. However, water films with thickness in the range of a few molecular layers would not be observed.

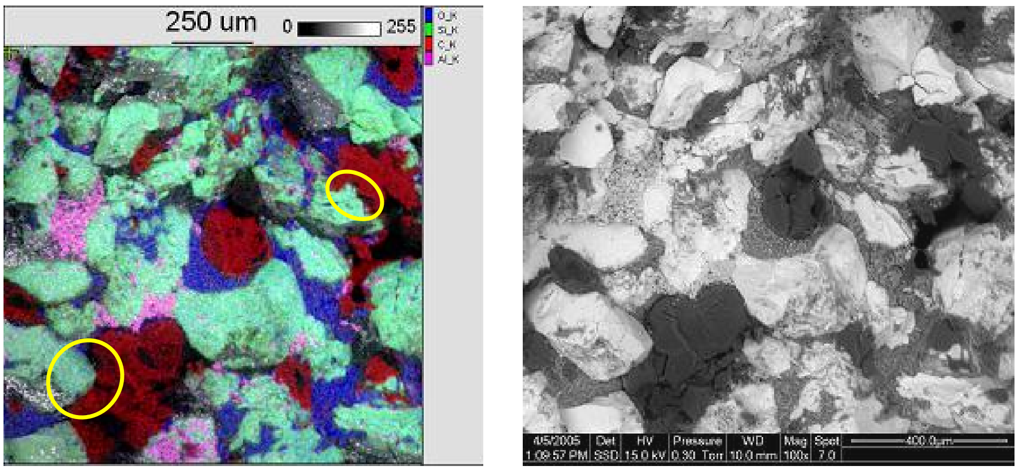


Figure 3. Element distribution (a) and electron image (b) close to the inlet of the core. Blue represents water, red represents oil, green represents quartz and pink represents kaolinite

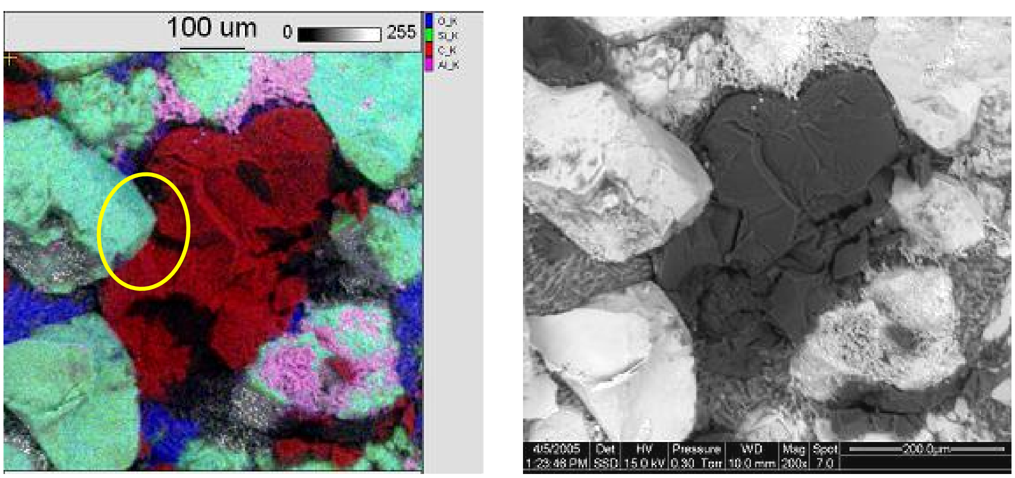


Figure 4. Element distribution (a) and electron image (b) close to the inlet of the core.

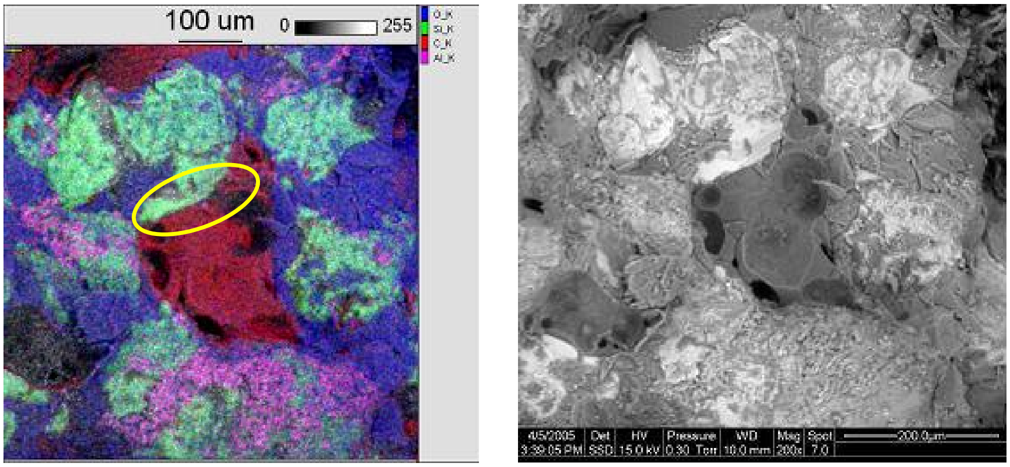

Figure 5. Element distribution (a) and electron image (b) close to the centre of the core.

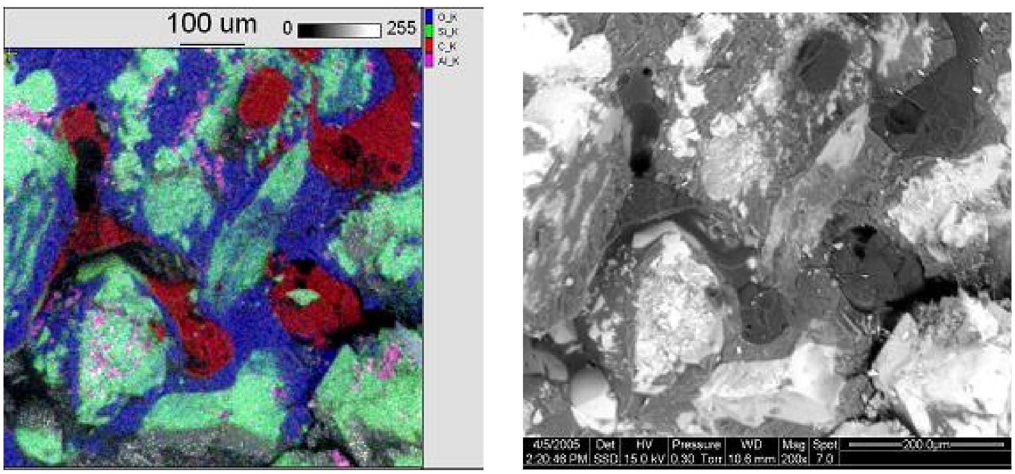

Figure 6. Element distribution (a) and electron image (b) close outlet of the core.

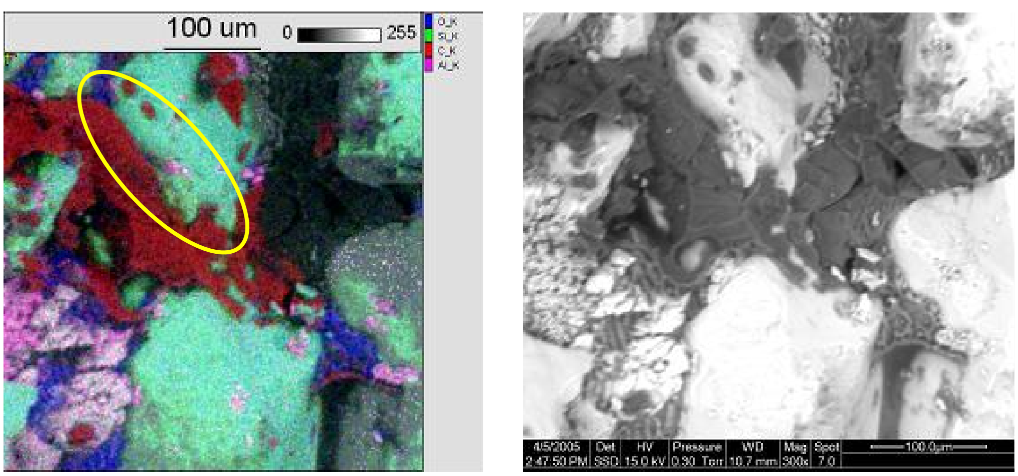

Figure 7. Element distribution (a) and electron image (b) close to the outlet of the core.

For the saturation calculations a few of the low magnification images were chosen, of which one is presented in Figure 8a. The oil and water images, extracted from the grey level image in Figure 8a, are shown in Figures 8b and 8c respectively. The results from the saturation calculations are shown in Table 1, and indicate a minor variation in saturation through the core. The centre of the core gives lower oil saturation than both inlet and outlet. The average saturation calculated from the images (36%) is close to the actual average saturation from the core flood measurements (35%). This might indicate that the limited number of images analysed are representative for the core. More analyses of each cross section of the core and more cross sections would have given a more reliable evaluation of the saturation profile through the core.

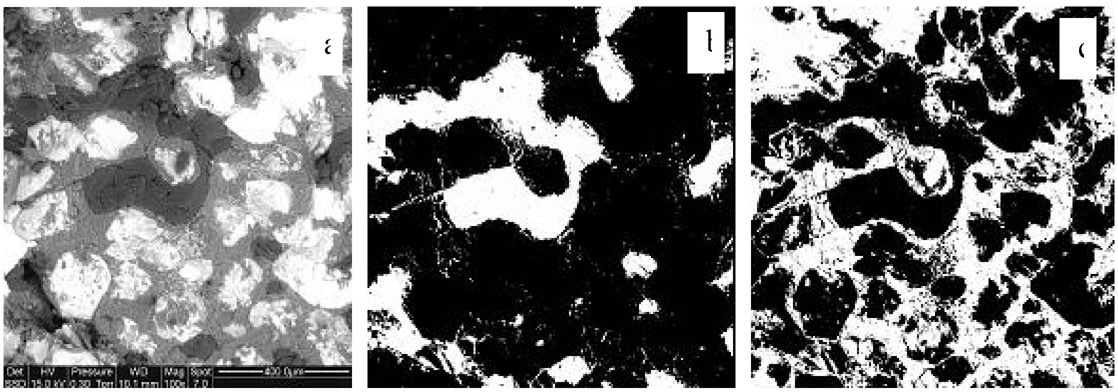

Figure 8. Electron image (a) with the extracted oil phase (b) and water phase (c)

Table 1. Oil saturation from image analyses

| Position | Area | Oil saturation [%] | Average saturation [%] |
|---|---|---|---|
| | 1 | 35 | |
| Inlet | 2 | 39 | 39 |
| | 3 | 42 | |
| Centre | 4 | 29 | 29 |
| | 5 | 30 | |
| Outlet | 6 | 36 | 40 |
| | 7 | 41 | |

## CONCLUSIONS

- No significant changes in wettability of the MIOR treated core are found. The majority of the micrographs show pore systems that appear to be water-wet, but some few pore walls are clearly oil-wet. This leads to the conclusion that the core is still mainly water-wet but with some mixed wet parts.
- It has been demonstrated that Cryo-ESEM can be used for detailed studies of saturation and wettability properties from sub-micron to centimetre scale. Sample size and analyzed areas are increased compared to previous Cryo-ESEM studies. No dopant is needed for contrast enhancement between the fluids.

- A new technique for estimating oil and water saturations from electron micrographs is demonstrated and appears to be promising for studying wettability changes on the pore scale. A large number of images are necessary to ensure that the results are representative for the whole core. Good agreement was found between the average saturation from the micrographs and the average saturation calculated from the core flooding experiment.
- The two techniques for wettability analyses and saturation calculations are promising methods for pore level investigations. More work is necessary to verify the reliability of the methods, and if changed wettability is an important MIOR mechanism.

## NOMENCLATURE

$N_c$ Capillary number, dimensionless
v Velocity, m/s
μ Viscosity, Pa s
σ Interfacial tension, N/m
Sor Residual oil saturation at increased capillary number, fraction
Sor* Residual oil saturation, fraction

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