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Reproducing wettability in sandstone reservoir core material in laboratory core restorations

Panagiotis Aslanidis *, Skule Strand, Ivan D. Pinerez Torrijos, Tina Puntervold

The National IOR Centre of Norway, University of Stavanger, 4036, Stavanger, Norway

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ABSTRACT

Replicating initial reservoir wettability conditions in core restoration by core cleaning and fluid restoration in the laboratory is of great importance. There are several core cleaning and restoration protocols used in the oil industry and academia that include the usage of different equipment, techniques, and materials. Strong solvents used in core cleaning can remove material that is part of the rock phase leading to more water–wet behavior. Large volumes of crude oil exposure in core restorations can result in high adsorption of polar organic components onto the mineral surfaces giving less water–wet behavior. Therefore, sufficient core cleaning should be targeted, involving no physical damage to the solid rock phase and effective crude oil exposure securing realistic water saturations, avoiding overexposure of crude oil.

The objective of this study was to establish an optimum core restoration process in terms of cleaning solvents and the amount of crude oil exposure, to re-establish the same core wettability from one core restoration to the next. Seven sandstone cores from a reservoir on the Norwegian Continental Shelf underwent a series of core restorations. Two different core cleaning procedures were used, in which mild (kerosene/heptane) and strong (toluene/methanol) solvents were involved, and furthermore, the cores were exposed to various volumes of crude oil.

Spontaneous imbibition experiments showed that mild core cleaning in combination with 5 pore volumes or more crude oil exposure rendered the cores less water-wet in successive core restorations. More rigorous cleaning with 5 pore volumes of crude oil exposure rendered the cores more water-wet in successive core restorations. From spontaneous imbibition results it was concluded that an optimum core restoration procedure involving mild cleaning and only 1 pore volume of crude oil exposure successfully reproduced core wettability in successive experiments.

1. Introduction

Predictions of reservoir performance are usually based on laboratory measurements of core properties (Jennings, 1957). Capillary forces during waterflooding are related to the wettability of rock surfaces, and reservoir wettability is recognized to be a critical issue in many types of oil recovery processes (Morrow et al., 1994). To have trustworthy results, it is important to mimic and reproduce the initial core wettability in laboratory experiments.

Laboratory restoration of preserved reservoir cores consists of two main operations, core cleaning and fluid restoration, both dependent on each other and affecting the final core wettability. There are various core cleaning strategies mentioned in the literature regarding hydrocarbons and brines extraction (Borre and Coffey, 2014; Conley and Burrows, 1956; Cuiec, 1975; Gant and Anderson, 1988; Grist et al., 1975; Guedez et al., 2020; Gupta et al., 2017; Jennings, 1957). Some solvents used for cleaning purposes are listed in Table 1 (Institute, 1998). In addition, several techniques and experimental assemblies have been suggested in the past decades, like cleaning by centrifuge (Conley and Burrows, 1956), distillation extraction method (Dean and Stark, 1920). Since there is a large variety of crude oil-brine-rock (COBR) systems, a different approach could be used in every case. Many solvents are not complete solvents for all types of oils and a clean extract may reflect oil solubility and not complete extraction (Institute, 1998).

Gant and Anderson (1988) did an extended investigation of the solubilization efficiency of several solvents (toluene, methanol, 2–butoxyethanol etc.) in a Dean–Stark apparatus on sandstone and dolomite cores after these had been contaminated with surfactants from

* Corresponding author.
E-mail address: panagaslanidis@gmail.com (P. Aslanidis).

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Table 1
Selected solvents and their use.

Solvents	Boiling Point, °C	Solubilizing
Acetone	56.5	oil, water, salt
Chloroform/methanol azeotrope (65/35)	53.5	oil, water, salt
Cyclohexane	81.4	oil
Ethylene Chloride	83.5	oil, limited water
Hexane	49.7–68.7	oil
Methanol	64.7	water, salt
Methylene chloride	40.1	oil, limited water
Naphtha	160.0	oil
Tetrachloroethylene	121.0	oil
Tetrahydrofuran	65.0	oil, water, salt
Toluene	110.6	oil
Trichloroethylene	87.0	oil, limited water
Xylene	138.0–144.4	oil

invert–oil–emulsion drilling muds. The results of this study showed that a 50/50 toluene/methanol mixture removed the surfactants slightly more efficiently than the three-step method involving three Dean–Stark extractions using different solvents every time. On the other hand, Dean–Stark extraction with toluene, 2–butoxyethanol, 2–methoxytetrahydrofuran and 1, 1, 1–trichloroethane gave unsatisfactory results. Guedez et al. (2020) investigated the effect of the huff-and-puff supercritical CO_2-based cleaning process on ultra–low permeable rocks. According to the outcome of that study, no precipitation of minerals and minimal dissolution of minerals occurred after mineralogy tests and scanning electron microscope (SEM) images. Jennings (1957) presented results of toluene extraction procedures on water–oil relative permeability. In that study it was observed that toluene significantly increased the permeability of the core material. In addition, it was stated that the analysed effluents showed removal of mostly paraffinic and aromatic hydrocarbons. Gupta et al. (2017) used high-pressure extractor and Soxhlet techniques to observe the effect of cleaning methods on petrophysical properties. It was concluded that the Soxhlet apparatus was more efficient due to the low solvent consumption compared to a high–pressure extractor. Furthermore, a higher cleaning efficiency of toluene, dichloromethane and chloroform was observed compared to that of n–heptane. Borre and Coffey (2014) used a new multi-step core cleaning procedure on carbonate core material that included two cycles of Soxhlet cleaning with pentane and methyl azeotrope with drying in-between. This process included an initial Soxhlet cleaning with pentane and drying at 60 °C. Subsequently, a second Soxhlet cleaning took place, but this time with methyl azeotrope for removing the remaining heavy oil and salts. Cuiec (1975) reported reproducible results in plugs that had been restored once, in spontaneous imbibition (SI) and forced displacement experiments using chloroform, ethanol, pyridine and hydrogen peroxide as cleaning agents as well as hydrochloric and acetic acids. He reported that the usage of toluene and chloroform regarding crude oil pollution was not recommended after observing that polluted samples could not be restored to initial conditions even after large amounts of solvents used. Grist et al. (1975) observed that toluene extraction did not affect the wettability as long as the process was not followed by methanol extraction and brine soaking. It was suggested that extraction with toluene/methanol or with chloroform/methanol especially for asphaltic crude oils was the optimum solution, since according to their remarks, methanol removes polar organic components and precipitated salts. From the core cleaning literature review, the main aim has been to bring the core back to water-wet conditions. During these operations strong solvents have been proposed which also affect bitumenic and salt precipitations that are part of the solid phase. These solid pore surface materials are not easily restored during the core and fluid restoration process.

The fluid restoration process, including establishing a correct water saturation with formation water (FW) and initial oil saturation by reservoir crude oil exposure, is an important part of the total wettability restoration process performed on cores in the laboratory. The importance of the polar organic components (POC) present in crude oils and their effect on surface wettability is well-known (Buckley and Morrow, 1990; Standnes and Austad, 2003). Previous experimental studies on outcrop sandstone cores have shown that polar organic bases present in crude oil instantly adsorbed onto the mineral surface at a larger extent than the polar organic acids, significantly affecting the core wettability (Mamonov et al., 2019). It was also observed that increased crude oil exposure further reduced the water wetness of the core. Similar observations have been confirmed on outcrop chalk, where increased crude oil exposure reduced the water wetness of chalk cores (Hopkins et al., 2016). However, in chalk it is the polar organic acids that have highest affinity for the calcite surfaces and that dictate the wettability (Puntervold et al., 2021).

In a recent core restoration study on chalk, it was observed that wettability in multiple restorations could be reproduced if the core was mildly cleaned with kerosene/heptane in front of a new fluid restoration (Piñerez et al., 2020), after initial water saturation (S_{wi}) of $S_{wi} = 0.1$ was established by the desiccator technique (Springer et al., 2003). The amount of crude oil exposure, in pore volumes (PV), needed to reproduce the wettability was limited to (1–S_{wi})PV (Piñerez et al., 2020). Larger volumes of crude oil gave significantly less water-wet cores.

The main objective of this study was to develop an optimum restoration process for reservoir sandstone cores to restore reproducible core wettability from one core restoration to the next. Both optimized solvent cleaning and crude oil exposure have been taken into account in a new, proposed cleaning and fluid restoration strategy for restoring the initial wettability of preserved reservoir sandstone core samples. In the present study, a Hassler core holder was used for flooding various solvents through the core in the core cleaning process. Flooding solvents through a core mounted in a Hassler core holder allows for easy control of the amounts of solvents used. Two distinctly different core cleaning solvent systems were applied involving mild (kerosene/heptane) and strong (toluene/methanol) solvents. The first solvent system aims to preserve wettability, while the latter consists of standard core cleaning solvents used by the industry to clean a core to completely water-wet conditions before restoring wettability. In every fluid restoration process $S_{wi} = 0.2$ was established by the desiccator technique (Springer et al., 2003), before the core was exposed to various PVs of crude oil (1 PV, 5PV or 11PV). After aging, the restored core wettability was evaluated by spontaneous imbibition oil recovery tests.

2. Experimental

2.1. Materials

2.1.1. Core material

Preserved reservoir sandstone cores were retrieved from two wells on the Varg field on the Norwegian Continental Shelf (NCS). The cores from the 9 S well were obtained from the upper sequence at depths less than 3479.50 m while the cores from the A5T2 well were taken from the lower sequence, at depths over 3493.75 m. Mineralogical composition at the respective depths was provided by the operating company and the mineralogical composition in wt% (weight per cent) representative of the cores used from each well is given in Table 2. The physical core properties are given in Table 3. Note that the permeability was measured at residual oil saturation (S_{or}) during the low salinity (LS) brine flooding in the core cleaning process (described later). Permeability varied between 2 and 22 mD in the A5T2 cores and between 5 and 17 mD in the 9 S cores. Pore size distributions of core plugs from the 9 S and A5T2 wells were determined by mercury injection at Stratum Reservoir, and the results are shown in Fig. 1.

2.1.2. Brines

The three brines used for the experiments are synthetic and were prepared in the laboratory; Varg formation water (FW) had total dissolved solids (TDS) or salinity of 201,600 ppm (mg/L), the low salinity

Table 2
Core mineralogy by XRD.

Minerals	Cores	
	P1–P2	P5–P10
Well	9 S	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
Plagioclase (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
Total (wt%)	100	100

brine (LS) consisted of 1000 ppm NaCl, and a fivefold diluted Varg FW (d_5FW) was used in the establishment of S_{wi} using the desiccator technique (described later). Brine compositions and properties are given in Table 4.

2.1.3. *Crude oil*

Stabilized stock tank oil from the Varg field, Oil V, was used in the oil recovery tests. The acid number (AN) and base number (BN) were analysed by potentiometric titration according to the procedures described by Fan and Buckley (2007), procedures that are modified from the standard methods ASTM664–89 and ASTM2896–88 for AN and BN measurements, respectively. Oil V density was measured at ambient conditions using an Anton Paar densitometer, and viscosity at 23 and 60 °C was measured by an Anton Paar MCR 302 rheometer. The Oil V properties are presented in Table 5.

2.1.4. *Other chemicals*

In the two core cleaning procedures, several chemical solvents purchased from Merck laboratories were used; low aromatic kerosene,

Table 4
Brine properties.

Ions	LS (mM)	FW (mM)	d_5FW (mM)
Na^+	17.1	2086.0	417.2
K^+	–	51.0	10.2
Ca^{2+}	–	536.0	107.2
Mg^{2+}	–	144.0	28.8
Cl^-	17.1	3526.0	705.2
Ba^{2+}	–	7.0	1.4
Sr^{2+}	–	8.0	1.6
Density (g/cm^3)	0.999	1.139	1.027
Bulk pH	5.7	5.9	5.8
Viscosity (cP)	0.95	1.45	1.01
TDS (mg/L)	1000	201,600	40,300

Table 5
Crude oil properties.

	Oil V
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^3)	0.845

Table 3
Core properties.

Core	P1	P2	P5	P6	P7	P9	P10
Well	9 S		A5T2				
Depth, m	3404.83	3404.88	3507.82	3507.87	3507.92	3528.82	3528.87
Cleaning	Kerosene/Heptane	Toluene/Methanol	Kerosene/Heptane				Toluene Methanol
Length (cm)	8.85	8.18	7.39	7.53	6.75	8.22	8.18
Diameter (cm)	3.80	3.80	3.8	3.8	3.8	3.80	3.80
Bulk volume (cm^3)	100.4	92.8	83.53	85.01	76.15	93.2	92.8
Dry weight (g)	203.52	188.47	168.18	171.82	152.2	179.65	167.62
PV (ml)	21.27	21.40	17.82	18.01	17.18	21.58	24.97
Porosity (%)	21.2	23.1	21.3	21.2	22.6	23.2	26.9
Permeability (md)	11.3	10.9	4.5	3.1	11.1	3.5	22.4

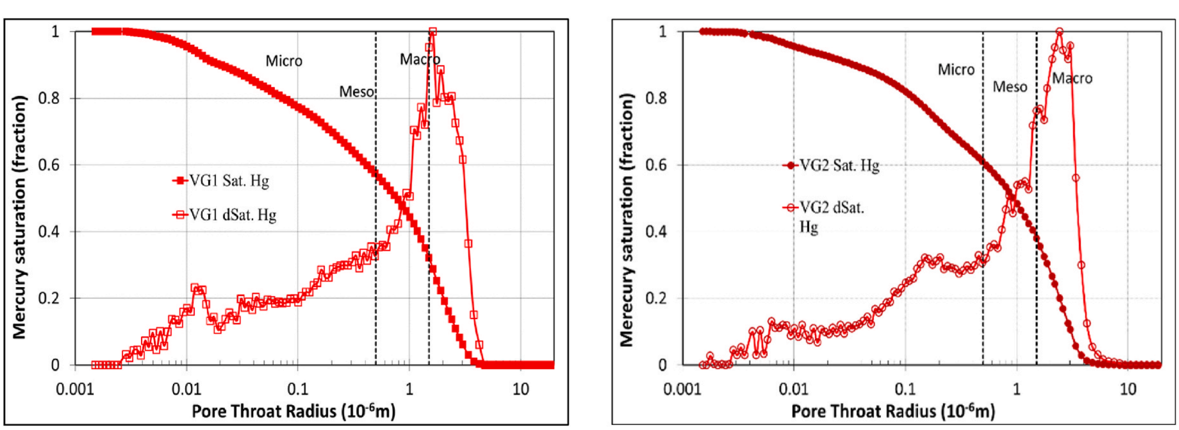

Fig. 1. Pore size distributions of the core plugs P1 (left) retrieved from the 9 S well and P9 (right) retrieved from the A5–T2 well.

n–heptane (hereafter referred to as heptane) both having reduced solubilization of large oil components, toluene, which is a strong solvent that efficiently removes heavy oil components and methanol, which can dissolve both polar oil components and water.

2.2. Core cleaning

In this study, two different approaches were used to clean the core material: (1) kerosene/heptane and (2) toluene/methanol. After cleaning with the various chemical solvents, 1000 ppm NaCl (LS) brine was injected to displace heptane or methanol while at the same time preventing any clay swelling. The cores were cleaned in a Hassler core holder at room temperature with a confining pressure of 20 bars. The solvents were injected at a flooding rate of 0.1 ml/min ΔP was monitored during each cleaning process to detect if any changes in core permeability had occurred between restorations. During the core cleaning process effluent samples were collected for visual inspection of the core's cleaned state (Institute, 1998). The core flooding setup for solvent cleaning can be seen in Fig. 2.

Cleaning with kerosene/heptane is an in-house procedure developed for maintaining the original wettability of the core by removing the mobile oil phase while minimizing desorption of adsorbed organic material at the rock surface (Shariatpanahi et al., 2012). Previous work on outcrop chalk has shown that approximately 75% of the adsorbed organic material remained adsorbed after the kerosene/heptane cleaning procedure (Hopkins et al., 2016). Cleaning by toluene/methanol, on the other hand, aims to clean the core to a very water–wet state by removing adsorbed organic material. Wettability is afterwards restored by crude oil exposure and subsequent aging.

2.2.1. Cleaning using kerosene/heptane

Low–aromatic kerosene is used to remove the mobile oil phase and non–polar oil constituents, whereas heptane displaces the kerosene fraction. LS brine is injected to displace the FW and any easily dissolvable salts and at the same time reduce the potential of clay swelling. The cores P1, P5, P6, P7 and P9 were initially flushed with approximately 10 PV of kerosene until a visually clear effluent was collected (Institute, 1998), followed by 5 PV of heptane and 10 PV of LS brine.

2.2.2. Cleaning using toluene/methanol

Toluene/methanol cleaning is a well-known 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, water and precipitated salts. The cores P2 and P10 were flooded with 5 PV of toluene followed by 5 PV of methanol, after which the cycle was repeated. Finally, the cleaning process was completed after injecting 10 PV of LS brine.

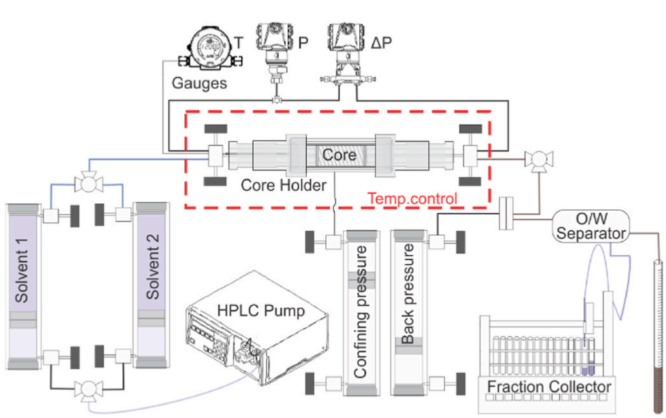

Fig. 2. Solvent cleaning setup.

2.3. Core fluid restoration

2.3.1. Establishing initial water saturation, S_{wi}

After the core cleaning procedure described above, the cores were dried at 90 °C, until constant weight, S_{wi} was established using the desiccator procedure described by (Springer et al., 2003). The dried cores were vacuum–saturated with fivefold diluted FW (d_5FW). A desiccator was used to gradually dry the cores to the predetermined weight corresponding to 20% S_{wi}. Finally, the cores were stored for 3 days to ensure even brine distribution inside the core. Note that the residual brine inside the core had attained its original FW composition after desiccation.

2.3.2. Crude oil exposure and aging

The Varg cores were exposed to various amounts of Oil V; 1 PV (more accurately (1–S_{wi})PV), 5 PV or 11 PV. The cores with $S_{wi} = 0.2$ were first shortly evacuated above water vapor pressure for less than 10 min in the Hassler core holder prior to oil exposure. The cores exposed to only 1 PV of Oil V were vacuum-saturated from both sides, and oil was injected at a rate of 0.5 ml/min to quickly fill the void space after evacuation. The cores exposed to 5 PV were vacuum-saturated with oil, and then flooded with 2 PV oil in both directions at a rate of 0.1 ml/min. The core exposed to 11 PV oil was first vacuum-saturated, before 5 PV oil was flooded in each direction at a rate of 0.1 ml/min. All oil floods were performed at 50 °C until the pressure reached 5 bar. To finish the oil exposure process, the cores were placed in aging cells surrounded by Oil V for 14 days at 60 °C. No pressure support was applied during aging, but a small increase in pressure inside the aging cell due to thermal expansion inside the aging cell is to be expected.

2.4. Oil recovery by spontaneous imbibition

A spontaneous imbibition (SI) oil recovery test is a method for evaluating the core wettability established after COBR interactions. The restored core is submerged in water or oil in an Amott cell (Amott, 1959). If the fluid that surrounds the core is the wetting phase of the system, then the surrounding fluid imbibes into the pores of the core and the mobile in-situ fluid is displaced. Fluid imbibition 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.

After aging, the core containing $S_{wi} = 0.20$ and Oil V was placed on top of marble balls inside the Amott imbibition cell and surrounded by the imbibition brine. Since the FW is already in chemical equilibrium with the COBR–system, it was used as imbibing fluid, securing no chemically–induced wettability alteration in the imbibition process. 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.

3. Results and discussion

The purpose of this study was to evaluate the wettability of reservoir sandstone cores restored using two different core cleaning procedures. Additionally, the aim was to find the optimum restoration process after which a reproducible wettability could be obtained. To achieve this, two distinctly different core cleaning methods were applied using kerosene/heptane and toluene/methanol. Furthermore, the effect of crude oil exposure in two differently cleaned sandstone rock systems was evaluated with respect to the core wettability generated.

When water wet-core material is exposed to crude oil, charged POC may adsorb onto oppositely charged mineral pore surfaces. Increased amount of crude oil exposure during a core restoration process, or in successive core restorations could increase the accumulation of these surface–active components, and result in a less water-wet behavior of the restored core, observable in spontaneous imbibition tests.

Strong solvents are used by the industry to achieve water-wet cores during core cleaning prior to fluid restoration. The solvents solubilize the residual oil together with POC attached to the mineral surfaces, but they also interact with the solid part of the rock surface, especially asphalts/bitumen and salt minerals. Mild core cleaning solvents will remove the residual oil but reduce the removal of POC, solid organics and salts that belong to the rock phase.

A combination of a mild but effective cleaning method, with sufficient crude oil exposure to fill the pores without creating a less water–wet surface is presented at this study. All cleaning processes described have been performed by core flooding through a Hassler core holder. No Soxhlet extraction has been used in any of the experiments.

3.1. Effect of crude oil exposure on core wettability

The effect of crude oil exposure on core wettability was investigated on reservoir cores after successive core restorations. Oil recovery by spontaneous imbibition (SI) was performed on the restored cores using FW as imbibing fluid. FW is already in equilibrium with the crude oil and rock surface and will therefore not generate any wettability alteration in the imbibition process. The spontaneous imbibition rate and ultimate recovery plateau will therefore indicate the core wettability (Anderson, 1986).

After a mild cleaning with kerosene/heptane and LS brine to remove easily dissolvable salts, fluid restoration was performed on the dried core P6 by establishing $S_{wi} = 0.2$ with FW using desiccator as described above. Then the core was exposed to 11 PV with Oil V before aging for 2 weeks at 60 °C. SI of FW at 60 °C was performed to evaluate the mobilization of oil by positive capillary forces after the first core restoration (R1). When oil production ceased, core cleaning, fluid restoration, aging and spontaneous imbibition were repeated in a second core restoration process (R2), and the results of both tests are presented in Fig. 3.

After the first restoration (R1) with 11 PV of Oil V exposure, core P6–R1 behaved quite water-wet reaching 21 %OOIP after 3 days and an ultimate recovery of 30 %OOIP after 22 days. However, after the second core restoration, core P6–R2 behaved significantly less water-wet. Only 11 %OOIP was produced after 3 days and reaching the ultimate recovery plateau of 21 %OOIP after 19 days. This represents a reduction of 30% in water-wetness compared to P6–R1. Core P6–R1 had been exposed to 11 PV of Oil V and produced 30 %OOIP, whereas after the second restoration the core P6–R2 had been exposed to a total of 22 PV of Oil V and produced only 21 %OOIP. This dramatic difference indicates that the injected volume of Oil V into core P6 affected the core wettability and needs to be considered when optimized core restoration procedures are designed for reservoir sandstone cores. The results also confirmed that mild core cleaning with kerosene/heptane was not efficient in removing POC attached to mineral surfaces, resulting in increased POC accumulation on pore surfaces and reduced water wetness of core P6 during successive core restorations. It should, however, be emphasized that the mild core cleaning procedure aims to preserve wettability by reduced removal of adsorbed POC from the rock surface. Mild core cleaning on outcrop chalk has shown that approximately 75% of the adsorbed POC remained adsorbed on the outcrop chalk surface after kerosene/heptane cleaning (Hopkins et al., 2017). The results on reservoir sandstone cores confirm trends observed previously in outcrop chalk (Piñerez et al., 2020), that increased crude oil exposure after mild core cleaning and restoration leads to a less water-wet core. Thus, the crude oil amount needs to be optimized for establishing a reproducible wettability.

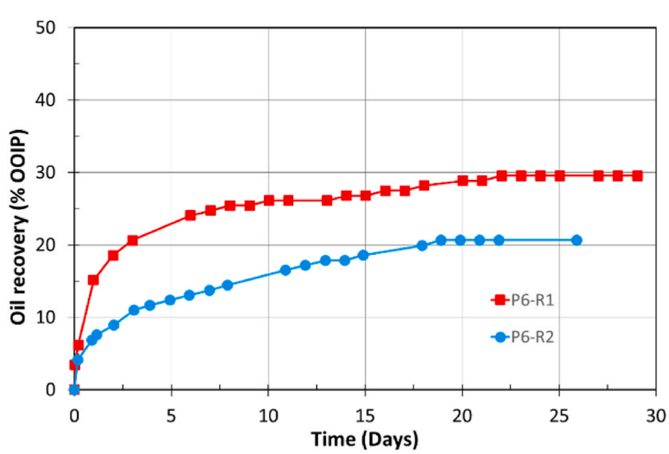

Fig. 3. Oil recovery by spontaneous imbibition at 60 °C from core P6, which was cleaned with kerosene/heptane and exposed to 11 PV of crude oil in both restorations R1 and R2.

3.2. Effect of kerosene/heptane cleaning solvents on core wettability

The main aim of using mild solvents kerosene and heptane in the restoration process, is preserving wettability. To examine the effect of mild core cleaning on core wettability in multiple restorations in further detail, the reservoir cores P9 and P1, originating from two different wells, were restored four times. In these experiments the amount of Oil V injected into the cores during each fluid restoration process was reduced to 5 PV. SI experiments were performed at 60 °C, using FW as the imbibing fluid, to evaluate core wettability after every cleaning and restoration process. As seen in Fig. 4, both cores P9 and P1 behaved quite water-wet after the first core restoration (R1).

Core P9 reached an ultimate recovery of 43 %OOIP after 30 days, while P1 reached an ultimate recovery of 34 %OOIP after 7 days. The quite water-wet behavior of cores P9 and P1 agree with that observed for core P6, which was restored with 11 PV of Oil V, Fig. 3. In the following restorations for core P9, Fig. 4 (left), the ultimate oil recoveries systematically declined, P9–R2 reached an ultimate recovery of 29 %OOIP, P9–R3 declined to 24 %OOIP, and P9–R4 only reached 17 %OOIP. Core P1 behaved similarly, Fig. 4 (right), with the oil recovery declining from 33 %OOIP in P1-R1 to 25 %OOIP in P1–R3, and to 14 %OOIP in P1–R4. The SI experiment after R2 failed and is therefore omitted.

The aim of the kerosene/heptane approach is to preserve the initial wettability of the cores during cleaning processes. The results on core P9 and P1 confirm this. The combination of mild core cleaning and only 5 PV of Oil V exposure during fluid restoration gave significantly less water-wet cores in successive core restorations and is thus not an optimal solution for reproducing core wettability in successive core experiments. 5 PV of Oil V exposure was too extensive, and increased accumulation of POC and reduced water wetness was observed. The question then asked was: could more rigorous cleaning solvents minimize changes in core wettability in successive core restorations?

3.3. Effect of toluene/methanol cleaning solvents on core wettability

Since the mild cleaning approach seemed to accumulate adsorbed POC and did not give reproducible results, a more rigorous method was selected. Toluene and methanol were used as cleaning agents in cores P10 and P2. The cores were restored three times each, and the amount of Oil V exposure remained the same at 5 PV in every fluid restoration. The SI results after each core restoration are shown in Fig. 5.

The ultimate oil recovery for P10–R1 was 40 %OOIP and increased to 43 %OOIP for P10–R2 and reached an ultimate recovery plateau of 45 % OOIP after P10–R3. The same behavior was also observed for P2. The ultimate oil recovery plateaus increased from 27 %OOIP to 33 %OOIP and 34 %OOIP in restorations R1, R2 and R3 respectively.

In the previous section it was seen that 5 PV Oil V exposure increased adsorption of POC and decreased water wetness after mild core cleaning.

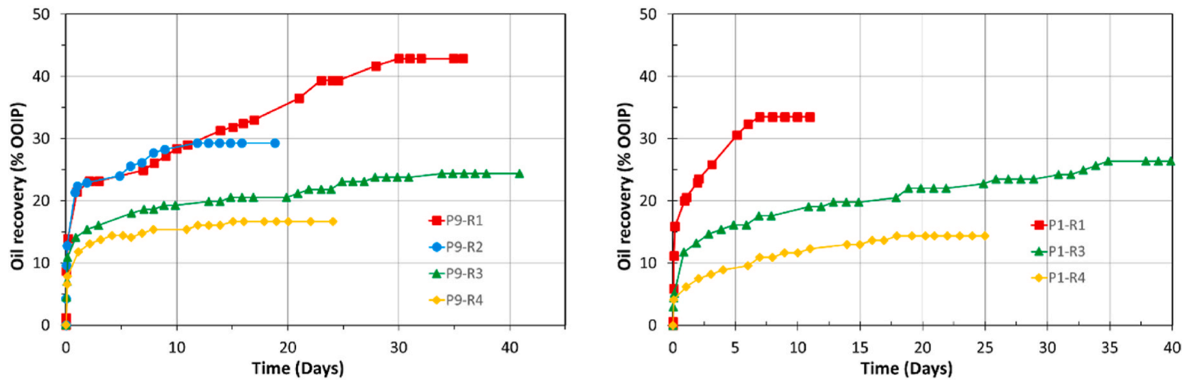

Fig. 4. Spontaneous imbibition of FW at 60 °C in the cores P9 (left) and P1 (right) cleaned with kerosene and heptane and exposed to 5 PV of crude oil.

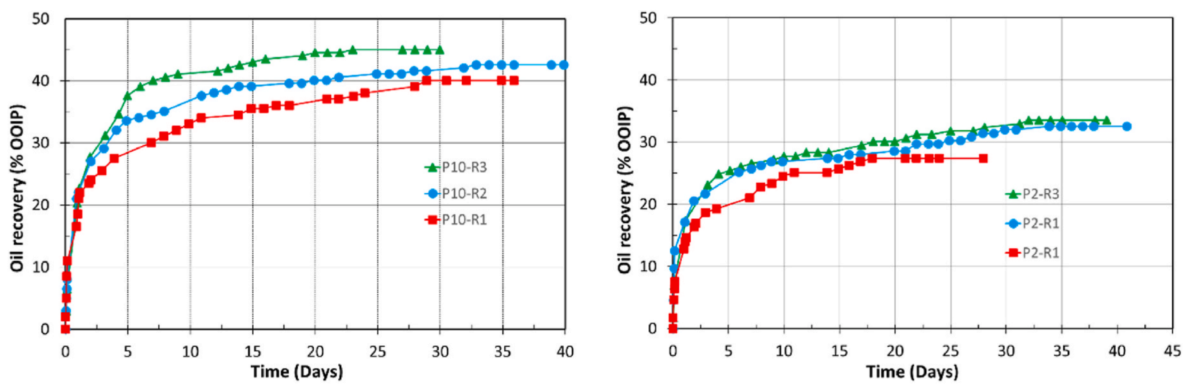

Fig. 5. Spontaneous imbibition of FW at 60 °C in the cores P10 (left) and P2 (right) cleaned with toluene and methanol and exposed to 5 PV of crude oil in every restoration.

Here the results show that the strong cleaning solvents used during fluid restorations promoted a gradual *increase* in the water wetness of the cores in successive core restorations. These results confirm efficient removal of adsorbed POC during core cleaning by toluene/methanol. Toluene/methanol potentially also removed components attached to the mineral surfaces controlling the total core wettability, such as bitumenic or salt precipitates that belong to the rock phase. These components cannot be easily and correctly restored in the cores during core restoration processes. However, these results can also indicate that the cores were not sufficiently cleaned before the first restoration, or that toluene/methanol is not able to remove all adsorbed POC from the reservoir sandstone surface.

3.4. Reproducing wettability in sandstone reservoir cores

The conclusions from the above attempts of reproducing the reservoir sandstone core wettability were that using mild solvents was not adequate to remove the POC adsorbed after 5 PV of Oil V exposure, but that a more rigorous approach removed more POC components after each restoration. Both methods proved to not be the best practice for restoring the core to its previous conditions. The challenge with core restoration after a rigorous approach with toluene/methanol is not knowing how many PVs of crude oil the core should be exposed to obtain a representative core wettability. As shown in Figs. 3 and 4, the extent of crude oil exposure affects the resulting wettability.

The better approach could be to maintain most of the POC controlling the initial core wettability in the preserved core, as done in the kerosene/heptane approach in Fig. 4, and afterwards limit the crude oil exposure in the fluid restoration process. This has been addressed in the final part of this work. The preserved reservoir cores P5 and P7 were cleaned by the mild cleaning solvents kerosene and heptane. Low aromatic kerosene solubilizes residual oil but not bitumenic solids. Heptane easily removes kerosene but will not dissolve salts as does methanol. After mild cleaning, core P5 and P7 were only exposed to 1 PV with Oil V, or (1-S_{wi})PV to be more precise, since S_{wi} was already established in the cores.

As seen from Fig. 6 (left), SI of core P7 reached ultimate oil recovery plateaus of 27 and 28 %OOIP in the first (R1) and second (R2) restorations respectively, at very similar imbibition rates, confirming a very close reproduction of core wettability.

In Fig. 6 (right), a successful reproduction of wettability was achieved after restorations R1 and R2 for core P5 with the exact same amount of oil produced, 29 %OOIP, and with similar imbibition rates. In restoration R3, the core P5 had been exposed to a total of 3 PV of Oil V, which might explain the observed decrease in the rate of imbibition. Nonetheless, the same ultimate oil recovery of 29 %OOIP was reached, indicating that no significant changes in the core wettability had taken place.

3.5. Evaluation of reservoir wettability

Smart Water or low salinity EOR are described as results of wettability alteration toward more water-wet conditions (Aghaeifar et al., 2015; Mamonov et al., 2019; Puntervold et al., 2021). To predict the EOR-potential by either method for a given reservoir, a reliable estimate of the reservoir wettability is needed. For strongly water-wet reservoirs the EOR-potential by wettability alteration will be very low and Smart Water or low salinity water injection are not recommended (Aghaeifar et al., 2015).

Reliable estimates of the reservoir wettability can be done through accurate core experiments in the laboratory. Improved laboratory procedures are needed, involving optimized cleaning procedures for

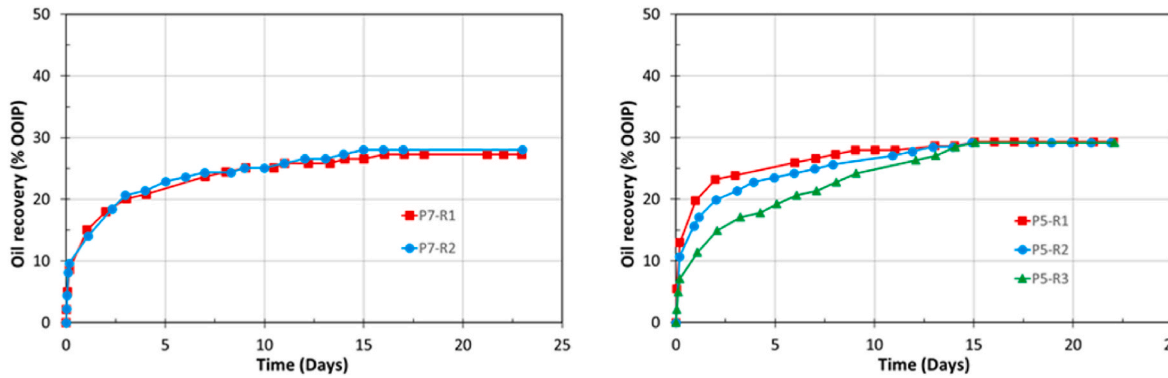

Fig. 6. Spontaneous imbibition at 60 °C of the cores P7 (left) exposed to and P5 (right) cleaned with kerosene and heptane and exposed to 1 PV of crude oil.

preserved reservoir cores, and reliable fluid restoration protocols in front of oil recovery tests, capillary pressure, and relative permeability measurements. This addresses the importance of this work.

In Fig. 7, the oil recovery results from the SI experiments performed after the first core restorations are compared. It can be observed that all cores clearly were on the water-wet side, giving ultimate recovery plateaus from 27 %OOIP to 45 %OOIP. Differences in the imbibition rates were also observed, with the various cores reaching recovery plateaus after 7–30 days of imbibition.

Even "sister'' cores drilled from the same seal peel behaved differently. This can be explained by effects of heterogeneity in the mineralogy and pore size distribution, supported by Fig. 1, in which the pore throat radii of the sandstone core material vary significantly and non-uniformly from approximately 50 nm to 10 μm.

As seen in this work, a less water–wet state could be expected when mild solvents (kerosene/heptane) are used in combination with a large volume of crude oil exposure during fluid restorations. When more rigorous solvents (toluene/methanol) are applied, slightly more water-wet cores are expected even at large oil volume exposure. All SI results are summarized in Table 6.

The overall summary of the spontaneous imbibition tests shows that all the restored reservoir cores were clearly on the water wet side, spontaneously imbibing water, and that the capillary forces mobilized and produced 27–45 %OOIP of the oil. These results confirm that capillary forces are important and need to be accounted for when ultimate recovery potential from reservoirs with heterogenous pore size distribution are to be evaluated in laboratory experiments, or in modelling and simulations of fluid flow in reservoirs.

The core wettability has a huge impact on the experimental results for capillary pressure, relative permeability, oil recovery, Smart Water and any other water-based chemical EOR methods. The large variation

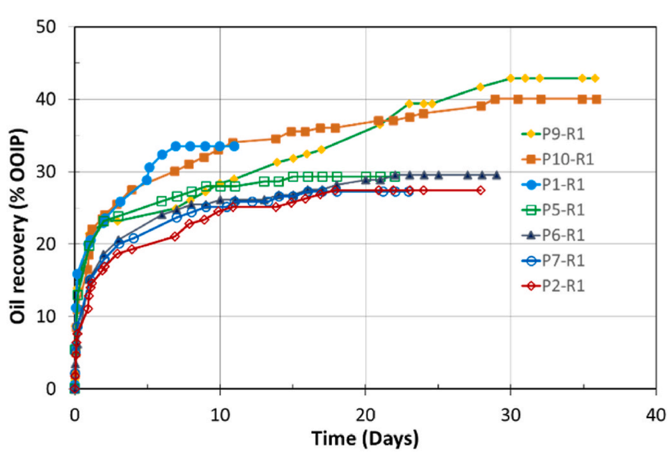

Fig. 7. Spontaneous imbibition results of all cores after the first restoration.

Table 6
Summary of all SI results from preserved reservoir cores.

Core	Solvent cleaning system	Oil V exposure in fluid restorations (PV)	SI (%OOIP)			
			R1	R2	R3	R4
P1	Kerosene/	5	34	–[a]	25	14
P9	Heptane	5	43	29	24	17
P5		1	29	29	29	–
P6		11	30	20	–	–
P7		1	27	28	–	–
P2	Toluene/	5	27	33	34	–
P10	Methanol	5	40	43	45	–

[a] Experiment failed.

in initial wettability results confirms that it will be very difficult to compare and discuss core analysis results from one core to the next. With improved understanding of parameters affecting core wettability during core cleaning and fluid restorations, laboratory procedures for reproducing the core wettability in successive core experiments on the same core could be developed. For this preserved reservoir core system, reproduction of core wettability in successive core experiments was successfully obtained by mild core cleaning with kerosene and heptane, followed by a minimum exposure of crude oil during fluid restoration.

4. Conclusions

The effect of core cleaning procedures and crude oil exposure in fluid restorations on the wettability of preserved reservoir sandstone cores retrieved from the NCS were examined in this study. Kerosene/heptane and toluene/methanol cleaning were used in the experimental procedures while the amount of crude oil exposure was 11 PV, 5 PV and 1 PV. The conclusions drawn from this work were the following:

1. Crude oil exposure of 5 PV and above in successive core restorations using the mild solvents kerosene and heptane, resulted in a decrease in oil recovery after each restoration process in cores P1, P6 and P9.
2. Cleaning with stronger solvents, toluene and methanol, showed an increase in oil recovery from cores P2 and P10 when these were exposed to 5 PV of crude oil during core restoration. This progressive increment in oil recovery might indicate that strong solvents can damage the rock surface by dissolving/removing material that contribute to the initial wettability of the rock.
3. Reservoir core wettability was successfully reproduced when cleaning with kerosene and heptane followed by 1 PV of crude oil exposure. A slight difference in imbibition rate was observed, although this difference did not have any impact on the ultimate oil recovery, which was similar in several repeated experiments.

Credit author statement

Panagiotis Aslanidis: Methodology, Experimental, Data Processing, Writing, Reviewing, Editing. **Ivan D. Pinerez Torrijos:** Experimental, Reviewing. **Skule Strand:** Conceptualization, Writing, Reviewing, Editing. **Tina Puntervold:** Conceptualization, Writing, Reviewing, Editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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