

# Core restoration and its effect on initial wetting of NCS sandstone reservoir cores

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**Abstract.** During the core cleaning and restoration process several crude oil – brine – rock factors affect the restored core wettability. Different solvents used during cleaning as well as the volume of oil flooded into the core are some of them. It has been observed in previous experiments, that different types of solvents can wash away different amounts of oil, influencing the later adsorption process of crude oil components. Similarly, increased amount of crude oil exposure affects the wettability of the system toward less water-wet state. Preserved reservoir cores from the Norwegian Continental Shelf were cleaned using kerosene and heptane and using toluene and methanol. In this paper, the effect of multiple core cleaning and restoration processes on initial wettability and on the ultimate oil recovery by spontaneous imbibition experiments was investigated. Not only did the two cleaning procedures affect wettability differently, but the experiments also showed that the cleaning method significantly influenced the wettability in the subsequent core restoration of this reservoir core material. For the cores cleaned with kerosene/heptane, cumulative increased crude oil exposure during the core restorations resulted in lower oil recovery by SI, thus the cores appeared less water-wet. The cores cleaned with toluene/methanol, better reproduced the previous wettability. Whether this restored wettability is more representative of the reservoir wettability is still unknown. However, it seemed that 5 pore volumes of crude oil exposure in each core restoration generated a reasonable wettability in these initially preserved reservoir cores.

## 1. Introduction

Several laboratory core experiments the past decade [1-14] have shown that the ionic composition of the brine injected can alter the wettability and drive more oil to production. In sandstone cores, the extra oil produced during Smart Water injection was accompanied by an increase in the produced water pH [15]. Surface chemistry phenomena like adsorption and desorption of ions on the mineral surfaces could increase the pH of the system leading to a more water-wet surface because of crude oil component desorption [2].

To observe any enhanced oil recovery (EOR) because of wettability alteration during Smart Water or low salinity brine injection, the reservoir needs to be in a mixed-wet state. If the reservoir is already very water-wet the potential for observing any extra oil production by modified water injection, is low. Therefore, the initial wettability of a reservoir and its correct determination is of utmost importance. However, knowing the initial wettability of the reservoir is very challenging because there exists, to date, no direct in-situ wettability measurement tools. Instead, the reservoir wettability is determined based on laboratory special core analysis

(SCAL) work on preserved reservoir material and reservoir log data.

From previous experiments on outcrop chalk cores, it was observed that different types of solvents remove different amounts of oil components from mineral surfaces and salts influencing the wettability, and later oil adsorption process during core restoration [16]. Similarly, increased exposure to crude oil during core restoration affected the wettability of chalk toward less water-wet [17].

In this paper, the effect of the core cleaning processes; by kerosene and n-heptane and by toluene and methanol, on initial wettability and on the ultimate oil recovery from reservoir sandstone cores after core restoration was investigated and compared by spontaneous imbibition (SI) experiments.

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## 2. Experimental

### 2.1. Core material

Reservoir core material was retrieved from two seal peels cored from the Varg field on the Norwegian Continental Shelf (NCS). The analysed cores were obtained from the upper sequence (at depths less than 3479.50m) of the 9S well and from the lower sequence (at depths over 3493.75m) of the A5T2 well. More specifically, cores P1 and P3 from the 9S well were located at 3404.83m and

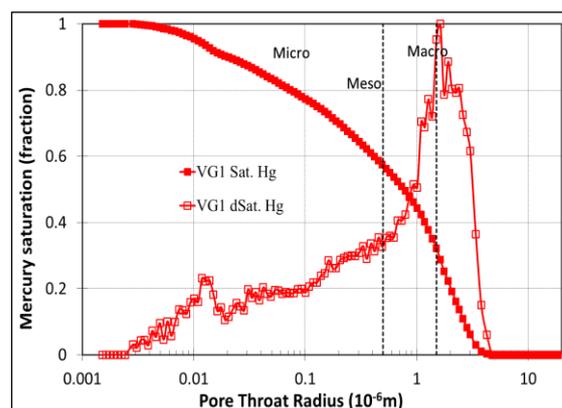
**Table 1:** Mineralogy of the core material

Mineral	Well	
	9S	A5T2
Illite+Mica [wt%]	15.4	9.5
Kaolinite [wt%]	1.1	1.1
Chlorite/Smectite [wt%]	0.1	0
Chlorite [wt%]	2.4	3.4
Quartz [wt%]	62.3	67.6
K Feldspar [wt%]	4.5	5.6
Plagio-clase [wt%]	8.4	8.7
Calcite [wt%]	0	0.6
Dolomite [wt%]	3.9	2.8
Siderite [wt%]	1.1	0
Pyrite [wt%]	0.8	0.8
<b>Total [wt%]</b>	<b>100</b>	<b>100</b>

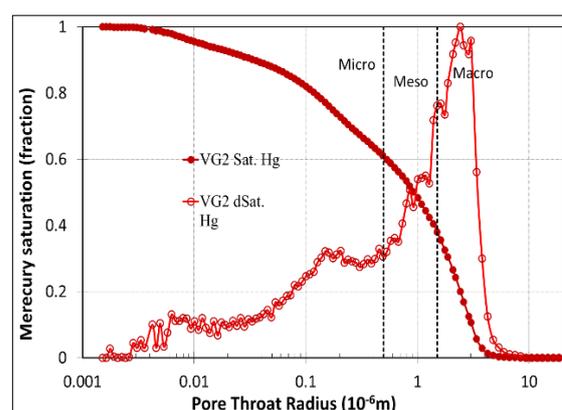
3404.88m depth, respectively, whereas the cores P9 and P10 were located at depths of 3528.82m and 3528.87m, respectively. The company operating the Varg field provided the mineralogy of the core material, and the data are given in **Table 1**. The physical properties of the core material are given in **Table 2**. Permeability was measured at  $S_{or}$  during the flooding of LS brine in the core cleaning process (described later), and it varied between 2-22 mD in the A5T2 cores and between 5-17 mD in the 9S cores. The pore size distribution of the core material was determined by Stratum Reservoir and the results are shown in **Fig. 1** and **Fig. 2** respectively.

**Table 2:** Core properties

Core	P1	P9	P2	P10
Well	9S	A5T2	9S	A5T2
<b>Cleaning</b>	Kerosene/Heptane		Toluene/Methanol	
<b>Length (cm)</b>	8.85	8.22	8.18	8.18
<b>Diameter (cm)</b>	3.80	3.80	3.80	3.80
<b>Bulk volume (cm<sup>3</sup>)</b>	100.4	93.2	92.8	92.8
<b>Dry weight (g)</b>	203.52	179.65	188.47	167.62
<b>Pore volume (ml)</b>	21.27	21.58	21.40	24.97
<b>Porosity (%)</b>	21.2	23.2	23.1	26.9
<b>Permeability (md)</b>	11.3	3.5	10.9	22.4



**Fig. 1:** Pore size distribution for the 9S well.



**Fig. 2:** Pore size distribution for the A5-T2 well.

From the results it is clear that the core material from both wells have heterogeneous pore size distributions, with pore throat radii varying between 0.002 to 4  $\mu\text{m}$ , with a large proportion of meso and micro pores.

During the core restoration procedure described later, the cores P1 and P9 from the 9S and A5T2 wells, respectively, underwent a kerosene/heptane cleaning restoration process, whereas the cores P2 and P10 from the 9S and A5T2 wells, respectively, were restored using a toluene/methanol cleaning restoration process.

### 2.2. Brines

The brines used for this study were two synthetic brines that were prepared in the laboratory; Varg formation water (FW) with total dissolved solids (TDS) or salinity of 201600 ppm and a low salinity brine (LS) consisting of 1000 ppm NaCl. Fivefold diluted Varg FW (d5FW) was used in the establishment of initial water saturation ( $S_{wi}$ ) using the desiccator technique (described later). The properties and composition of the brines are given in Error! Reference source not found..

**Table 3:** Brine properties

Ions	LS	FW	d5FW
[Na <sup>+</sup> ], mM	17.1	2086.0	417.2
[K <sup>+</sup> ], mM	-	51.0	10.2
[Ca <sup>2+</sup> ], mM	-	536.0	107.2
[Mg <sup>2+</sup> ], mM	-	144.0	28.8
[Cl <sup>-</sup> ], mM	17.1	3526.0	705.2
[Ba <sup>2+</sup> ], mM	-	7.0	1.4
[Sr <sup>2+</sup> ], mM	-	8.0	1.6
Density (g/cm <sup>3</sup> )	0.999	1.139	1.027
Bulk pH	5.7	5.86	5.75
Viscosity (cP)	0.945	1.45	1.01
TDS (mg/L)	1000	201600	40.3

### 2.3. Oils

Stock tank reservoir crude oil from the Varg field was used in the oil recovery tests by SI. The crude oil acid number (AN) and base number (BN) were analysed by potentiometric titration according to the procedures described by Fan and Buckley [18], procedures that are modified from the standard methods ASTM664-89 and ASTM2896-88 for acid and base number measurements, respectively. The density of the crude oil was measured at ambient conditions using an Anton Paar densitometer. The viscosity of the crude oil was measured by an MCR 302 rheometer by Anton Paar at 23 and 60 °C. The crude oil properties are presented in **Table 4**.

**Table 4:** Crude oil properties

Crude Oil	Varg
AN (mgKOH/g)	0.13
BN (mgKOH/g)	1.25
Viscosity at 60°C (cP)	4.3
Viscosity at 23°C (cP)	11.3
Density at 23°C (g/cm <sup>3</sup> )	0.845

### 2.4. Chemicals

During core cleaning, several chemical solvents were used; low aromatic kerosene, n-heptane (hereafter referred to as heptane) having reduced solubility of larger oil components, toluene and methanol. Toluene is a strong solvent that efficiently removes heavy oil components whereas methanol can dissolve oil and water. The above chemicals were purchased from Merck laboratories.

### 2.5. Core cleaning and restoration

In this work two different cleaning procedures used in the core wettability restoration process were compared. Note that the procedures used are chosen for comparative purposes and are not necessarily examples of what is considered current best practices for core restoration procedures.

#### 2.5.1. Core cleaning

A Hassler core holder was used for all cleaning processes. This equipment consisted of two threaded ends, two pore fluid lines (distributors) and a viton type rubber sleeve. The system was supported by a confining pressure at 10 bars using water or nitrogen as the confining fluid. A back pressure of 4 bars was used to avoid two phase flow and the inlet and outlet pressures were monitored by pressure gauges. Fluids were injected through the system by a piston cell connected to an HPLC pump. All cleaning processes were performed at room temperature (23 °C) and at a rate of 0.1 ml/min.

#### 2.5.2. Core cleaning solvents

Two different core cleaning procedures were used in this comparative study: (1) kerosene/heptane cleaning and (2) toluene/methanol cleaning. The kerosene/heptane core cleaning procedure was developed in-house, while the toluene/methanol cleaning procedure is representative of the standard methods preferred by the industry. The same amounts of solvents were injected in each restoration process using either method.

##### Kerosene/heptane cleaning

The cores P1 and P9 were first flooded with approximately 10 pore volumes (PV) of kerosene until a clear effluent was obtained. Kerosene flooding was succeeded by the flooding of 5 PV of heptane to displace the kerosene. Finally, the cleaning process was completed by injecting 10 PV of LS brine to displace the heptane and remove easily dissolvable salts.

The aim of this suggested cleaning procedure is to preserve the initial reservoir wettability by only displacing the mobile crude oil during core cleaning, leaving the adsorbed oil components and initial wettability intact, and to replicate the initial wettability in multiple core restorations. Low-aromatic kerosene is used to remove the mobile oil phase and non-polar oil constituents, whereas heptane finally displaces the kerosene fraction. LS brine is injected to displace the FW and any easily dissolvable salts while preventing the clays from swelling.

##### Toluene/methanol cleaning

This cleaning procedure included the usage of toluene and methanol, injected in two repeated cycles. Firstly, cores P2 and P10 were flooded with 5 PV of toluene followed by 5 PV of methanol. This process was repeated once using the same amount of solvents. Finally, the cleaning process was completed after injecting 10 PV of LS brine. Toluene/methanol cleaning scheme is a well-known, standard cleaning method, which, in contrast to kerosene/heptane cleaning, tends to remove most of the material that contribute to the natural wettability of the rock. Toluene removes the oil phase, including polar organic components, asphaltenes and bitumenic precipitates, while methanol dissolves polar compounds (e.g. resins), water and precipitated salts.

### 2.5.3. Establishing initial water saturation, $S_{wi}$

Initial water saturation ( $S_{wi}$ ) was established using the desiccator procedure described by Springer et al. [19] After the cleaning procedure, the cores were dried at 90 °C, until constant weight. Then they were vacuum-saturated with five times diluted FW ( $d_5FW$ ). Subsequently, a desiccator was used to gradually dry the cores to a predetermined weight, corresponding to 20%  $S_{wi}$ , which was chosen for this study. Finally, the cores were stored for 3 days in order to ensure an even brine distribution inside the core.

### 2.5.4. Crude Oil Exposure and aging

At  $S_{wi}$ , the Varg cores were exposed to a total of 5 PV of Varg crude oil. First the core was shortly vacuumed and saturated with crude oil in the Hassler core holder. Due to the heterogeneity of the porous media, oil flooding from one direction might not be sufficient. Therefore, the cores were flooded from both sides, 2PV in each direction at a rate of 0.1 ml/min. The above process took place at 50 °C to facilitate the oil flooding process. To finish, the cores were placed in aging cells surrounded with Varg oil for 14 days at 60 °C.

## 2.6. Spontaneous imbibition tests

A spontaneous imbibition (SI) test is a method for evaluating the wettability of a crude oil – brine – rock (COBR) system. The test consists of an Amott [20] cell, which contains a core submerged in oil or water. If the fluid that surrounds the core is the wetting phase of the system, then it will imbibe into the pores of the core displacing the fluid that already exists inside. The imbibition of the fluid is monitored with time, by collecting the displaced fluid in a graded burette. Evaluation of wettability with this method can be done by observing the ultimate recovery of the fluid displaced and the rate of imbibition of the wetting phase.

The aged core, containing crude oil and  $S_{wi}$  of 20 %, was placed on top of three marbles inside the Amott imbibition cell and surrounded by the imbibition brine. FW was used to evaluate the wettability of the core without causing any chemical induced wettability alteration during imbibition. The FW is already in chemical equilibrium with the COBR-system. The produced crude oil was collected in a graded burette, and the recovery in %OOIP (oil originally in place), was determined versus time of imbibition. After the SI experiment was terminated the core was again cleaned and restored with initial water and oil and aged in a subsequent restoration process.

## 3. Results and discussion

The purpose of this study was to evaluate the wettability of the Varg reservoir cores after performing two distinctly different core cleaning methods using toluene/methanol and kerosene/heptane cleaning approaches. Secondly, the

effect of crude oil exposure into the two differently cleaned sandstone rock systems was evaluated.

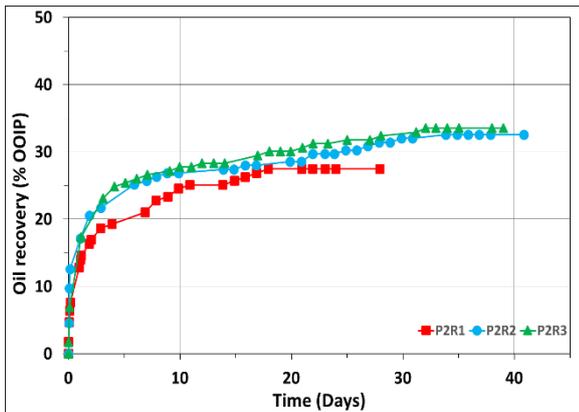
### 3.1. Restoration effects on wettability in toluene/methanol cleaned cores

Two reservoir cores, P2 and P10, were cleaned with toluene and methanol, which are the standard cleaning solvents used by the industry to clean reservoir cores in core restoration procedures [16]. Only core flooding was used in the cleaning process, no Soxhlet extraction was performed. Toluene is a good solvent, and the purpose of this cleaning procedure is to efficiently clean the core toward a more water-wet state. Thereafter, the initial water saturation was established, before crude oil exposure (5PV) and aging for 14 days at 60 °C. After completing the aging period, the cores were put for SI with FW brine to evaluate the wettability of the core. By imbibing FW there was no chemical induced wettability alteration taking place as the core was already in equilibrium with its formation brine, used for establishing  $S_{wi}$ . The oil recovery by SI curves are shown in Fig. 3 and Fig. 4 for core P2 and P10, respectively. After the first restoration (R1) both cores behaved mixed-wet but clearly on the water wet side as shown by the production profile and quite high ultimate oil recoveries of 27 and 40 %OOIP, respectively. When oil production ceased and the plateau had been reached, the cores underwent a subsequent restoration process involving cleaning,  $S_{wi}$  establishment, crude oil exposure and aging according to the same procedures as described above in section 2.5. After new core cleaning/restoration processes additional second (R2) and third (R3) restoration oil recovery tests were performed, and only small differences in recovery were observed.

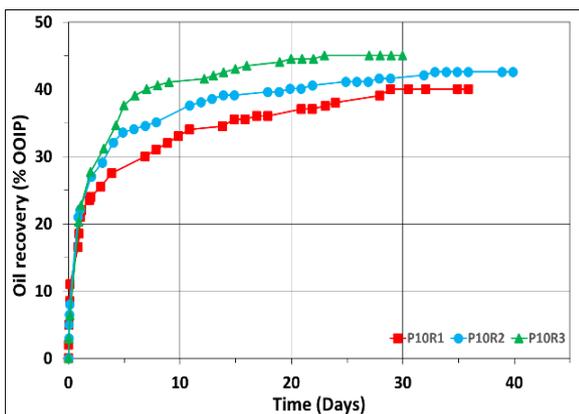
The *P2 core* produced a total amount of 27%, 33% and 34% OOIP after first, second and third restoration respectively.

The *P10 core* produced a total amount of 40%, 43% and 45% OOIP after first, second and third restoration respectively.

In summary, the toluene/methanol cleaned cores P2 and P10 showed a small change regarding wettability alteration toward slightly more water-wet conditions after each restoration process. This might suggest that toluene and methanol efficiently removed adsorbed organic components before the wettability restoration, and that the increased amount of solvents flooded through the core by time in multiple restorations improved the water wetness of the reservoir cores.



**Fig. 3:** SI experiments at 60 °C of the toluene/methanol cleaned core P2 with FW as imbibing fluid. R1, R2 and R3 refer to restoration number 1,2 and 3, respectively.



**Fig. 4:** SI experiments at 60 °C of the toluene/methanol cleaned core P10 with FW as imbibing fluid. R1, R2 and R3 refer to restoration number 1,2 and 3, respectively.

### 3.2. Restoration effects on wettability in kerosene/heptane cleaned cores

The kerosene/heptane core cleaning procedure is used with the aim of preserving the initial wettability during core cleaning. Core P1 and core P9 were cleaned with kerosene and heptane followed by the same restoration process as that for the toluene/methanol cleaned cores. Thus, the only difference was the core cleaning procedure. After completed aging, the cores were put for SI with FW to evaluate initial wettability.

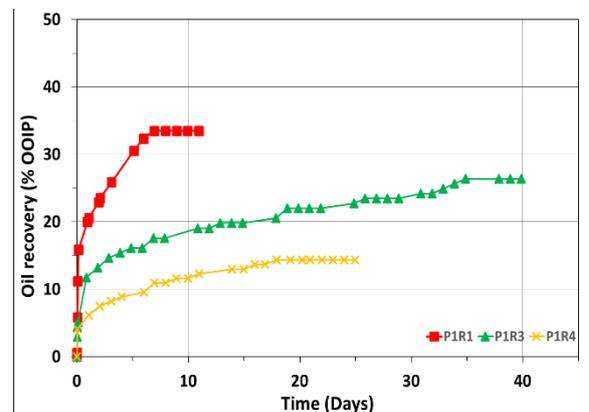
The oil recovery by SI curves are shown in **Fig. 5** and **Fig. 6**. After the first restoration (R1) both cores behaved mixed-wet, but clearly on the water wet side as shown by the production profile with ultimate oil recoveries of 34 and 43 % OOIP, for P1 and P9 respectively. When oil production ceased and the plateau had been reached, the cores underwent a subsequent restoration process involving cleaning,  $S_{wi}$  establishment, crude oil exposure and aging according to the same procedures as described above in section 2.5. After the kerosene/heptane core cleaning and an additional second (R2), third (R3) and fourth (R4) restoration the oil recovery tests were repeated. In contrast to the results observed with toluene/methanol cleaned cores, large restoration effects

were observed, even though the same core treatments were performed in each restoration. The kerosene/heptane cleaned cores P1 and P9 showed significant wettability alteration toward less water-wet state.

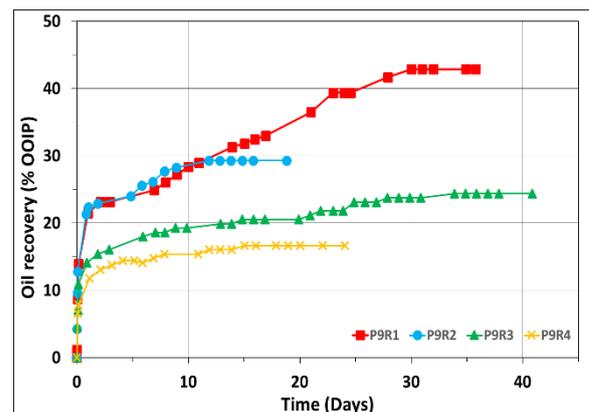
The *P1 core* produced a total amount of 34%, 25% and 14% OOIP after the first, third and fourth restoration, respectively, as clearly seen in **Fig. 5**.

The *P9 core* produced a total amount of 44%, 29%, 24% and 17% OOIP after the first, second, third and fourth restorations, respectively, shown in **Fig. 6**.

Since kerosene and heptane cleaning is aimed at preserving wettability in the cores and only desorbing a limited amount of crude oil components from the rock surface, the cumulatively increased oil exposure after every restoration lead to a decrease in water wetness in the core. The kerosene/heptane cleaning procedure is confirmed to not remove all adsorbed crude oil components from the surface during cleaning. These results for reservoir cores are in agreement with similar experiments on outcrop sandstone cores [3]. From these results it can also be found that 5 PV of Varg crude oil exposure is too extensive for reproducing wettability between restorations after kerosene/heptane cleaning. Future studies should focus on finding the optimum amount of Varg crude oil exposure to reproduce wettability in these reservoir cores.



**Fig. 5:** SI experiments at 60 °C of the kerosene/heptane cleaned core P1 with FW as imbibing fluid. R1, R3 and R4 refer to restoration number 1, 3 and 4, respectively.



**Fig. 6:** SI experiments at 60 °C of the kerosene/heptane cleaned core P9 with FW as imbibing fluid. R1, R2, R3 and R4 refer to restoration number 1, 2, 3 and 4 respectively.

### 3.3. Representative reservoir wettability?

In this paper the effect of cleaning solvents on initial wettability was investigated. All oil recovery results using both cleaning procedures are summarized in **Table 5**.

If the results after toluene/methanol and kerosene/heptane cleaning procedures are compared for each well, it can be seen that the ultimate oil recoveries after R1 are quite similar, although there is a slight tendency of the kerosene/heptane cleaned cores P1 and P9 behaving somewhat more water-wet than their toluene/methanol cleaned sister cores P2 and P10. At this point, it is not clear whether these results are because of core heterogeneity effects, or if they are real core cleaning effects. Reservoir cores are generally more heterogeneous than outcrop cores, thus direct comparisons between sister cores are in some cases difficult. Apart from that it could be that more rigorous cleaning by toluene/methanol allows for a larger extent of adsorption of polar crude oil components than does kerosene/heptane cleaning, which leaves most of the already adsorbed crude oil components on the rock surface, perhaps limiting more oil component adsorption. However, this is speculations, and further studies are needed to understand this observation.

Previous studies on both sandstone and carbonate outcrops have shown that increased amount of crude oil exposure into the core during core restoration decreases water wettability [16], [17]. Therefore, the question is which core cleaning and restoration method better represents the true wettability of the reservoir. What is important to highlight when interpreting these results is that the amount of crude oil exposure was the same and equal to 5 PV in both restoration procedures, thus only small differences in initial wettability should be expected. However, if crude oil exposure increased, then larger deviations from true reservoir wettability should be expected. Therefore, a kerosene/heptane cleaning procedure aiming to preserve initial wettability in reservoir cores is presumed being a better approach. The challenge is knowing how many PV of crude oil should be injected into a toluene/methanol cleaned core to obtain a representative reservoir wettability. By comparing kerosene/heptane and toluene/methanol cleaned core it seems like 5 PV of Varg crude oil injection is reasonable in these Varg reservoir cores.

**Table 5:** SI summary results

Core	Cleaning system	SI (%OoIP)			
		R1	R2	R3	R4
P1	Kerosene/Heptane	34	-*	25	14
P9		43	29	24	17
P2	Toluene/Methanol	27	33	34	-
P10		40	43	45	-

\*Spontaneous imbibition experiment failed

## 4. Concluding remarks

The effect of two different cleaning systems on the wettability of sandstone reservoir cores retrieved from the NCS were examined in this study. Kerosene/heptane and toluene/methanol cleaning were used during the experimental procedures. Kerosene/heptane cleaning is aimed at preserving wettability during the core restoration procedure, while the toluene/methanol cleaning is aimed at cleaning the core to water-wet conditions followed by crude oil component adsorption during the core wettability restoration.

Multiple restorations under kerosene/heptane cleaning conditions showed significant alteration of the wettability toward less water-wet state because of successive adsorption of organic polar components onto the sandstone surface after each restoration process. Preservation of polar organic components and thus preservation of the initial wettability of the system is an indication that kerosene/heptane cleaning solvents do not significantly interfere with the natural wettability of the system.

On the other hand, the toluene/methanol cleaning approach showed opposite results, where the cores seemed to become slightly more water-wet after multiple restorations. This observation indicates that the usage of stronger cleaning solvents removes crude oil wetting compounds that are responsible for the initial wettability of the rock, and that increased cleaning solvent volumes improve water wetness.

Based on the results obtained in this study, it seems that toluene/methanol cleaned Varg reservoir cores followed by 5 PV Varg crude oil exposure generated a wettability similar to that of the kerosene/heptane cleaned Varg cores. It must, however, be noted that the amount of crude oil exposure is of extreme importance, because increased exposure as demonstrated by the kerosene/heptane cleaning restoration effect, has dramatic impact on wettability also in toluene/kerosene cleaned cores.

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