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Filtration of Emulsions

Part 1

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REPORT OF ANALYSIS

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1 SUMMARY

Low field NMR has been used to characterize emulsion droplets before and after filtering through glass filters with different pore size. It was demonstrated that stable emulsions with droplet size up to 24 μm seemed to pass the filter (with pores down to the range 10-16 μm) without coalescing. Emulsions prepared at low mixing speed were either unstable or coalescence occurred during or after filtration.

The filtrating process seemed to change the emulsion state under certain conditions. The clue to better understanding would obviously be linked to the rheological properties of the w/o interface, and especially so the elasticity of the interface.

2 INTRODUCTION

This research initiative is a scientific collaboration between Ugelstad Laboratory and IRIS in the field of the role of emulsions in the reservoirs and in the enhanced recovery.

In this first approach we have studied the transportability of emulsions with different sizes through porous solid materials with different pore size. The study involved droplet characterization before and after filtration through the pores, and brine profile after filtration. The characterization was done with low field NMR, and in addition some samples were analyzed by microscope and electrocoalescence (critical electric field emulsion stability cell).

3 SAMPLES

Crude oil

Primol

Span 80

3.5 % NaCl in water (brine)



4 EXPERIMENTAL

4.1 Experimental procedure

Emulsions with 10, 15 and 30 % water cut (WC) were prepared by mixing oil and brine, always adding to a volume of 30 mL. Brine and oil were mixed with an Ultra Turrax (IKA, T18 with a 10 mm head) with speeds up to 24 000 rpm to create small droplets and a 4 bladed propeller at lower speeds, down to 500 rpm, to create larger droplets. Both crude oil and model oil were tested. Emulsions based on model oil (Primol) were added 0.5 % Span 80 as surfactant before preparing the emulsion.

Emulsions were filtered through glass filters with porosity 2, 3 and 4. Water jet pump and peristaltic pump were used to pump the sample through the filter.

Glass filters used:

- Porosity 4: 10-16 μm
- Porosity 3: 16-40 μm
- Porosity 2: 40-100 μm

The droplet size distribution (DSD) before and after filtering the emulsion was determined by low field NMR.

4.2 NMR

4.2.1 NMR – brine profile

Figure 4.2.1 shows the NMR sequence used for measuring the water profile. Initially, the NMR signal contains a crude oil and water contribution. Because of a significantly lower transverse relaxation time for the crude oil, one may use the C_1 loop to suppress the contribution from the crude oil. The first gradient echo is then from water only, and a Fourier transform of this echo yields a water profile. The second loop is used to measure a gradient echo at an even longer observation time, which gives another water profile. These two profiles may then be used to correct for transverse relaxation of the water signal, ending with a third water profile, which is unaffected by transverse relaxation processes. This profile is proportional to the water content along the sample, and by calibrating with a sample that contains 100% water; one can measure the water content along the height of the emulsion. C_1 is determined by measuring the signal for pure oil phase and determining at which C_1 value the entire oil signal has vanished.

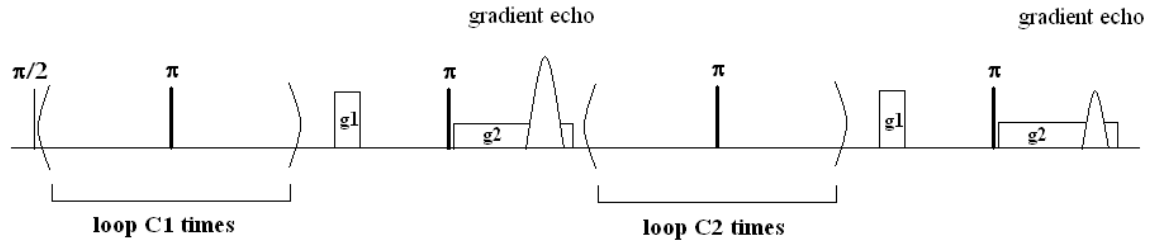


Figure 4.2.1: NMR sequence used for acquiring the water profile.

The NMR measurements were performed on a low field NMR spectrometer supplied by Anvendt Teknologi AS. The magnetic field is 0.48 T, capable of delivering a gradient strength up to 4 T/m. The duration of the sinusoidal gradient pulse is 0.8 ms, and the direction of the gradient is along the length of the tube. The NMR sample tubes of 18 mm diameter were filled with ~4 mL of the emulsion.

Details about the NMR procedure are given in Opedal et al. "Emulsion Stability Studied by Nuclear Magnetic Resonance (NMR)", Energy & Fuels, 2010, 24, 3628-3633 and in Simon et al. "Separation Profile of Model Water-in-Oil Emulsions followed by Nuclear Magnetic Resonance (NMR) measurements: Application Range and Comparison with a Multiple-light Scattering Based Apparatus", Journal of Colloid and Interface Science 356, 2011, 352-361.

4.2.2 Droplet size distributions (DSD) by NMR

There is a situation where the surface relaxation term is absent in the solution of the diffusion propagator, i.e. the short time expansion. By assuming piecewise smooth and flat surfaces and that only a small fraction of the particles are sensing the restricting geometries, the restricted diffusion coefficient can be written as

$$\frac{D(t)}{D_0} \approx 1 - \frac{4}{9\sqrt{\pi}} \sqrt{D_0 t} \frac{S}{V} + \phi(\rho, R, t) \quad [1]$$

where $D(t)$ is the time dependent diffusion coefficient, D_0 is the unrestricted diffusion coefficient, in bulk fluid, and t is the observation time. The higher order terms in t , $\phi(\rho, R, t)$, hold the deviation due to finite surface relaxivity and curvature (R) of the surfaces. At the shortest observation times these terms may be neglected such that the deviation from bulk diffusion depends on the surface to volume ratio alone. In a porous system a large span in pore sizes must be assumed, and thus eq. 1 must be expected to be valid also for a heterogeneous system. If ξ_i is the volume fraction of the droplets with surface to volume ratio $(S/V)_i$, eq. 1 can be expressed as

$$\sum_i \xi_i \frac{D_i}{D_0} \approx \sum_i \xi_i \left[1 - \frac{4}{9\sqrt{\pi}} \sqrt{D_0 t} \left(\frac{S}{V} \right)_i \right] = \left(1 - \frac{4}{9\sqrt{\pi}} \sqrt{D_0 t} \left(\overline{\frac{S}{V}} \right) \right) \quad [2]$$



Measurements of the early departure from bulk diffusion combined with a linear fit of the experimental data to the square root of time will thus result in a value for the average surface to volume ratio $\overline{(S/V)}$.

Transforming a T_2 distribution to a droplet size distribution

Assuming that the water molecules are probing the droplets within the sample, there is a simple relation between T_2 values and the droplet sizes

$$T_2 \approx \frac{V}{S\rho} \quad [3]$$

This couples the surface to volume ratio to the surface relaxivity, ρ , and makes it difficult to assign the T_2 distribution to a (V/S) distribution. However, if we make the assumption that eq. 3 holds for any droplet size, with ξ_i being the volume fraction of droplets with surface to volume ratio $(S/V)_i$ and corresponding relaxation time T_{2i} , we may write

$$\sum_{i=1}^n \xi_i \frac{1}{T_{2i}} = \sum_{i=1}^n \xi_i \rho_i \left(\frac{S}{V}\right)_i \approx \rho \sum_{i=1}^n \xi_i \left(\frac{S}{V}\right)_i = \rho \overline{\left(\frac{S}{V}\right)} \quad [4]$$

Here we have made the basic assumption that the surface relaxivity ρ is independent of droplet size.

The left hand side of eq. 4 is the harmonic mean $\overline{1/T_2}$ of the T_2 -distribution weighted by the fraction ξ_i of nuclei with relaxation time T_{2i} and n is the number of subdivisions of droplet sizes. This average can be calculated from the T_2 -distribution obtained in a CPMG measurement where the magnetization attenuation $M^{obs}(t)$ is converted to a T_2 distribution by solving an inverse problem using e.g. an Inverse Laplace Transform (ILT) routine. Then the surface relaxivity ρ can be calculated from eq. 4 if the average surface to volume ratio $\overline{(S/V)}$ is already found from the diffusion experiment. Finally, the measured T_2 -distribution can be transformed into an absolute droplet size distribution (V/S) by means of the relationship inherent in eq. 4.

To sum up, the procedure for deriving absolute droplet size distributions is as follows:

- 1) The average surface to volume ratio $\overline{(S/V)}$ is found from fitting eq. 2 to a set of diffusion measurements at short observation times.
- 2) The average (S/V) can be correlated to the average $(1/T_2)$ found from a CPMG experiment. From eq. 3 eq. 4 can then be written as

$$\left(\overline{\frac{1}{T_2}}\right) \approx \rho \left(\overline{\frac{S}{V}}\right) \quad \Rightarrow \quad \rho = \left(\overline{\frac{1}{T_2}}\right) \times \left(\overline{\frac{S}{V}}\right)^{-1} \quad [5]$$

Hence we find the relaxivity, ρ which then is assumed to be droplet size independent.

- 3) Under the assumption of droplet size independency of the relaxivity the value of ρ can then be used in eq 3 thus resulting in a linear relation between T_2 and the volume to surface ratio which is a measure of the droplet size. By multiplying the T_2 distribution by the calculated surface relaxivity the distribution is normalized to a droplet size distribution in absolute length units.



4.2.3 Separation of high viscosity oil and water signal

There are several ways to separate the NMR contribution of the oil and water components. The most straightforward way is when the viscosity of the oil is much higher than that of the water phase. Then the longitudinal and/or the transverse relaxation times will be significantly different, and one may store the NMR signal for full recovery of the crude oil signal back to thermal equilibrium while the water signal still can be measured on. In figure 4.2.2 we have displayed the T_2 distributions of water in oil emulsion system for short and long z -storage (Δ) intervals. By increasing the duration of the z -storage one can thus omit the oil signal. The two peaks at short Δ correspond to the oil signal (left peak) and water signal (right peak).

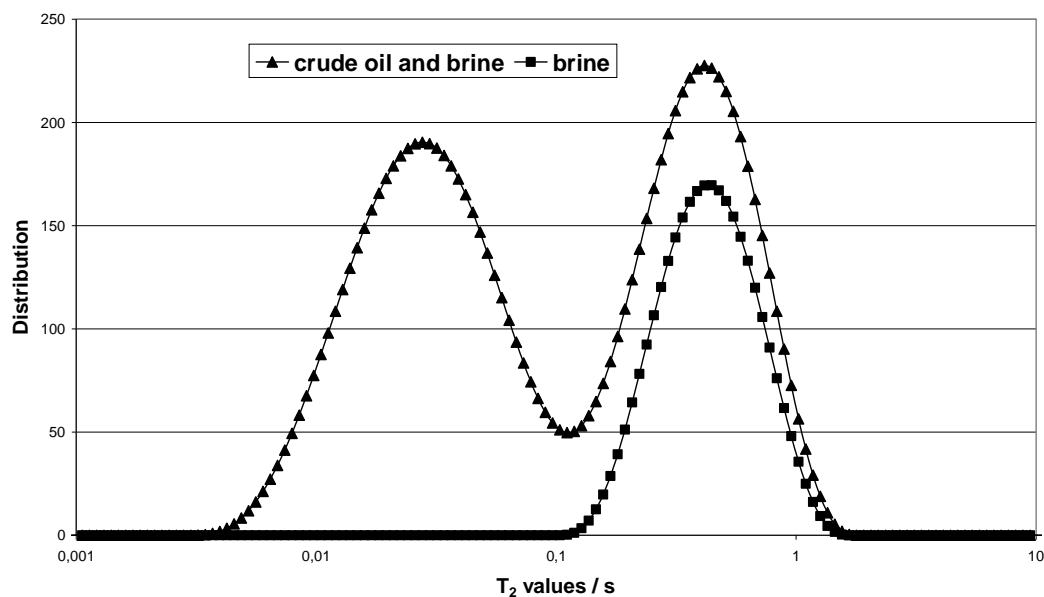


Figure 4.2.2: The effect of using z -storage delay Δ to obtain the T_2 distribution of water alone.

As the crude oil is a relatively high viscous liquid it turns out that this is the best way of resolving the water signal from the oil signal. Further details and references may be found in Sørland et.al [http://www.uni-leipzig.de/diffusion/pdf/volume5/diff_fund_5\(2007\)4.pdf](http://www.uni-leipzig.de/diffusion/pdf/volume5/diff_fund_5(2007)4.pdf)

Dynamic Pulsed-Field-Gradient NMR, <http://link.springer.com/book/10.1007%2F978-3-662-44500-6>

4.2.4 Presentation of NMR Data

The droplet sizes are presented as droplet size distribution (DSD) and mode in this report. To describe the mode value (μm): This is the most common value of highest point of the frequency curve. In a normal or Gaussian distribution the mean, median and mode will lay in exactly the same positions (fig. 4.2.4.1) and the average DSD and mode will be the same. (The figures are from *Basic principles of*



particle size analysis, Dr. Alan Rawle.) If there are created more big droplets the mode will shift towards a higher value. In many cases the mode and average droplet size would be the same.

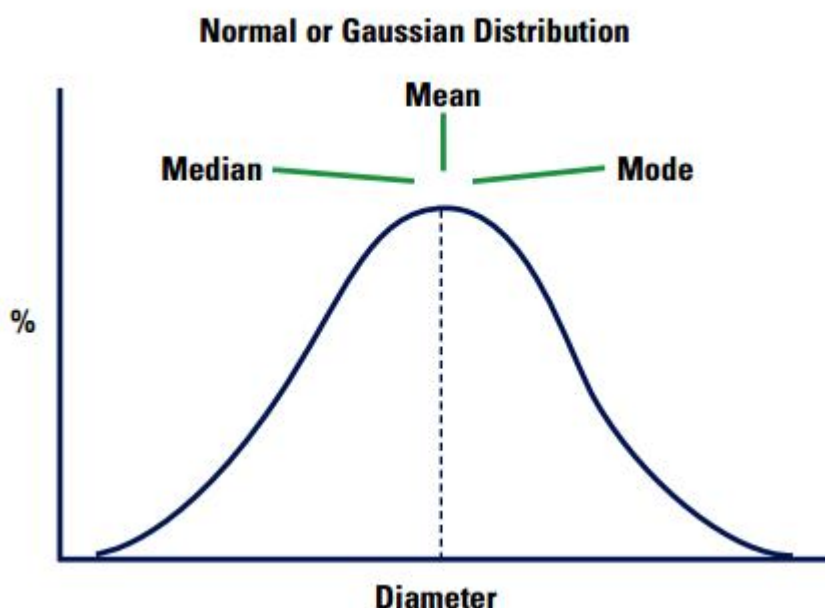


Figure 4.2.4.1: Frequency curve for normal or Gaussian distribution.

However, the distribution is bimodal as shown 4.2.4.2 the mean, median and mode will not be in the same position. The mode will lie at the top of the higher curve because this is the most common value of the diameter.

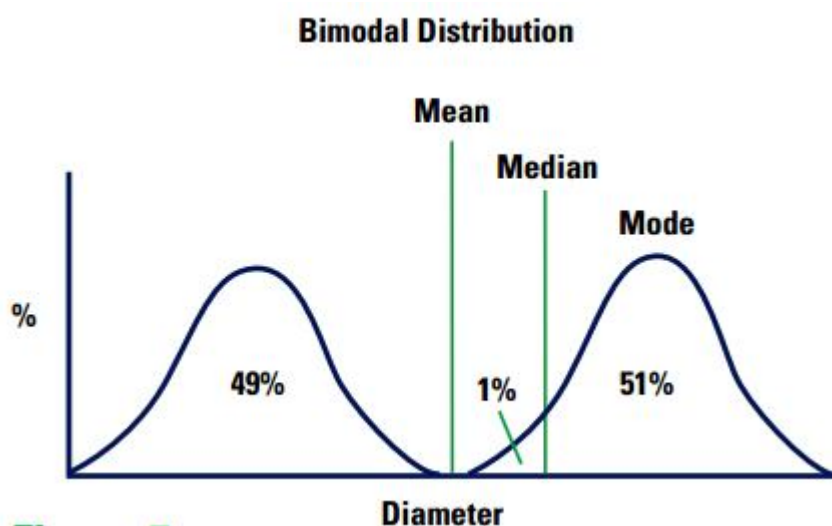


Figure 4.2.4.2: Bimodal distribution curve.

4.3 Determination of Emulsion Stability: Electrocoalescence

The emulsions were analyzed by e-critical measurements directly after emulsion preparation. A drop of the homogeneous sample was applied to the e-critical cell.



4.3.1 Principle

From the knowledge about electrocoalescers that are being commonly used in the oil industry to enhance water separation from crude oil, an electric field cell for determination of emulsion stability has been developed (Aske, Kallevik et al. 2002 [1]). When no electric field is applied to an emulsion, the water droplets are randomly distributed according to Stokes law of sedimentation and Brownian motion. Applying an electric field to the emulsion will polarize the water droplets, and they start to line up between the electrodes in the cell. At some point a water-continuous bridge will form between the electrodes due to droplets coalescence and the ions in the water phase will lead to an increase in conductivity. The point where the conductivity raises is hence a measurement of the emulsion stability. Figure 4.3.1 shows how a w/o emulsion behaves when an electric field is applied to the emulsion.

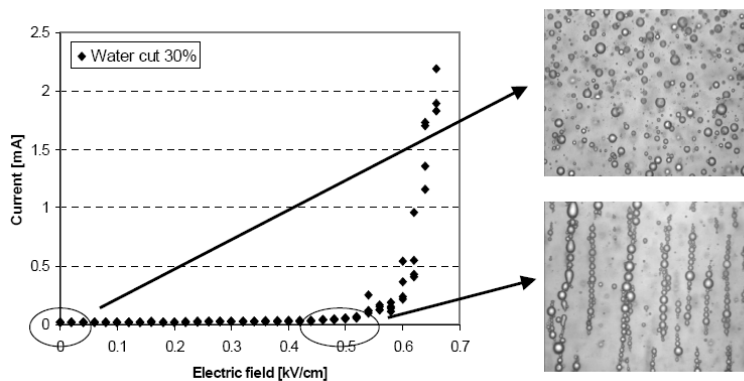


Figure 4.3.1: Behavior of w/o emulsion in an electric field (Aske, Kallevik et al. 2002).

The electric field cell consists of two brass plate electrodes separated by a thin Teflon plate. The thickness of the Teflon plate can be varied and it has a 10 mm diameter sample hole in the center (Figure 4.3.2).

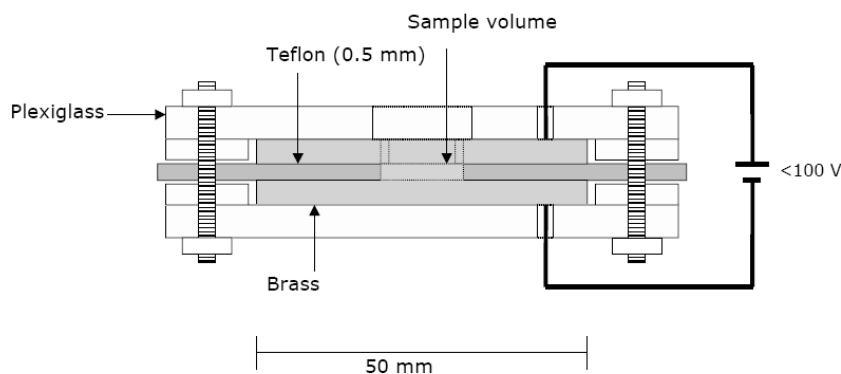


Figure 4.3.2: The critical electric field cell.

The brass plate electrodes are connected to a power supply (Agilent 6634B) that can increase voltage stepwise up to 100 V DC and measure current through the emulsion. A sudden increase in current running through the sample will indicate that the electric field has broken the emulsion. The corresponding electric field is the e-critical value.



The e-critical measurements depend on several experimental parameters, such as water content, applied electric field gradient and temperature. It is therefore important that e-critical measurements are conducted at the same conditions when comparing different emulsion systems.

4.4 Optical Microscopy

The crude oil B and NIMR were visually investigated by microscopy. Nikon Eclipse ME 600 digital video microscope together with Image Pro Plus 5.0 software was used to acquire pictures of the emulsions water droplets. The lens used was the CFI LU Plan Epi 10X with an N.A. (numerical aperture) of 0.30. With the wavelength of the light source being 0.55 μm , the resolving power of this lens was 0.91 μm . A CoolSNAP-Pro cfw 4 megapixel cooled CCD camera (monochrome) from Media Cybernetics® was used to digitalize the images. (This camera incorporates a CCD (charge-coupled device), a 12-bit scientific digitizer and low-noise electronics to produce 36-bit digital monochrome images at greater than 100 \times 1000 resolution.)

5 RESULTS AND DISCUSSION

5.1 Oil Characteristics

The crude oil used in this project was a heavy crude oil with density 0.9224 g/cm^3 and viscosity 85 mPa s, and the model oil was primol with density 0.8521 g/cm^3 and 61 mPa s at 40°C.

5.2 Filter 2, 40 - 100 μm pores

5.2.1 Crude Oil

The filtering of crude oil emulsions (w/o, 30 % WC) at 40°C with water jet pump through filter with pore size 40 – 100 μm did not seem to affect the emulsions (tab. 5.2.1), neither for the emulsions prepared with droplet size 1 - 22 μm nor the emulsion prepared with droplet size in the range 1 – 36 μm . The mode was 6 before and after filtering for the emulsions prepared with the smallest droplets (24 000 rpm in 2 min), and the droplet sizes did not change after filtration.

To create bigger droplets the emulsion was prepared by stirring at 5000 rpm for 2 min, which also gave less stable emulsion. The mode moved from 6 μm to 13 μm after filtration, and the droplet size distribution moved from 1 - 22 μm to 2 - 81 μm . A layer of concentrated emulsions with big droplets at the bottom of the NMR tube were detected (see attachments). This could be explained by less stable emulsion due to aging, but could also be an indication that the droplets coalesced when they were filtered, especially so for emulsions with larger initial droplets.



Table 5.2.1: NMR results before and after filtering w/o emulsions through filter 2.

Name of sample	Pump	Pore size	Mixing parameters	DSD before filtering [μm]	DSD after filtering [μm]	Mode before filtering [μm]	Mode after filtering [μm]	Brine profile
Crude oil emulsion 30wc, p1	Water jet pump 2 min filtration	Glass filter 40-100 μm	24000 rpm 2 min	1-22	1-23	6	6	No free water after filtration
Crude oil emulsion 30wc, p2	Water jet pump 10 s filtration	Glass filter 40-100 μm	24000 rpm 2 min	1-22	1-23	6	6	No free water after filtration
Crude oil emulsion 30wc, p1	Water jet pump 20 s filtration	Glass filter 40-100 μm	5000 rpm 2 min	1-36	2-81	6	13	Concentrated layer of big droplets after filtration

5.3 Filter 3, 16 – 40 μm pores

5.3.1 Crude Oil

Water in crude oil emulsions were prepared by mixing at 24 000 rpm for 1 and 2 min at 40°C (30 % WC). The samples were filtered through a glass filter with pore size 16 - 40 μm (tab. 5.3.1). The samples had droplet sizes up to 24 μm before filtration. There was a difference in filtration time on these two parallels, most likely because the water jet pump is not very precise. The first parallel was filtered for 5 min and free water was detected after filtering. For the second parallel which was filtered for 1 min there was no free water, but a layer of concentrated big droplets in the bottom of the NMR tube and the mode shifted from 7 μm to 8 μm , with droplets up to 35 μm . It seemed like the filtration induced coalescence since there was a high concentration of water in the bottom of the tubes of the two parallels.

No coalescence was detected when stirring for 2 min at 24 000 rpm, and the mode and droplet size was quite similar as when filtering with filter 2 (40 – 100 μm), which could indicate that the droplets with size up to 23 μm were filtered directly through the filter pores.

Emulsions with larger droplets resulted in a thick concentrated layer of big droplets at the bottom of the NMR tube, which made it impossible to determine the droplet size distribution after filtering.



Table 5.3.1: NMR results before and after filtering w/o emulsions through filter 3.

Name of sample	Pump	Pore size	Mixing parameters	DSD before filtering [μm]	DSD after filtering [μm]	Mode before filtering [μm]	Mode after filtering [μm]	Brine profile
Crude oil emulsion 30wc, p1	Water jet pump 5 min filtration	Glass filter 16-40 μm	24000 rpm 1min	1-22	1-25	6	6	Free water after filtration
Crude oil emulsion 30wc, p2	Water jet pump 1 min filtration	Glass filter 16-40 μm	24000 rpm 1 min	1-24	1-35	7	8	Concentrated layer of big droplets after filtration
Crude oil emulsion 30wc, p1	Water jet pump 4 min filtration	Glass filter 16-40 μm	24000 rpm 2 min	1-23	1-21	6	5	No free water after filtration
Crude oil emulsion 30wc, p1	Water jet pump 4 min filtration	Glass filter 16-40 μm	2000 rpm 5 min	2-34	NA	9	NA	Concentrated layer of big droplets after filtration

5.3.2 Primol and Span 80

Model oil emulsions were prepared by mixing primol added span 80 with 15 % water for 5 min at 2000 rpm. Droplet size before filtration was below 30 μm and mode around 9 μm (tab 5.3.2.1). After filtration through a filter with pore size 16-40 μm the mode shifted towards slightly smaller droplet size distribution and there was detected a layer of bigger droplets at the bottom of the NMR tube, which indicates some coalescence of larger droplets.

Microscopic images of the model oil emulsion showed some larger droplets after filtration through filter 3, which was the same seen in the bottom of the NMR tube (fig. 5.3.2.1 and 5.3.2.2).

Table 5.3.2.1: NMR results before and after filtering w/o emulsions through filter 2.

Name of sample	Pump	Pore size	Mixing parameters	DSD before filtering [μm]	DSD after filtering [μm]	Mode before filtering [μm]	Mode after filtering [μm]	Brine profile
Primol and span 80 15 % WC, p1	Hydraulic pump 3 min	Glass filter 16 – 40 μm	2000 rpm 2 min	1-26	1-18	8	5	Slightly larger droplet at the bottom om the tube
Primol and span 80 15 % WC, p2	Hydraulic pump 3 min	Glass filter 16 – 40 μm	2000 rpm 2 min	2-29	2-23	9	7	Slightly larger droplet at the bottom om the tube

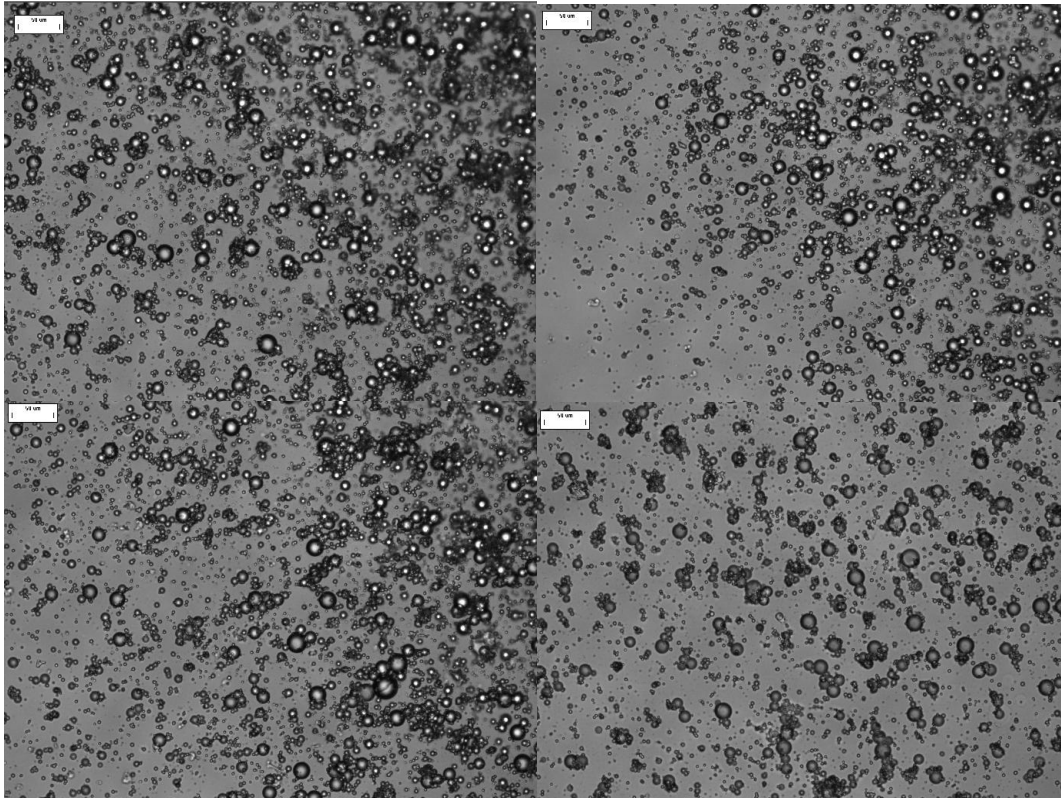


Figure 5.3.2.1: 4 microscopic images of one model oil emulsion (w/o) before filtering. 10 times magnification. The white marker in the left corner of the pictures is 50 μm .

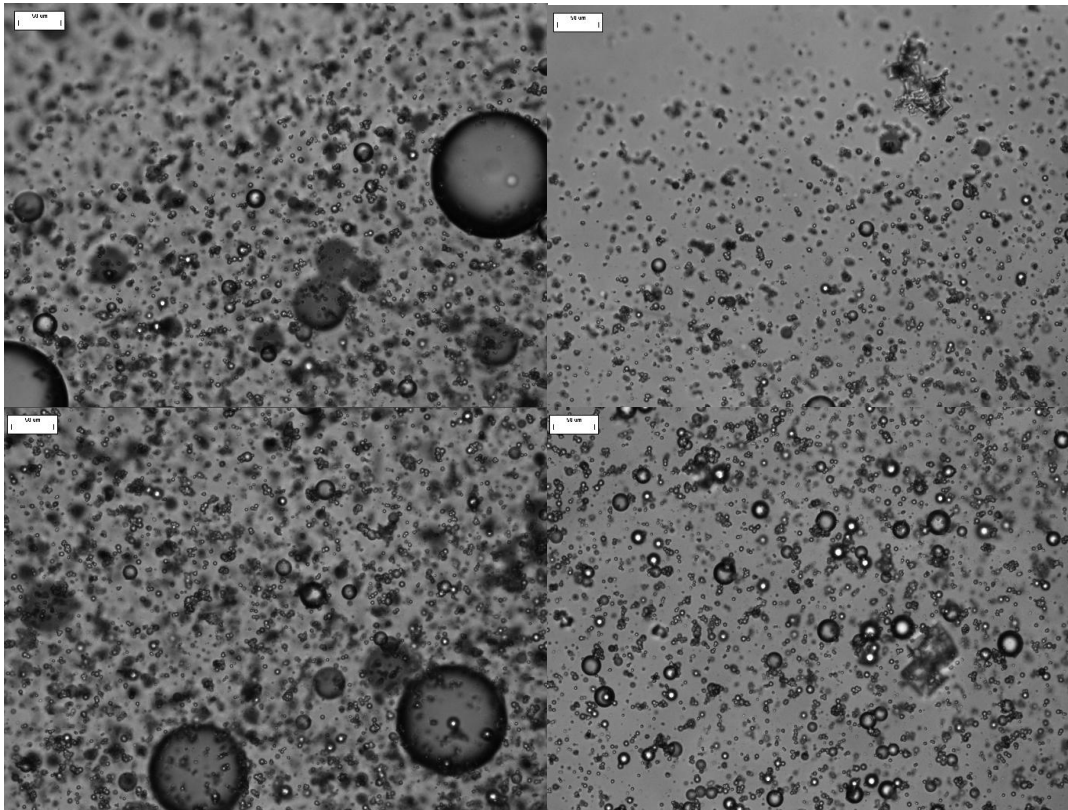


Figure 5.3.2.2: 4 microscopic images of one model oil emulsion (w/o) after filtering through filter 3 with pore size 16-40 μm . 10 times magnification. The white marker in the left corner of the pictures is 50 μm .



5.4 Filter 4, 10-16 μm pores

5.4.1 Crude Oil

Similar stable crude oil emulsions (w/o) as studied in chapter 5.2.1 and 5.3.1, stirred for 2 min at 24 000 rpm, were filtered through filter 4. It seemed like the droplets with maximum size 23 μm were filtered through pores with size 10 - 16 μm pores (tab 5.4.1.1), since the NMR results were similar before and after filtering.

Table 5.4.1.1: NMR results before and after filtering w/o emulsions through filter 4.

Name of sample	Pump	Pore size	Mixing parameters	DSD before filtering [μm]	DSD after filtering [μm]	Mode before filtering [μm]	Mode after filtering [μm]	Brine profile
Crude oil emulsion 30wc, p1	Water jet pump 4 min filtration	Glass filter 10-16 μm	24000 rpm 2 min	1-23	1-23	6	6	No free water after filtration
Crude oil emulsion 30wc, p2	4 min	Glass filter 10-16 μm	24 000 rpm 2 min	1-23	1-24	6	6	No free water after filtration
Crude oil emulsion 30wc, p1	Water jet pump 6 min filtration	Glass filter 10-16 μm	2000 rpm 5 min	2-30	3-75	9	15	Concentrated layer of big droplets after filtration
Crude oil emulsion 30wc, p2	Water jet pump 12 min filtration	Glass filter 10-16 μm	2000 rpm 5 min	2-29	3-71	9	14	No free water after filtration
Crude oil emulsion 30wc, p1	Water jet pump 5 min filtration	Glass filter 10-16 μm	500 rpm 5 min	0-35	3-137	9	21	Concentrated layer of big droplets after filtration??
Crude oil emulsion 15wc, p1	Water jet pump 4 min filtration	Glass filter 10-16 μm	2000 rpm 5 min	1-33	3-57	9	12	Concentrated layer of big droplets after filtration



After preparing emulsions with mixing speed 2000 rpm for 5 min (30 % WC) it looked like the first part passing the filter contained a large amount of water (fig 5.4.1.1). The filtration was very slow, and the sample was taken before the filtration had finished. Some of the water droplets coalesced during or just after filtration (fig 5.4.1.2) and there were observed large water droplets in the bottom of the Erlenmeyer flask (fig. 5.4.1.3). These big droplets were either not sampled or out of the detection range of the NMR instrument and were most likely left at the bottom of the flask after transferring the emulsion to the NMR tube. The mode shifted from 9 μm to 14 μm and 15 μm for the two parallels (tab. 5.4.1.1) (see also chapter 5.4.2, for microscopic images).



Figure 5.4.1.1: Filtration of crude oil emulsion (w/o) through filter 4 with pore size 10 - 16 μm . It looked like the first part passing the filter contained a large amount of water.

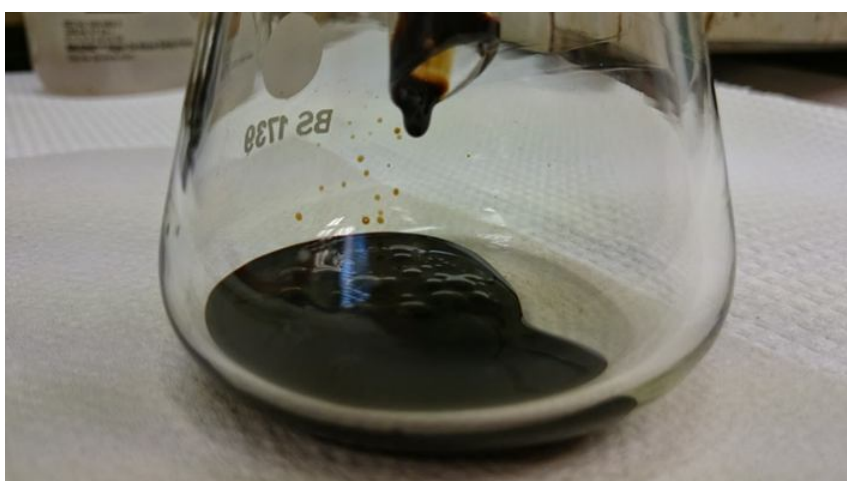


Figure 5.4.1.2: Filtration of crude oil emulsion (w/o) through filter 4 with pore size 10 - 16 μm . It looked like the first part passing the filter contained a large amount of water.



Figure 5.4.1.3: Filtration of crude oil emulsion (w/o) through filter 4 with pore size 10 - 16 μm . The picture shows the water droplets in the bottom of the Erlenmeyer flask after removing a part of the sample for NMR.

For an emulsion containing 15 % WC which was mixed at 2000 rpm for 5 min, the mode shifted from 9 μm to 12 μm , and a concentrated layer of big droplets was detected at the bottom of the NMR tube.

A test was performed for an emulsion with 30 % WC with an even lower mixing speed; 500 rpm for 5 min. The mode shifted from 9 μm to 21 μm after filtration, with droplets up to 137 μm after filtering, which could confirm that the emulsions were too unstable at low mixing speeds.

5.4.2 Microscopy after Filtering Water in Crude Oil Emulsions through Filter 4

Microscopic images of an water in crude oil emulsions prepared by mixing for 5 min at 2000 rpm was captured after filtration through filter 4 (10 - 16 μm). The emulsion was very unstable. The images showed that after filtration double emulsions had appeared, with droplets inside another droplet, which probably was caused by the filtration. Figure 5.4.2.1 – 5.4.2.3, all from this sample, shows this double emulsion. Water in oil in water emulsion has most likely been formed.

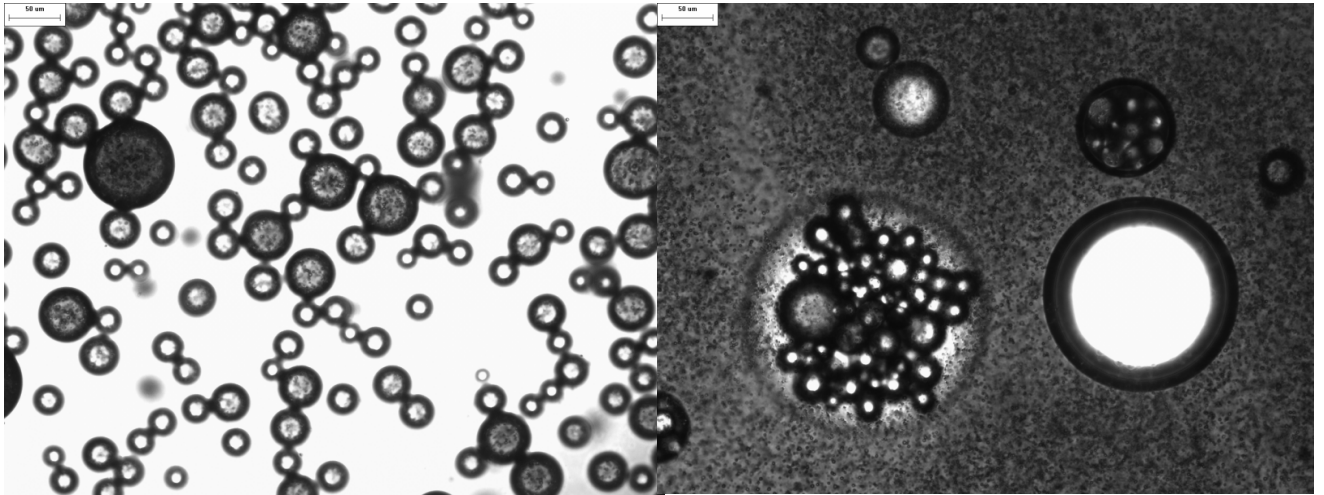


Figure 5.4.2.1: Two microscopic images of one crude oil emulsion after filtering through filter 4 with pore size 10 – 16 μm . 10 times magnification. The white marker in the left corner of the pictures is 50 μm .

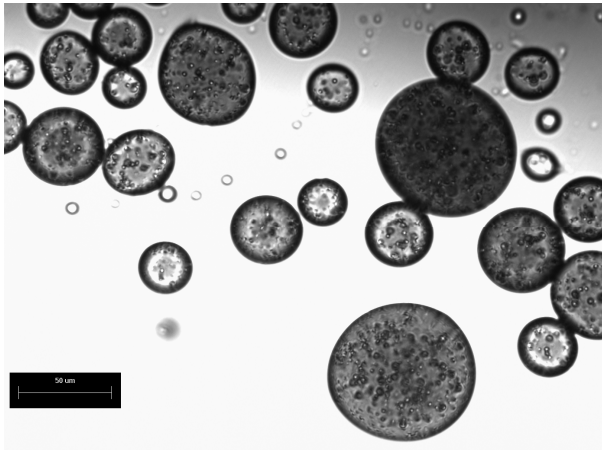


Figure 5.4.2.2: Microscopic images of one crude oil emulsion after filtering through filter 4 with pore size 10 – 16 μm . 10 times magnification. The white marker in the left corner of the pictures is 50 μm .

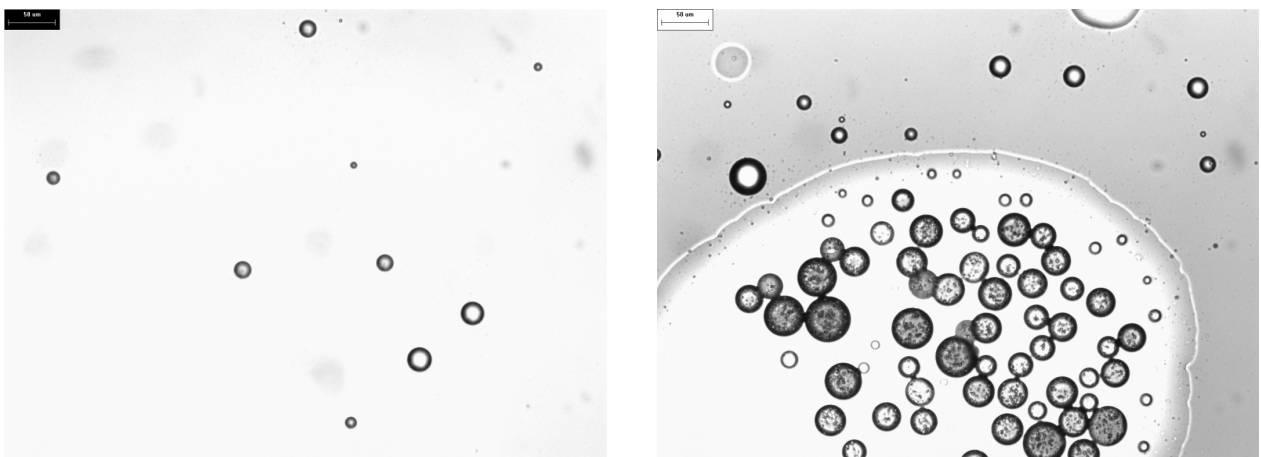


Figure 5.4.2.3: Two microscopic images of one crude oil emulsion after filtering through filter 4 with pore size 10 – 16 μm . 10 times magnification. The white/black marker in the left corner of the pictures is 50 μm .



5.4.3 Primol and Span 80

Similar model oil emulsions as in chapter 5.3.2 with droplet size up to 26 μm were filtered through filter 4 with pore size 10 – 16 μm . The first part passing the filter was a clear slightly viscous liquid, resembling primol. The filtration was very slow and after a while there was a foam-like filtrate. After analyzing the NMR data it could look like the water droplets partly were filtered out of the emulsion with mainly model oil passing the filter since the NMR signal was too low after filtration and the water could not be detected (tab. 5.4.3.1). The brine profile experiment confirmed this since less water was detected after filtration.

Microscopic images showed the same; there was more water droplets present before filtration (fig. 5.4.3.1 and 5.4.3.2).

Table 5.4.3.1: NMR results before and after filtering w/o emulsions through filter 4.

Name of sample	Pump	Pore size	Mixing parameters	DSD before filtering [μm]	DSD after filtering [μm]	Mode before filtering [μm]	Mode after filtering [μm]	Brine profile
Primol and span 80 15 % WC, p1	Hydraulic pump 12 min	Glass filter 10-16 μm	2000 rpm 2 min	1-25	ND	7	ND	Some water was present
Primol and span 80 15 % WC, p2	Hydraulic pump 14 min	Glass filter 10-16 μm	2000 rpm 2 min	1-25	ND	7	ND	Some water was present
Primol and span 80 15 % WC, p3	Hydraulic pump 15 min	Glass filter 10-16 μm	2000 rpm 2 min	2-26	ND	8	ND	Some water was present

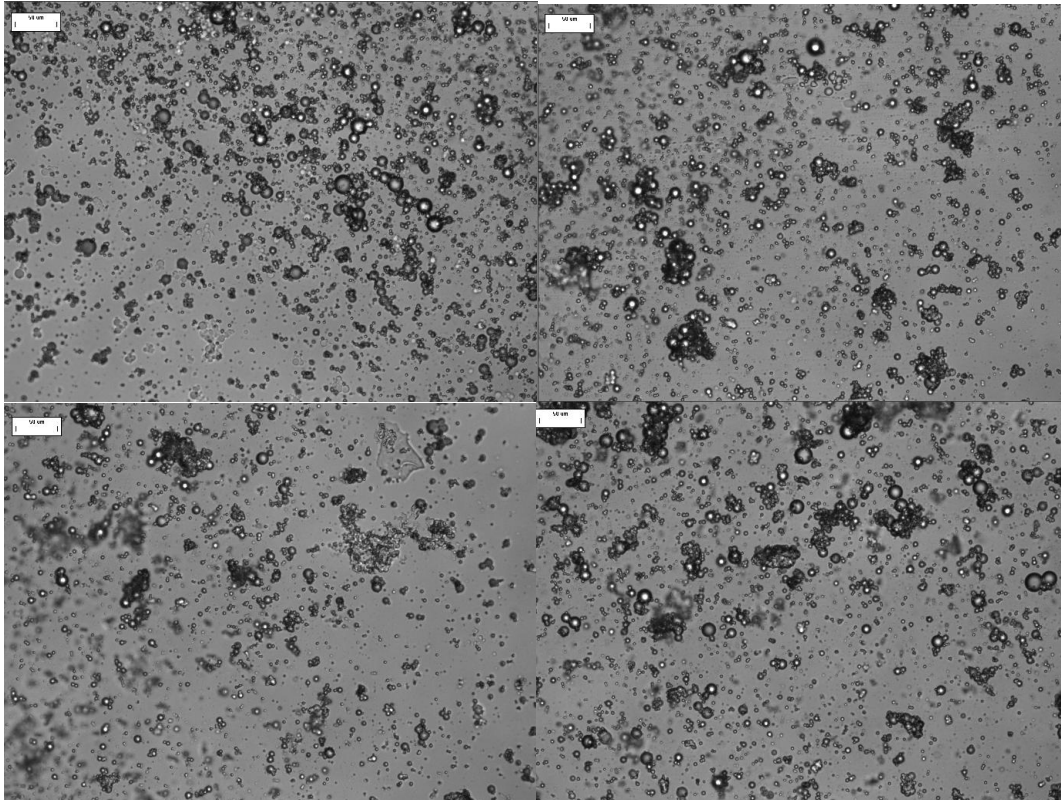


Figure 5.4.3.1: Four microscopic images of one model oil emulsion before filtering. 10 times magnification. The white marker in the left corner of the pictures is 50 μm .

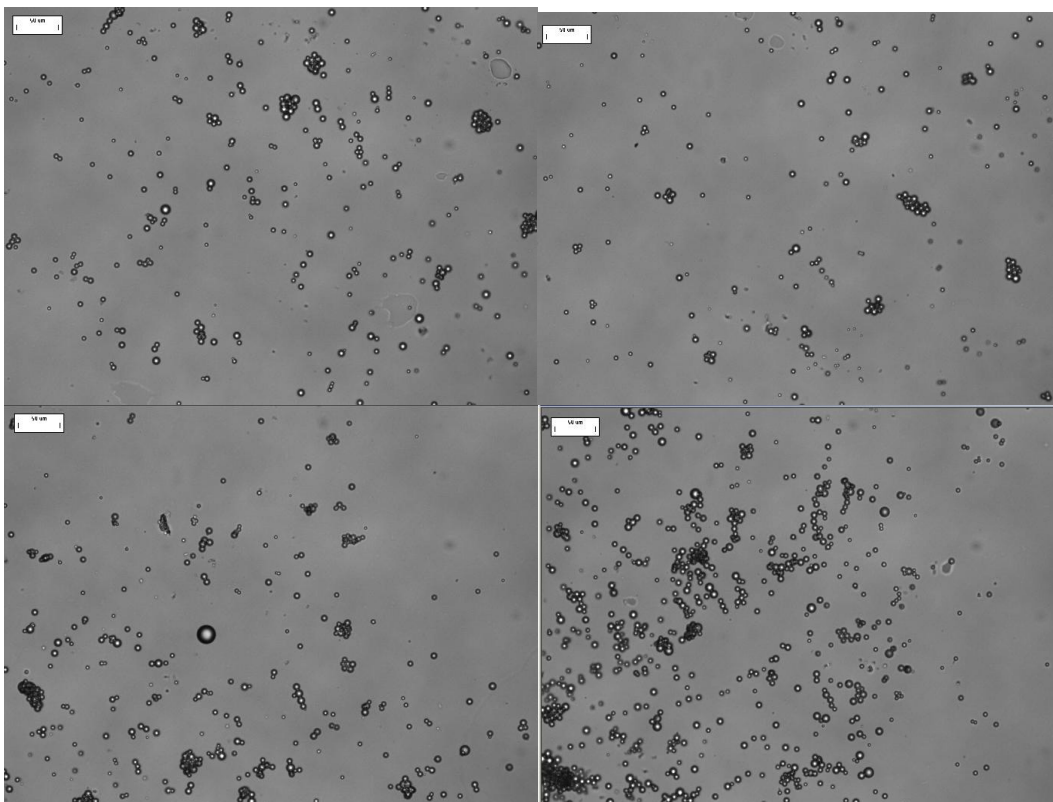


Figure 5.4.3.2: Four microscopic images of one crude oil emulsion after filtering through filter 4 with pore size 10 – 16 μm . 10 times magnification. The white marker in the left corner of the pictures is 50 μm .



5.4.4 Filtration of Unmixed Crude Oil and Water

A layer of crude oil was placed on the filter and a layer of water was poured on top of it. This experiment was performed to see if it was possible to

5.5 Ecrit Behavior before and after Filtration

Ecrit experiments were conducted before and after filtration of model oil emulsion through filter 3 and 4 (tab. 5.5.1). It was observed that when the filtration lasted for a relatively long time (12 and 14 minutes) the ecrit max value was higher, which indicates less water in the sample. This could be explained by unstable emulsions or that the water was filtered out of the emulsion.

Table 5.5.1: Ecrit max before and after filtration.

Porosity	Sample (Primol/ 0.5% Span)	Ecrit max [kV/cm]
3	15wc_before_por3_p1	0,49
3	15wc_after_por3_p1	0,61
3	15wc_before_por3_p2	0,38
3	15wc_after_por3_p2	No ecrit max
4	15wc_before_por4_p1	0,37
4	15wc_after_por4_p1	1,22
4	15wc_before_por4_p2	0,43
4	15wc_after_por4_p2	0,47

6 CONCLUSION

This study demonstrated that stable emulsions with droplet size up to 24 μm seemed to pass the filter with pores down to the range 10-16 μm without coalescing. Emulsions prepared at low mixing speed were either unstable or coalescence occurred during filtration. The filtering process seemed to change the emulsion state under certain conditions. Coalescence can occur, also formation of double emulsions. The clue to better understanding would obviously be linked to the rheological properties of the w/o interface, and especially so the elasticity of the interface.

Future studies should focus on different filters and packed columns of solid particles, different filtration rates, different droplet sizes and surfactant concentrations (giving rise to different interfacial rheologies), in addition development of NMR is needed.



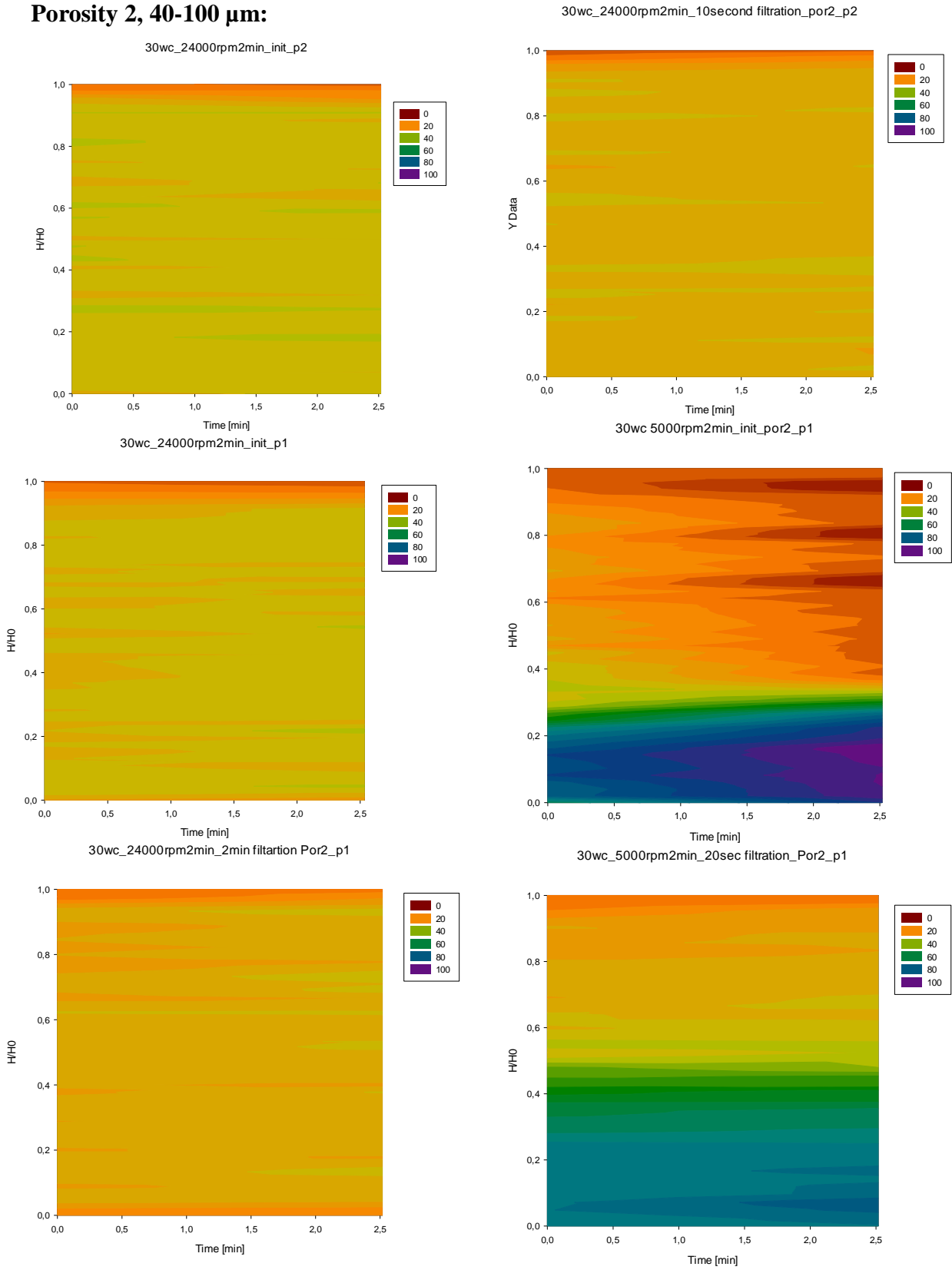
7 APPENDIX

The figures in the appendix show the brine profile before and after filtration. Before filtrations is the figure to the left and the one the right are after filtrations. The blue indicates droplets with size 60-80 μm and purple color indicates free water.



Figures: Crude oil B.

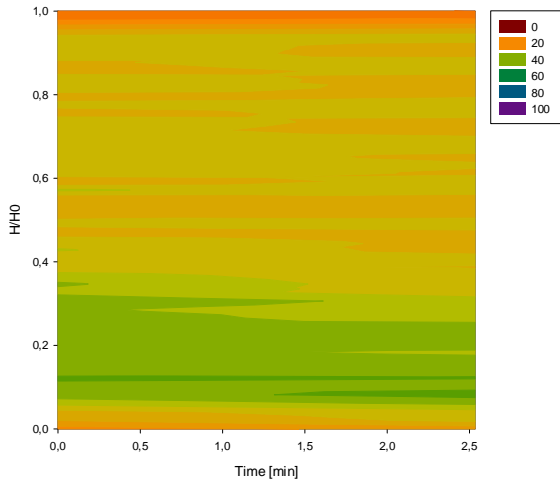
Porosity 2, 40-100 μm :



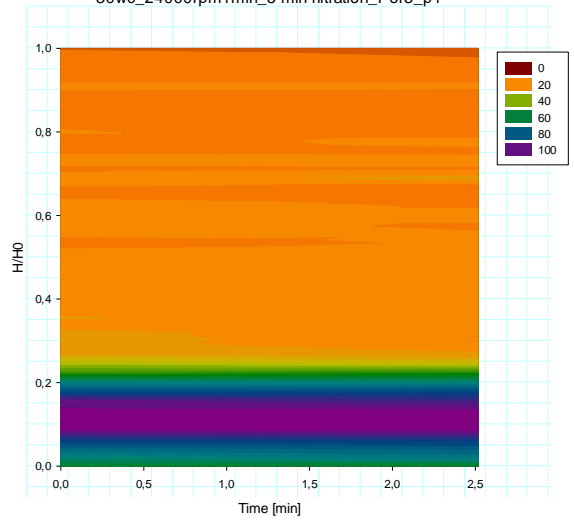


Porosity 3, 16-40 μm :

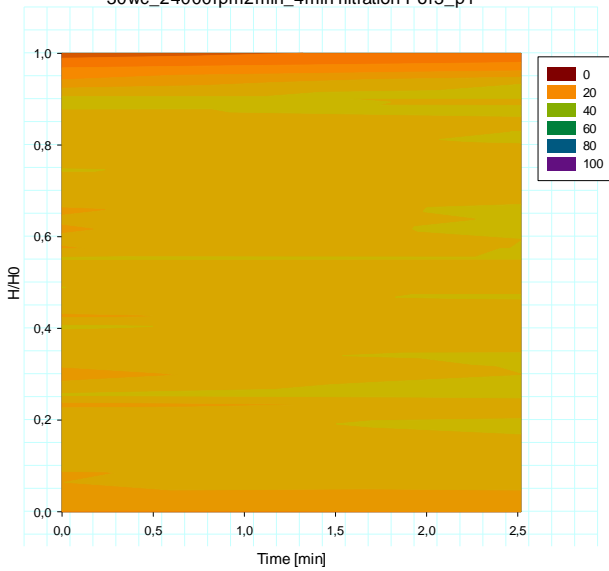
30wc_24000rpm1min_before filtration_Por3_p1



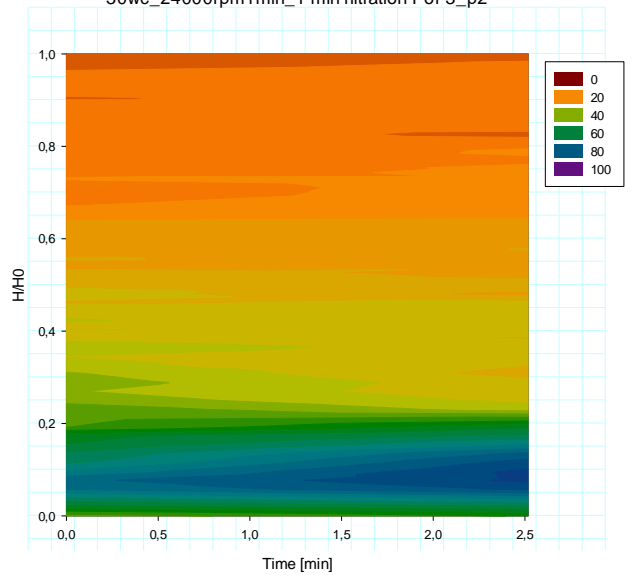
30wc_24000rpm1min_5 min filtration_Por3_p1



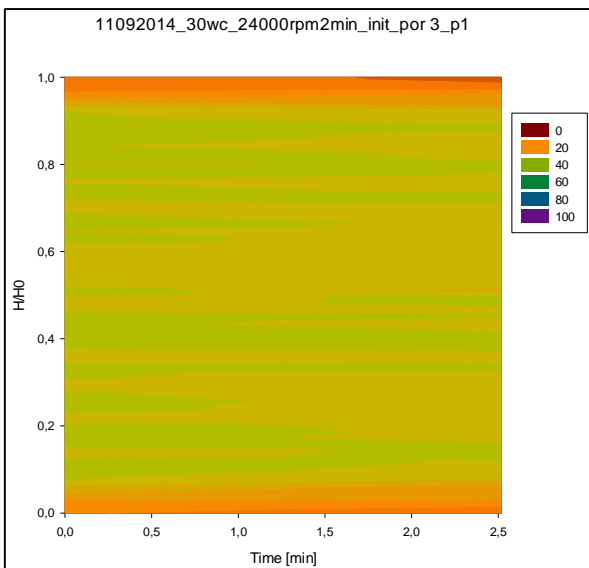
30wc_24000rpm2min_4min filtration Por3_p1



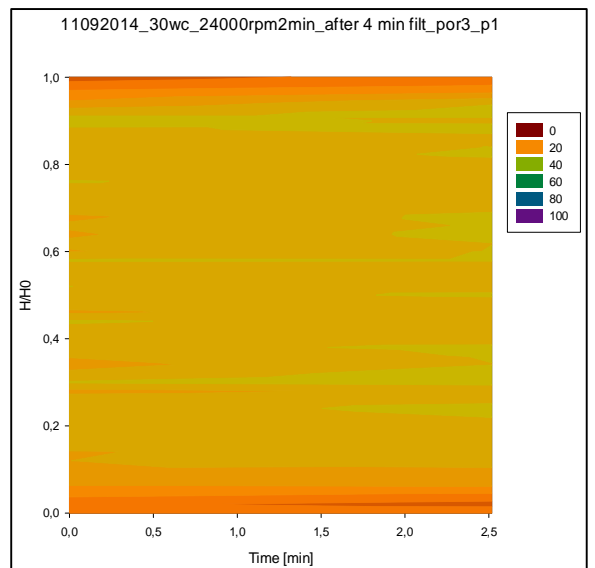
30wc_24000rpm1min_1 min filtration Por 3_p2

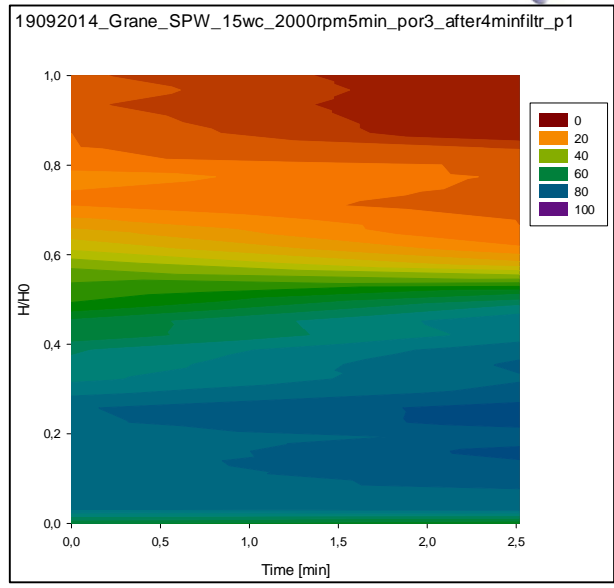
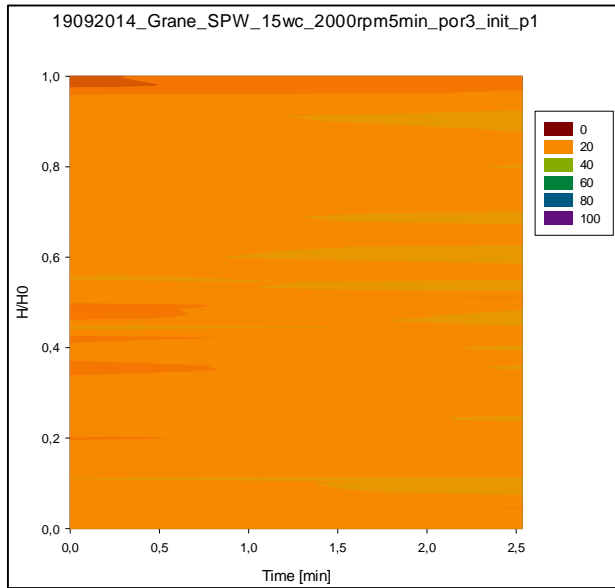


11092014_30wc_24000rpm2min_init_por 3_p1



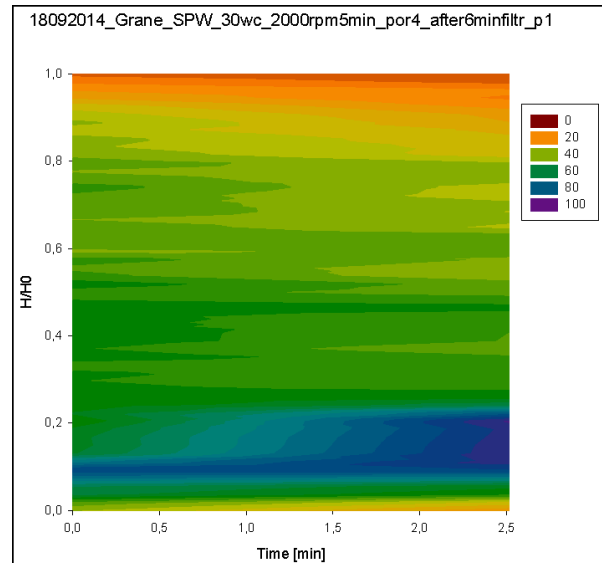
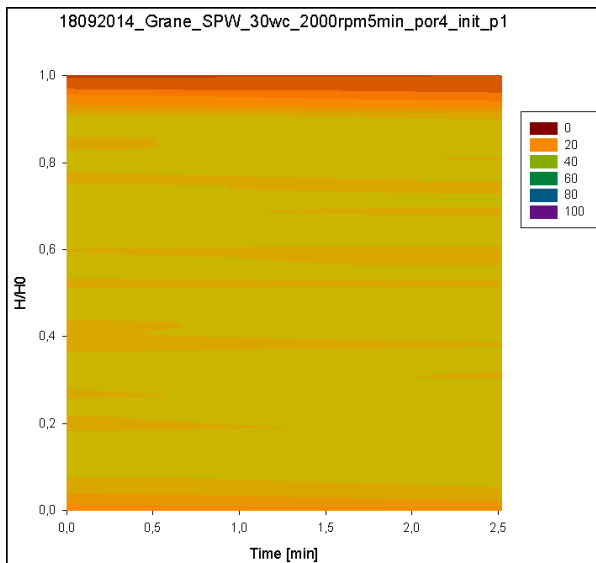
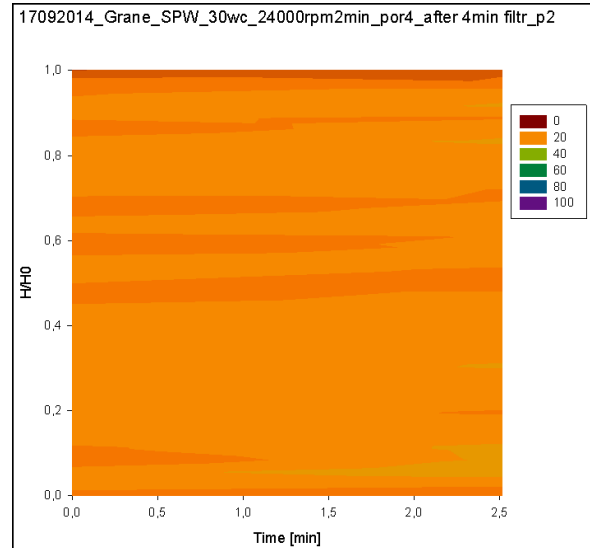
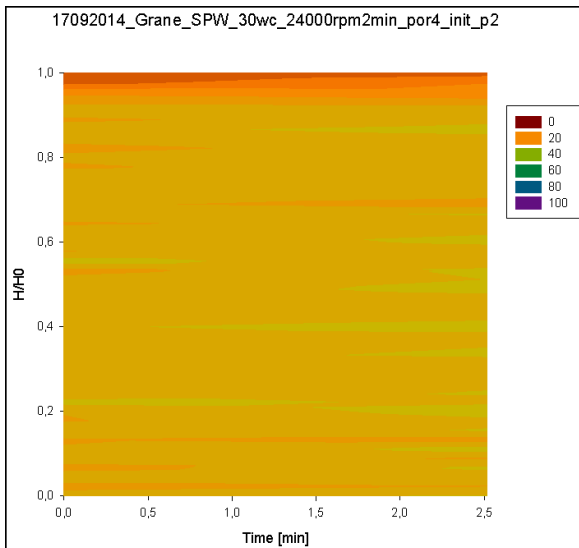
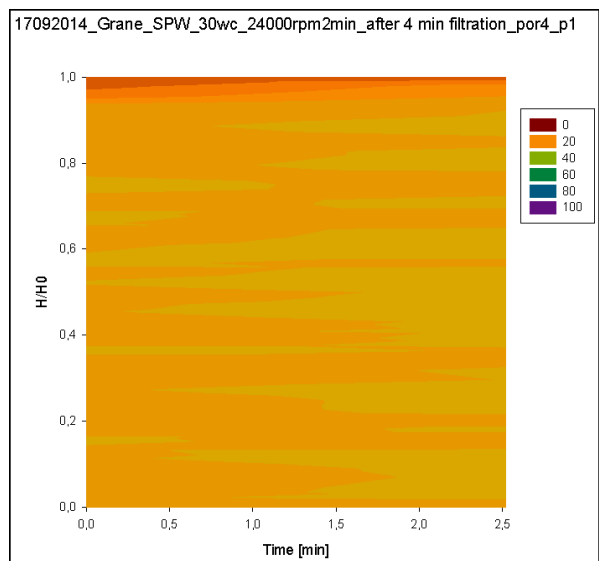
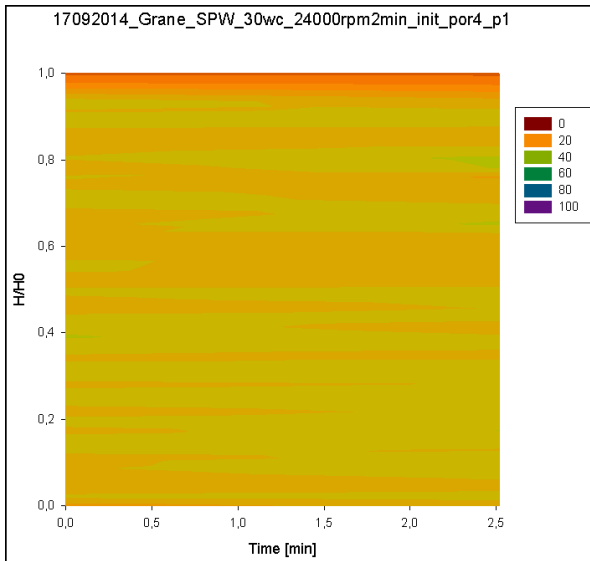
11092014_30wc_24000rpm2min_after 4 min filt_por3_p1

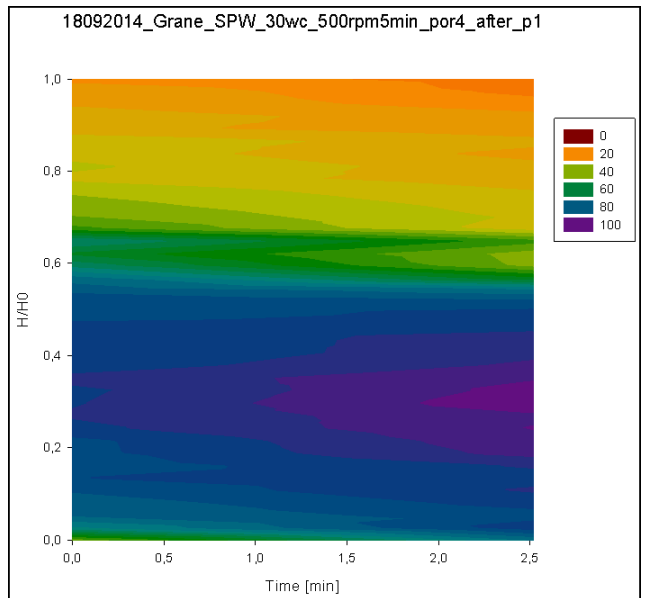
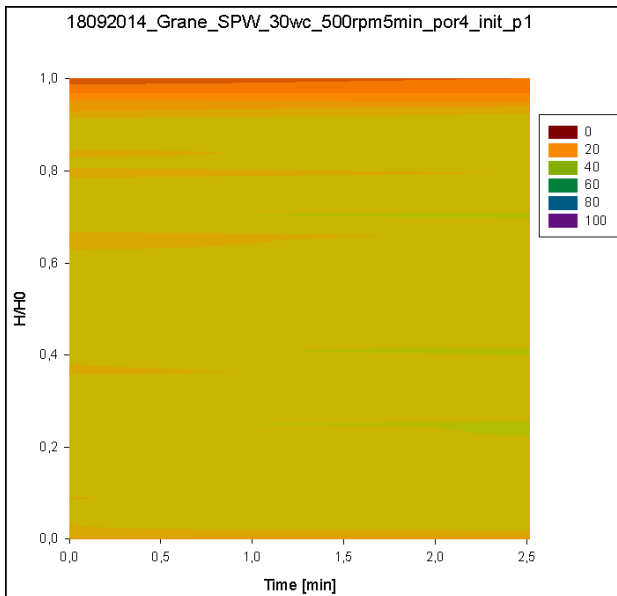
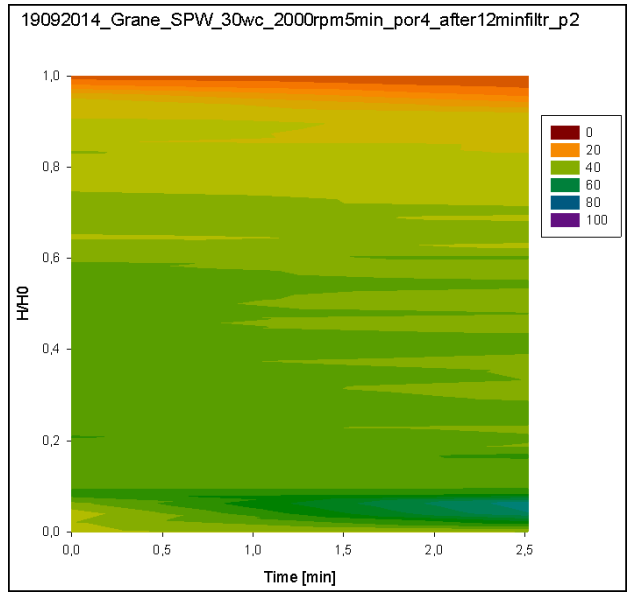
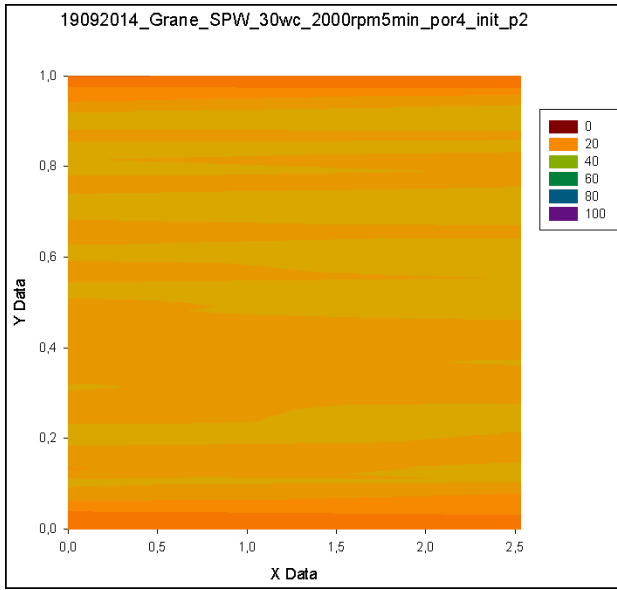






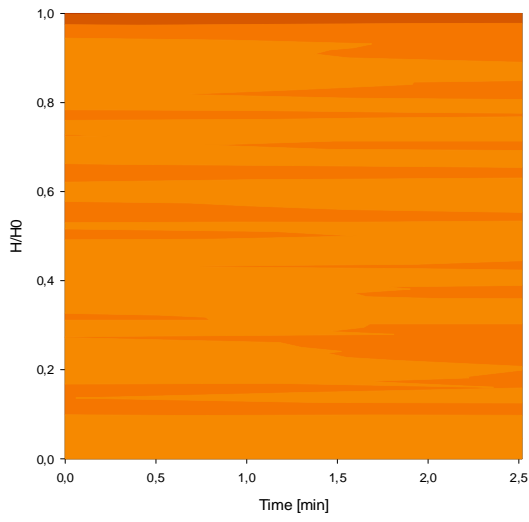
Porosity 4, 10-16 μm :



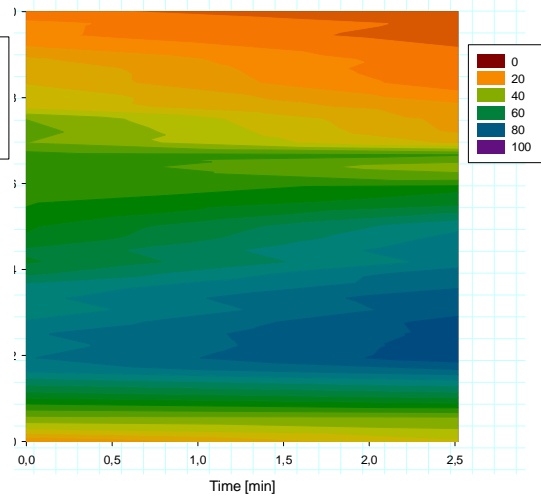




19092014_Grane_SPW_15wc_2000rpm5min_por4_init_p1



2014_Grane_SPW_15wc_2000rpm5min_por4_after5minfiltr_p1

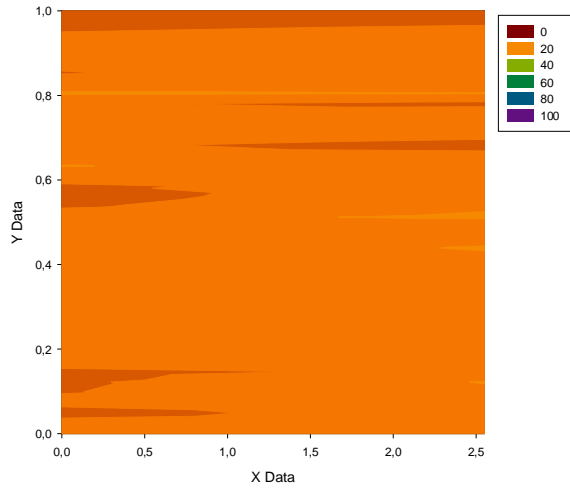




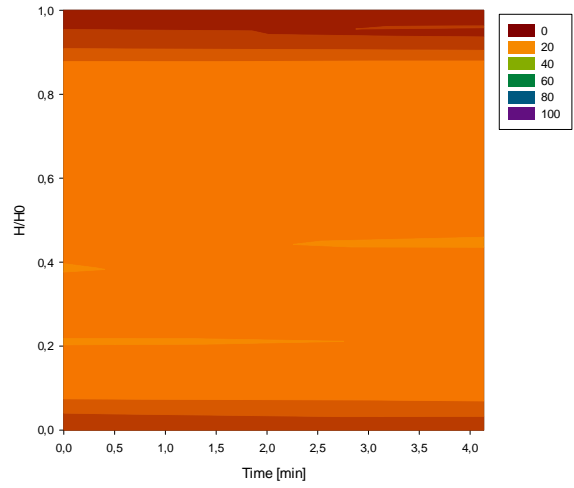
Model oil (primol + span80)

Filter 3

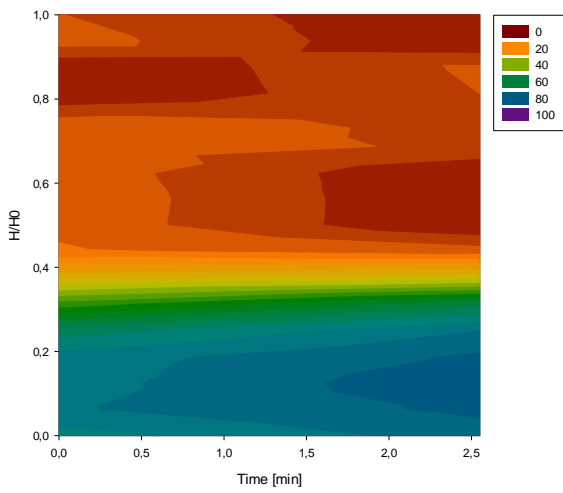
250914_PrimSpanBrine_15wc_2000rpm5min_por3_init_p1



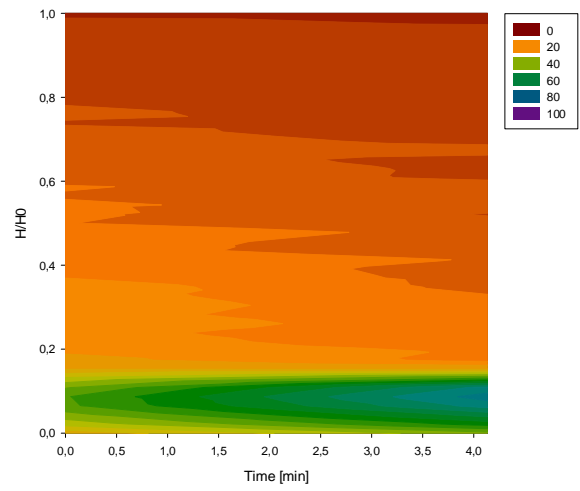
260914_PrimSpanBrine_15wc_2000rpm5min_por3_init_p2



250914_PrimSpanBrine_15wc_2000rpm5min_por3_after 2min40sec filt_p1



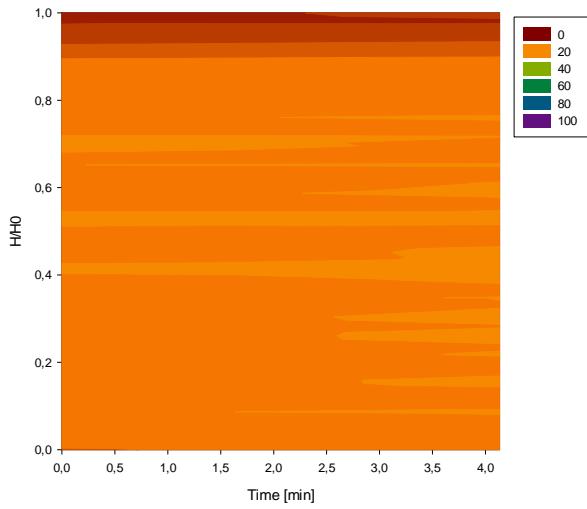
260914_PrimSpanBrine_15wc_2000rpm5min_por3_after3,5minfiltr_p2



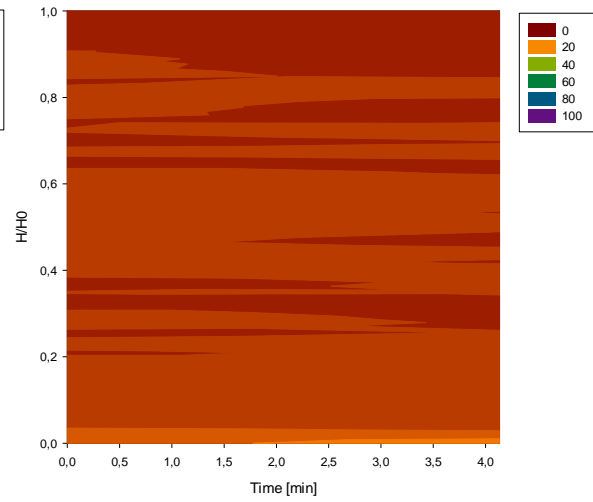


Filter 4

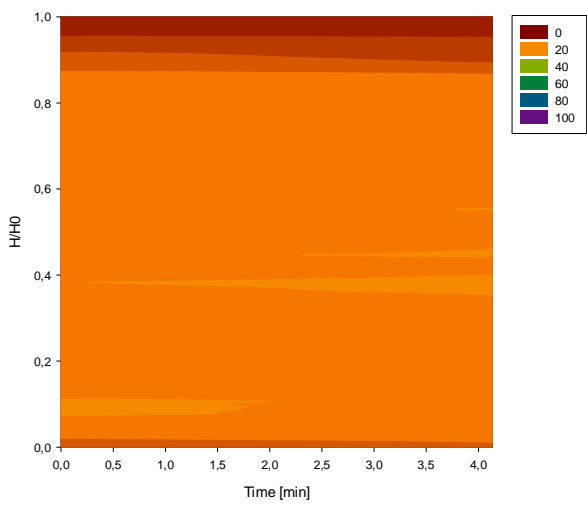
260914_PrimSpanBrine_15wc_2000rpm5min_por4_init p1



260914_PrimSpanBrine_15wc_2000rpm5min_por4_after14minfiltr p1



260914_PrimSpanBrine_15wc_2000rpm5min_por4_init p2



260914_PrimSpanBrine_15wc_2000rpm5min_por4_after7minfiltr p2

