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University of Stavanger

Core restoration

A guide for improved wettability assessments

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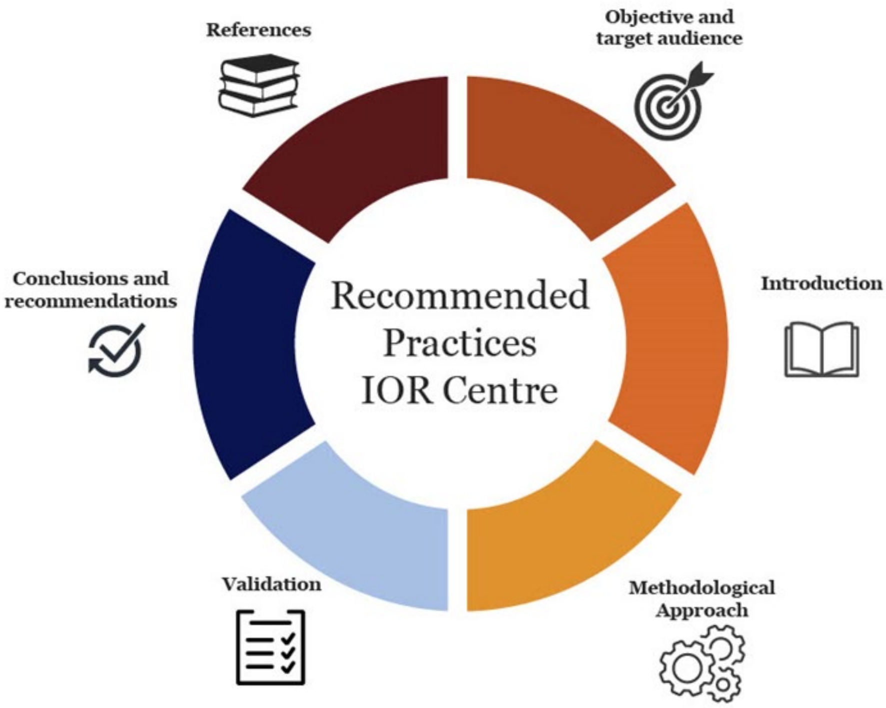

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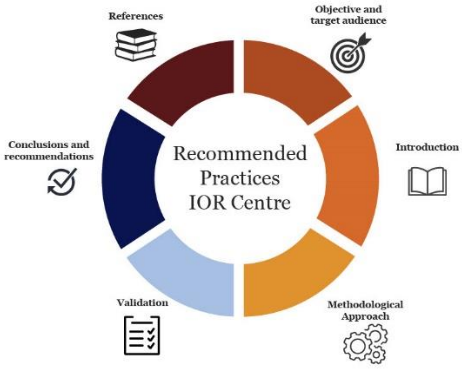
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Objectives and target audience

The initial wetting of a reservoir sets a limit for the EOR potential during water-based recovery operations and "Smart Water" injection. For this reason, an improved understanding of the factors influencing the wetting can help to control and better forecast oil production during water-based floods. To preserve and reproduce the original reservoir wettability is a challenging task and wrong cleaning and core restoration procedures can lead to incorrect wettability estimations and thus induce serious errors when evaluating the initial wettability of a reservoir system or its EOR potential by water-based methods.

Thereby, there is a need to improve the chemical knowledge on interactions among the rock, brine and fluids present in reservoir systems. This will help to understanding the influence of the parameters affecting wettability during cleaning and core restoration processes. Understanding which are the main parameters influencing oil recovery processes is of great relevance.

The objective of this document is to provide suggestions for added-value experiments, complementing and challenging the standard RCA and SCAL procedures, prior to performing experimental research in which wettability and wettability alteration processes are important. Lessons learned will be highlighted and new ideas to optimize core restoration protocols to preserve and closely reproduce wettability are put forward. These recommended practices target core restoration procedures after the core material has been received in the laboratory.

The target audience for this document is engineers and scientists with an interest in core preparation for wettability studies.

Introduction

Smart Water EOR effects observed in both carbonate and sandstone systems are wettability alteration processes toward more water-wet conditions, increasing the oil mobilization from heterogeneous pore networks by increased positive capillary forces. To perform reliable oil recovery studies in the laboratory, improved knowledge about factors affecting reservoir wettability is needed.

Core sampling and core analyses programs are important pieces of a complete formation evaluation campaign, providing valuable information for improved reservoir characterization. The data produced in the core analyses programs is used as reference data for calibration of reservoir models and for validation of other formation evaluation. Porosity and permeability properties are validated by RCA, but more complex properties like wettability are derived from SCAL data.

It is well known that both reservoir core sampling and laboratory core analyses are expensive operations, which can lead to few and possibly inadequate reservoir characterizing studies. For making the best field depletion strategy decisions, laboratory studies should be performed on representative reservoir core samples. This is particularly important for evaluating EOR-potential by water-based EOR-methods, such as Smart Water injection.

To avoid contaminating and compromising the cored material, the choice of drilling/coring fluid is of vital importance. Especially if the cored samples will be used for fluid flow analyses and wettability studies in core floods or SCAL. Since wettability influences both fluid location in the porous media and fluid flow properties, the most reservoir-relevant studies should be performed in core material, in which the original reservoir wettability has been preserved. The original core wettability can be altered in several ways, and invasion of drilling mud into the porous media is one way. Surface-active material, such as emulsifiers, are present in the drilling mud, and have ability to alter surface properties, such as wettability. Currently, the drilling fluid selection is based upon the requirements of drilling and logging operations, paying less attention to core analyses objectives (McPhee et al. 2015). Several factors can influence contamination by the drilling fluid, for example mud design, fluid chemistry, mud rheology, coring pressure, reservoir temperature and original wettability. All these parameters must be considered to minimize alteration of core and fluid properties if a program of RCA or SCAL will be run. There is evidence in literature both confirming and disconfirming invasion of oil-based-mud (OBM) and water-based-mud (WBM), thus it is dependent on both mud properties and reservoir conditions (Richardson et al. 1997, Ringen 2001).

After bringing the core samples to the surface, core preservation is an important task. The process should be rapid to retain the fluids inside the core. There are different methods of core preservation, with dry and wet storage being common. Dry storage consists of using a material to isolate the fluids inside the core material and avoiding air exposure. This is commonly done by using foil laminates and dip-coating or removable plastics. Wet storage, often used for short-term preservation, is done by sealing the cores in tin cans surrounded by mineral oil or drilling fluid. The recommended preservation standard for wettability studies is to combine dip and coating, as it is done with the film-foil-wax method (API 1998). This method consists of first wrapping the core in an impervious plastic film, then wrapping it in aluminum foil for extra protection, before ending with covering the core with a thin film of paraffinic wax. The wax helps retain the fluids in place, and it is recommended to not heat up the wax more than strictly necessary to reduce fluid evaporation (ESSO 1966, McPhee et al. 2015). **Figure 1** shows an example of several seal peels sealed by wax and two cores preserved with the film-foil-wax method.

(a) (b)

Figure 1. (a) Seal peels by wax dipping, (b) Preserved cores plugs with the film-foil-wax method

When the cores arrive at the laboratory for RCA and SCAL, core cleaning is the first step carried out in the core restoration process. Wettability is the main factor affecting fluid flow in porous media, and the laboratory core cleaning strategy needs to consider the importance preserving or restoring core wettability prior to SCAL (Treiber and Owens 1972, Gant and Anderson 1988, Piñerez et al. 2020).

After core cleaning the next step in the core restoration process is fluid restoration and generation of initial core wettability. The fluid restoration involves establishing initial water and oil saturations, crude oil exposure and aging. In each step, several experimental parameters and processes are involved, and it is important to have control of them, or at least to be aware of their effect on the wettability generated in the restoration process. Some of the more important parameters are brine and crude oil compositions, and presence of polar organic components (POC) such as acids or bases in the crude oil (quantified by the acid and base numbers, AN and BN). POC adsorption, quantity of oil injected into the core material during fluid restoration, and aging conditions are also of importance.

Methodological approach and validation

This document focuses on how preserved cores are handled and treated for optimizing the value of RCA and SCAL work performed in the laboratory. The different core restoration stages discussed are shown in the following diagram, **Figure 2**.

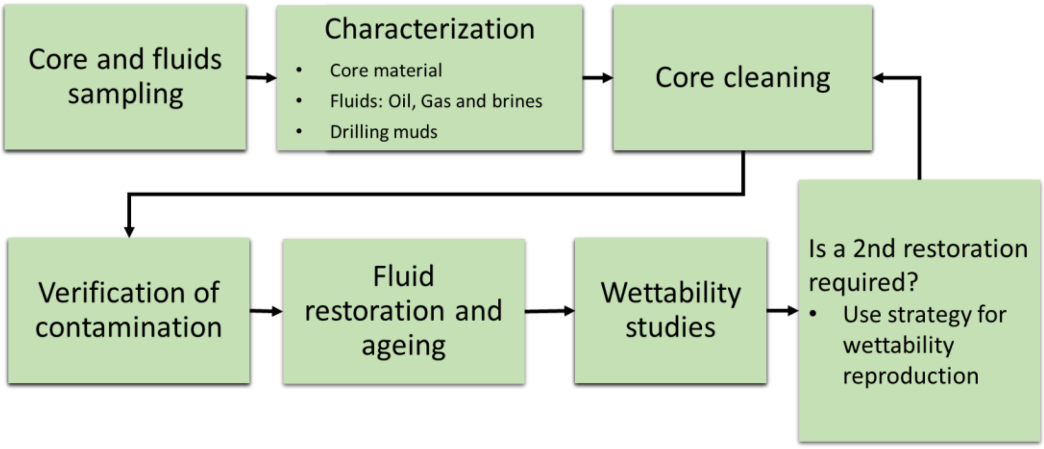


Figure 2. Optimized core restoration workflow.

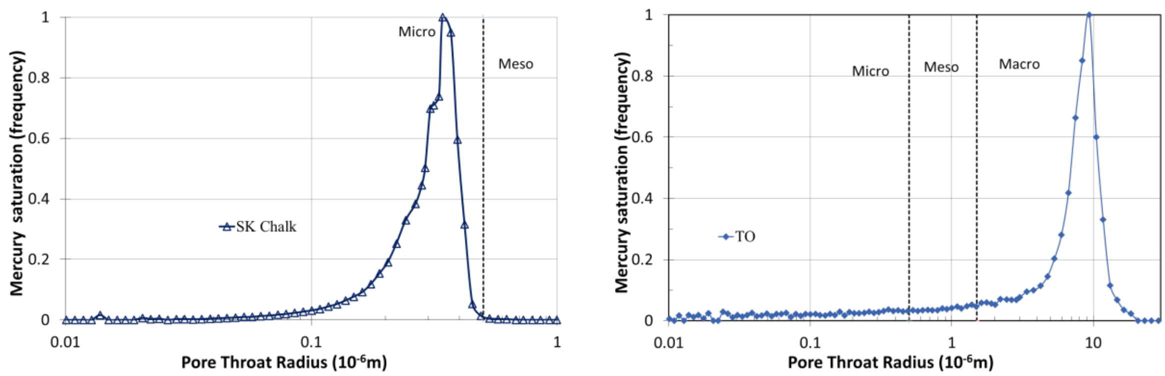
Laboratory core handling and characterization

Core analyses data are valuable for estimation of the in-place reservoir resources and the recovery potential. The quality of the data retrieved is dependent on the selection of representative core material for the individual reservoir section which are being evaluated. The compositions of the formation water and crude oil are also essential for establishing representative core conditions.

Core mineralogy and heterogeneities

Computed tomography (CT) scanning of the individual, preserved cores before core analyses can detect anomalies and help in the selection of representative core samples. Mercury injection capillary pressure (MICP) data give information about expected pore size distribution of the matrix in the selected reservoir section, providing important characterization data for fluid flow understanding.

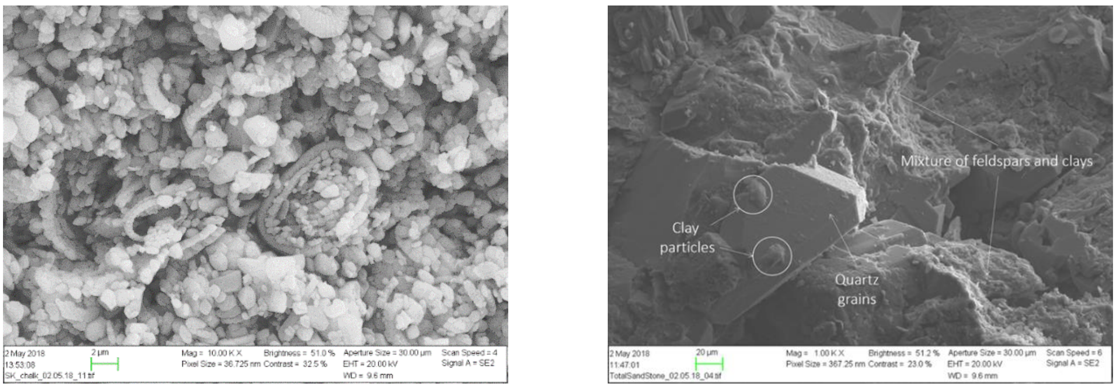
In **Figure 3**, MICP data from carbonate (left) and sandstone (right) rock material are shown. The pore size distribution of Stevns Klint chalk is shown in **Figure 3 (a)**. The pores range from ~0.01 to 0.7 µm. Most of the pores are in the micro pore region and close to 0.3-0.4 µm. The pore size distribution of a sandstone outcrop provided by Total E&P is shown in **Figure 3 (b)**. The pores range from ~0.01 to 100 µm, with the majority located in the macro pore region close to 10 µm.



(a) **(b)**

Figure 3. Pore size distribution of (a) Stevns Klint (SK) chalk and (b) Total outcrop (TO) sandstone determined from mercury injection capillary pressure measurements.

Differences in mineral composition and distribution are important, especially in SCAL wettability studies. Possessing carbonate or sandstone main mineralogy characteristic will define how the wettability and wettability alteration processes take place, since their chemical reactivity is distinctly different. Thus, an adequate mineralogical characterization is important. Analyses such as X-ray powder diffraction (XRD), elementary composition by dissolution, specific surface area by Brunauer-Emmett-Teller (BET), cation exchange capacity (CEC) and scanning electron microscope (SEM) analyses combined with energy dispersive spectroscopy (EDS) are useful for gaining knowledge about elemental composition of the rock. SEM-images of carbonate (left) and sandstone (right) are shown in **Figure 4**.



(a) **(b)**

Figure 4. SEM-images of (a) Stevns Klint outcrop chalk at magnification 10000 X and (b) Total outcrop sandstone at magnification 1000 X. In the latter silicates such as clay, quartz, and feldspars were specifically identified.

Additional mineralogical characterization methods to understand fluid rock interactions during waterflooding, are available in the report “Toolbox for mineralogical research for EOR experiments”.

Drilling fluid contamination

Early detection of possible mud contaminants in the core material is highly important. Fjelde et al. (2015) performed a study that aimed to detect mud invasion and remove mud components from sandstone material. It was pointed out that emulsifiers from OBM invasion can be detected by measuring interfacial tension (IFT) between the effluent and water during solvent cleaning of core plugs. Contamination of sandstone core plugs with mud filtrate from OBM containing asphalt products, was found to alter the wettability to less water-wet conditions, **Figure 5.** Later, contamination of core plugs with mud filtrate from OBM containing no asphalt products was found to give more water-wet conditions, **Figure 6.** The measured capillary desaturation curve (CDC) for a sandstone reservoir rock with potential OBM contamination was found to depend on the cleaning procedure (Fjelde et al., 2015). The authors obtained a typical CDC after methanol cleaning, with typical CDC values representative of water-wet rock after strong cleaning with methanol/toluene, acetic acid, and ethanol. However, the strong cleaning might have altered the rock composition by dissolution of carbonate minerals by acetic acid.

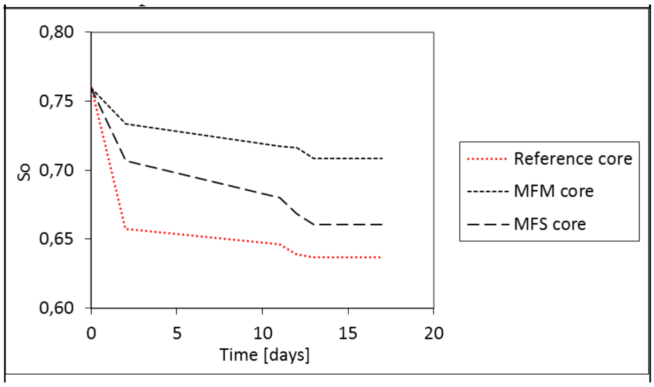


Figure 5. Change in oil saturation (S_o) by spontaneous imbibition of formation water in two core plugs exposed to OBM filtrate containing asphalt product and one core plug not exposed (reference core) (Fueled et al., 2015). (MFM and MFS: mud filtrates with mineral and synthetic base oils, respectively).

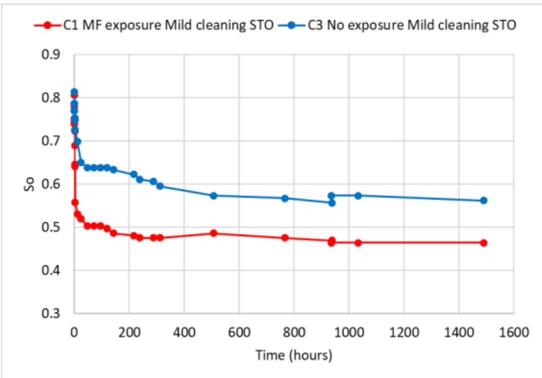


Figure 6. Change in oil saturation (S_o) by spontaneous imbibition of formation water at 60°C in a core plug exposed to OBM filtrate without asphalt product (C1) and a core plug not exposed (C3). Both core plugs were cleaned by injection of STO at S_{wi} (Fjelde 2021).

When working with OBM, it is often very useful to run mud invasion tests by obtaining a mud filtrate and subsequently injecting it into the core samples. This is an equivalent to a “worst case scenario” where the mud filtrate gets in contact with a large fraction of the porous media. Then a comparison of the core’s wettability by spontaneous imbibition at room temperature with heptane as model oil and formation water (FW) as the imbibing fluid is performed. If there are significant changes in the spontaneous imbibition processes, it can be a strong indication of wettability alteration by mud contamination, and through the Amott water index these changes can be quantified. Additionally, the mud filtrate can be sampled and analyzed for AN and BN. This is done to detect the presence of surface-active polar components that can alter the core wettability.

Invasion of water-based mud (WBM) can be detected by analyzing the ionic composition of the effluent during cleaning of core plugs, looking for potassium in KCl-based WBM, by ionic chromatography (Fjelde et al. (2015). High concentration of potassium in invaded mud filtrate can reduce the adsorption of divalent cations onto clay surfaces and thereby alter the wettability to more water-wet conditions. Mud particles and polymers were detected by SEM-analysis, also after waterflooding, **Figure 7**, which show that it can be difficult to remove mud particles from reservoir core plugs after mud invasion.

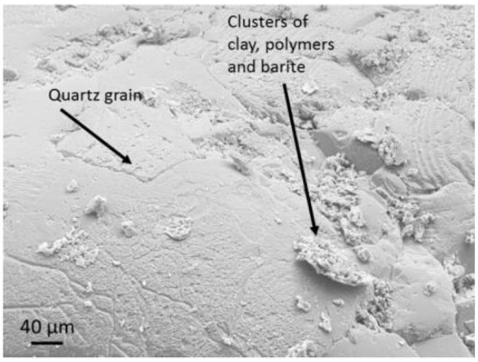


Figure 7. Detection of mud additives by SEM and EDS on a core slice (Fjelde et al., 2015).

Polymer flooding experiments were carried out in sandstone core plugs contaminated by OBM filtrate containing no asphalt products and in core plugs without contamination as reference. S_{wi} was first established in all core plugs before aging with STO was carried out. Mud filtrate, prepared by filtration through a 3µm filter, was injected into two core plugs. One contaminated core plug was then cleaned by injecting STO (mild cleaning). The other contaminated core plug was first cleaned by toluene/methanol cycles (strong cleaning) before S_{wi} was reestablished and aging with STO was carried out. Two reference core plugs without mud filtrate exposure were prepared with mild and strong cleaning. When the polymer floods were carried out at 60°C, the reference core plugs showed similar differential pressure (dP) profiles, **Figure 8**, independent of the cleaning method.

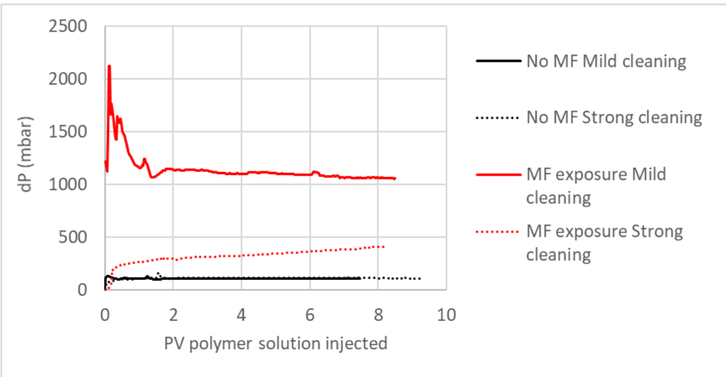


Figure 8. Differential pressure (dP) during polymer flooding experiments using core plugs with and without mud filtrate contamination. The core plugs were prepared by either mild cleaning (STO injection) or strong cleaning (toluene/methanol cycles).

The exposed core plug with mild cleaning, gave very high dP from the beginning of the polymer flood. For the exposed core plug with strong cleaning, a gradual increase in dP was observed during the polymer flood. The exposed core plug gave higher residual oil saturation than the reference core plugs without mud filtrate exposure. The permeability of formation water after the polymer floods was for the contaminated core plugs only 10% of the permeability of the non-contaminated core plugs. The results showed that neither mild nor strong cleaning were able to remove the formation damage caused by mud filtrate invasion. It was concluded that using mud contaminated core plugs can give incorrect and a too low estimate of the polymer flooding potential.

The main conclusions of this work were that mud components, which may affect wettability and permeability and therefore fluid flow properties, should be removed by core cleaning. If this is not achieved, the core plugs should not be used for wettability, SCAL and EOR studies. If contaminated reservoir core plugs must be used, the core plugs with the lowest contamination should be used for the most important experiments. The degree of mud invasion can be determined by analyzing core plugs for the volume of mud filtrate in the core plugs. The mud filtrate volume in core plugs can be estimated from mud component effluent profiles during cleaning, e.g., potassium ions in WBM and base oil in OBM. Furthermore, important minerals should not be removed from the original reservoir rock during cleaning. It is recommended to evaluate the possibility of mud contamination effects, and appropriate characterization tools for mud contamination should be a combination of analytical and characterization techniques like IFT, ion chromatography/ICP, and SEM. A mud invasion test can give information about the impact of wettability of a contaminated core when compared to a non-contaminated reference core, providing a context of contamination that can be quantified by spontaneous imbibition as described by Bobek et al. (1958).

Core restoration

Reservoir wettability influences several fluid flow parameters, like capillary pressure and relative permeabilities of oil and water. Core restoration procedures are fundamental for obtaining reliable oil recovery forecasts. Thus, the cleaning methods should adapt to obtaining reliable wettability estimations.

Core plug cleaning

Typical experimental set-ups for core plug cleaning are Soxhlet extraction or solvent flushing by direct pressure in a core holder with one or several solvents (API 1998). Typical solvents used are acetone, chloroform, cyclohexane, hexane, methanol, tetrahydrofuran, toluene, and other aliphatic hydrocarbons or combinations thereof depending on the fluids and core conditions (API 1998, McPhee et al. 2015). Sequential use of different organic solvents for core cleaning has been reported to be more efficient than single solvent cleaning (Cuiec 1975). Nevertheless, the use of multiple solvent combination can lead to different effects on the restored core wettability (Shariatpanahi et al. 2012). For this reason, the selection of efficient cleaning solvents is of high importance, especially when preserved core material must be restored for wettability studies.

The literature shows that in many cases mud invasion is expected by default due to lack of a structured coring plan that aims to fulfill the SCAL study requirements during core sampling (Bobek et al. 1958, McPhee et al. 2015). To be on the safe side the preserved core plugs are often cleaned to a complete water-wet state. Thus, in this regard many of the classical core cleaning methods aim to remove mud filtrates (Wendell et al. 1987, Gant and Anderson 1988, Fjelde et al. 2015, Fjelde 2017), precipitated salts and native crude oil to achieve a water-wet state. However, nowadays several tools for detecting mud invasion exist, among them tracers or doped water are typical alternatives, but their use is not widespread.

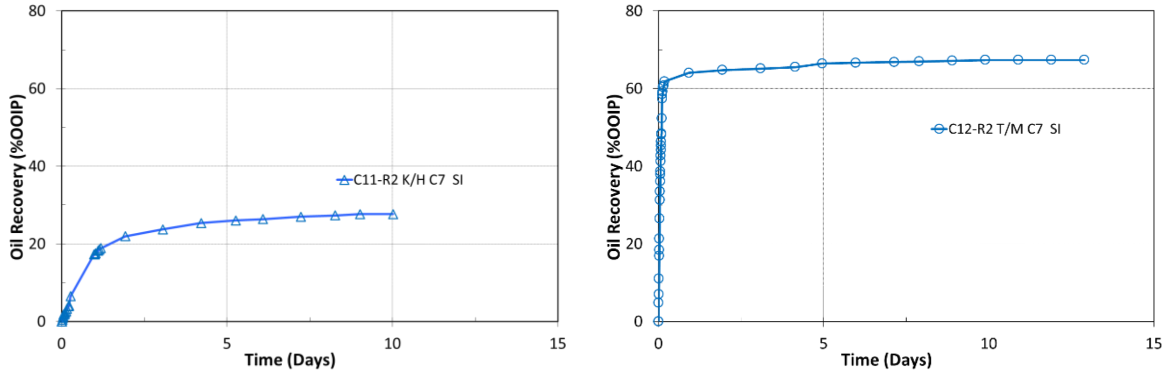
With this classic approach, which consists of rendering the core plugs completely water-wet, some of the initially adsorbed polar organic components (POC) present in the original oil that invaded the reservoir will be removed by strong organic solvents. In consequence, the original reservoir wettability has been destroyed. In addition, salts and bitumen that form part of the solid reservoir phase can be altered or removed during strong cleaning. Then, to restore the core plug wettability in the laboratory becomes challenging. The main reason is that the sampled crude oil used in core plug restoration will differ in chemical composition from the crude oil that created the initial reservoir wettability (Puntervold 2008). The chemical composition may have changed during geological time due to chemical adsorption, desorption, and decomposition processes (Shimoyama and Johns 1971, Shimoyama and Johns 1972). Furthermore, the use of classic solvents also represents a technical challenge for the core properties. Jennings (1957) showed results of the effect of toluene cleaning on water and oil relative permeability. He pointed out that toluene significantly increased the permeability of the core material. Grist et al. (1975) showed 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 and methanol or chloroform and methanol was highly efficient, especially for asphaltic crudes, as methanol removes water, polar organic components, and precipitated salts.

Mild cleaning vs harsh cleaning

In a recent study on outcrop chalk, it was observed that wettability can be reproduced if the core cleaning strategy consisted of mild solvents, with low aromatic kerosene for displacing the mobile crude oil and n-heptane for displacing the resident kerosene (Piñerez et al. 2020). The use of kerosene-n-heptane aims to preserve the initial wettability during cleaning process, as opposed to the harsh cleaning method that aims to clean the core to a complete water-wet state.

The two solvent cleaning methods were compared: toluene-methanol-distilled water flooding (harsh cleaning) and kerosene-heptane-distilled water flooding (mild cleaning). It was observed that capillary forces increased after both cleaning schemes. However, the harsh cleaning method with toluene-methanol resulted in higher oil recoveries than the mild cleaning, confirming that the cores became more water-wet after harsh cleaning. Hence, the toluene and methanol flooding sequence, as expected, had greater solvation effect on the adsorbed POC on the chalk mineral surfaces.

The mild core cleaning process was carried out with flooding reduced volumes of kerosene and heptane through the core to preserve adsorbed POC, wetting the mineral surfaces. In the subsequent core restoration process, less POC and thus a lower amount of crude oil is needed for reproducing the initial core wettability. Using this core restoration technique can help to obtain more representative reservoir core wettability than that obtained using harsh solvents, since removal of the adsorbed POC that created the initial wettability is minimized. Spontaneous imbibition test results by Piñerez et al. (2020) after mild and harsh cleaning of outcrop chalk using heptane as the oil phase are shown in **Figure 9.** The results showed very distinct results using one cleaning scheme over the other. As expected, toluene-methanol cleaning removed more POC from pore surfaces bringing the core to a more water-wet state, while kerosene-heptane cleaning left the core in a less water-wet state.



(a) (b)

Figure 9. SI at 23 °C on the solvent cleaned cores, saturated 100% with heptane, DI was the imbibing fluid. (a) Heptane recovery (%OOIP) from the kerosene-heptane cleaned core C11. (b) Heptane recovery (%OOIP) from the toluene-methanol cleaned core C12.

In a similar study using Varg reservoir sandstone cores by Aslanidis et al. (2022) the same harsh (toluene-methanol-1000 ppm NaCl) and mild (kerosene-heptane-1000 ppm NaCl) cleaning processes were compared. Toluene and methanol were tested as cleaning agents in the cores P10 and P2. The 1000 ppm NaCl brine is used to prevent clay swelling. The cores underwent 3 restoration processes and the amount of crude oil used during saturation was 5 PV in each restoration process. The SI results after each core restoration are shown in **Figure 10**.

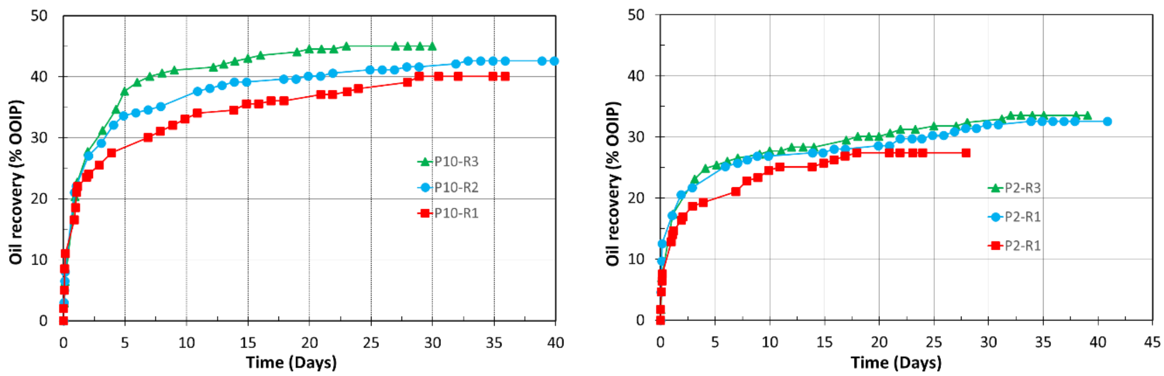
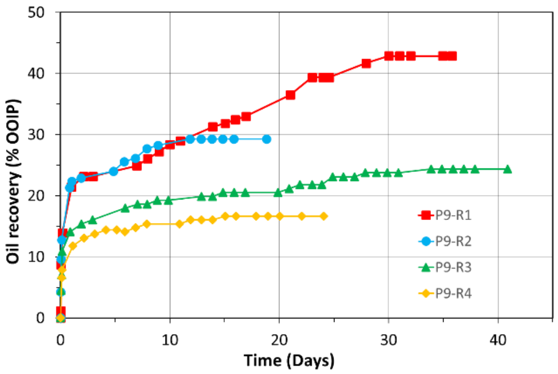


Figure 10. Spontaneous imbibition of FW at 60°C of the cores P10 (left) and P2 (right) cleaned with toluene and methanol, S_{wi}=0.2, and exposed to 5 PV of crude oil in every restoration and aged at 60°C for 14 days.

A gradual increase in the water wetness was observed as the successive core restorations took place, confirming the efficient removal of the previously adsorbed POC by using toluene and methanol. However, restore original reservoir wettability by reestablishing the adsorption of original POC and salt precipitates is challenging after cleaning the core to a very water-wet state. An important observation was that even though the cores were flooded until a clear effluent, the cores seemed not sufficiently cleaned before the first restoration, meaning that toluene-methanol was not able to remove all adsorbed POC from the reservoir sandstone surface. This can be seen by the increasing water wetness after multiple cleaning cycles.

The effect of mild cleaning on core wettability was studied on reservoir cores P9 and P1. The cores were restored four times and flooded with 5 PV in every restoration. Spontaneous imbibition experiments were performed at 60°C with formation water (FW) as the imbibing fluid. The results in **Figure 11** show a reduction in water-wetness as the successive restorations were carried out, confirming that POC were retained on the surfaces and wettability partly maintained during the mild cleaning process**.**



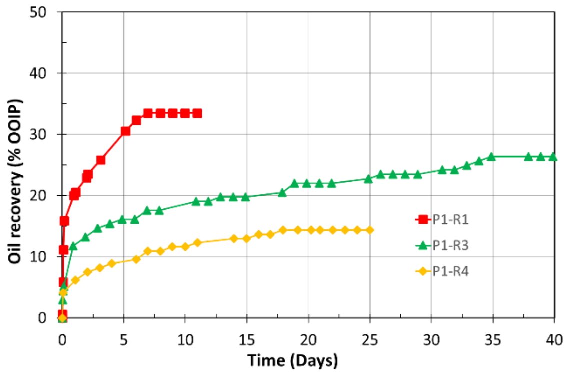


Figure 11. Spontaneous imbibition of FW at 60°C of the cores P9 (left) and P1 (right) cleaned with kerosene and heptane, S_{wi}=0.2, and exposed to 5 PV of crude oil in every restoration and aged at 60°C for 14 days.

Aqueous phase cleaning

Gypsum or anhydrite in carbonate rock can have a significant effect on initial wettability and if present the core must be handled correctly. In a study on outcrop Stevns Klint chalk by Puntervold et al. (2007a) it was found that the sulfate salts played significant role in the restoration process and resulting core wettability, with staggering differences if the cores were cleaned with distilled water to remove the dissolvable salts. Ultimate recovery of 60 %OOIP was achieved for the non-cleaned core after 3 days and for the cleaned core the ultimate oil recovery of 26 %OOIP was reached after 12 days, **Figure 12.** Clearly the presence of anhydrite resulted in a more water-wet chalk core by preventing adsorption of POC.

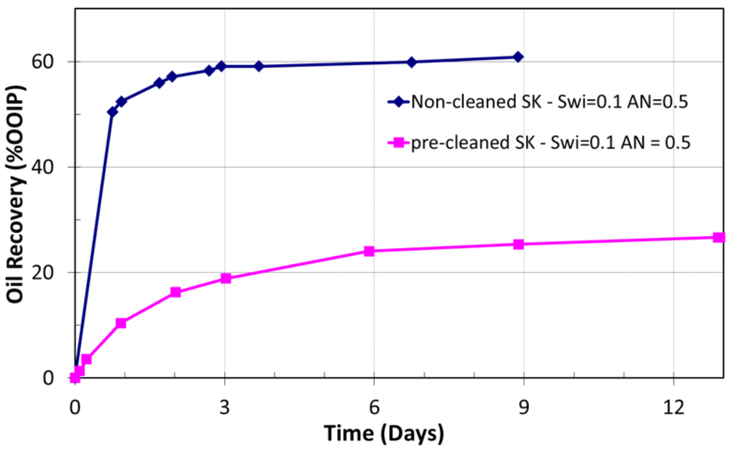


Figure 12. Effect of cleaning outcrop chalk on oil recovery by spontaneous imbibition. One Stevns Klint chalk core was precleaned with distilled water and one Stevns Klint core was used without precleaning. Both cores contained S_{wi} =10% FW without sulfate, both were exposed to 5PV of crude oil with AN = 0.5 mgKOH/g. Spontaneous imbibition was performed using the same FW at 90 °C (Puntervold et al. 2007a).

The effect shown by Puntervold et al. (2007) in the case of outcrop chalk restoration for wettability studies indicates that cores, also outcrops, should be properly cleaned before performing parametric studies. Since the quarry, from which the Stevns Klint chalk was obtained, is located close to the sea, seawater influx and subsequent drying has influenced the content of anhydrite in the outcrop chalk material. However, in reservoir cores gypsum/anhydrite minerals are part of the formation, therefore it is advisable to detect their presence during the cleaning process but not to remove them, since they influence the wetting state of the rock (Gant and Anderson 1988, Austad et al. 2015, Pinerez Torrijos et al. 2017). Sulfate bearing salts in carbonate formations could contribute to water-wet areas of the rock surface, hence limited brine or distilled water flooding is recommended during core restoration, and the temperature should be low to not change the composition of gypsum (Gant and Anderson 1988). The effect of these minerals on wettability is stronger in carbonates because sulfate and calcium are wettability alteration ions. In sandstones the calcium eluted from the cleaning process should be measured since it is important for wettability studies. The technique recommended to detect these salts is ion chromatography, since the detection limit by standard X-ray power diffraction will not be sufficient for detecting its presence at low concentrations, <1 wt% (Lopez-Salinas et al. 2011, Austad et al. 2015).

Fluid saturation and aging

Consistent methods for fluid saturation are a main requirement in wettability, SCAL, and EOR studies. Initial water and oil saturation can be achieved in different ways, but the results can end up in highly scattered values, and such deviations can lead to major errors in the entire production forecast program. In the following section convenient procedures are discussed.

Formation water

To establish S_{wi}, equilibrated formation water (FW) should be used. If the brine is synthetic the composition should match the original FW composition, this means that no simplification in the composition should be carried out, since all ions present can influence the wettability of the system (Anderson 1986, Buckley 1994, Buckley and Liu 1998, Buckley et al. 1998, Buckley and Fan 2007). FW salinity can vary widely, from less than 20 000 ppm to more than 250 000 ppm. Sodium and chloride are typically the main ions as well as divalent cations like calcium and magnesium, and the concentration of Ca^{2+} is usually higher than that of Mg^{2+} in FW.

Initial water saturation

If a SCAL program is being run, reproducibility of experiments and conditions is of utmost importance. Therefore, the right steps and methodologies should be taken to ensure comparability of the tests performed. Initial water saturation is established in the classical way of saturating the cores with formation water before desaturation until reaching S_{wi}. Desaturation can be performed in several ways: by oil displacement, by capillary desaturation with the porous plate or by desiccation. Both the porous plate technique and the desiccator technique provide a highly uniform distribution of ions within the porous media. It is seen in the literature that S_{wi} established by oil injection or porous plate can result in high variation of results (Jadhunandan and Morrow 1995). Thus, desiccator or porous plate desaturation is preferred over dynamic oil displacement to achieve the initial water saturation. In a study done by the Smart Water group at University of Stavanger the effect on spontaneous imbibition of core plugs restored with porous plate and the desiccator technique were compared, **Figure 13**.

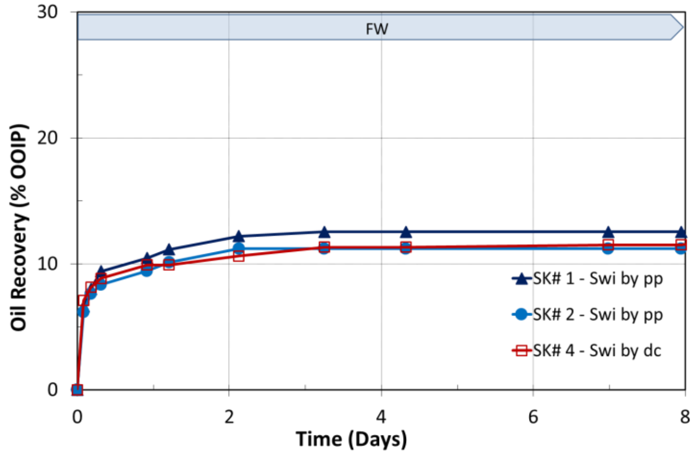
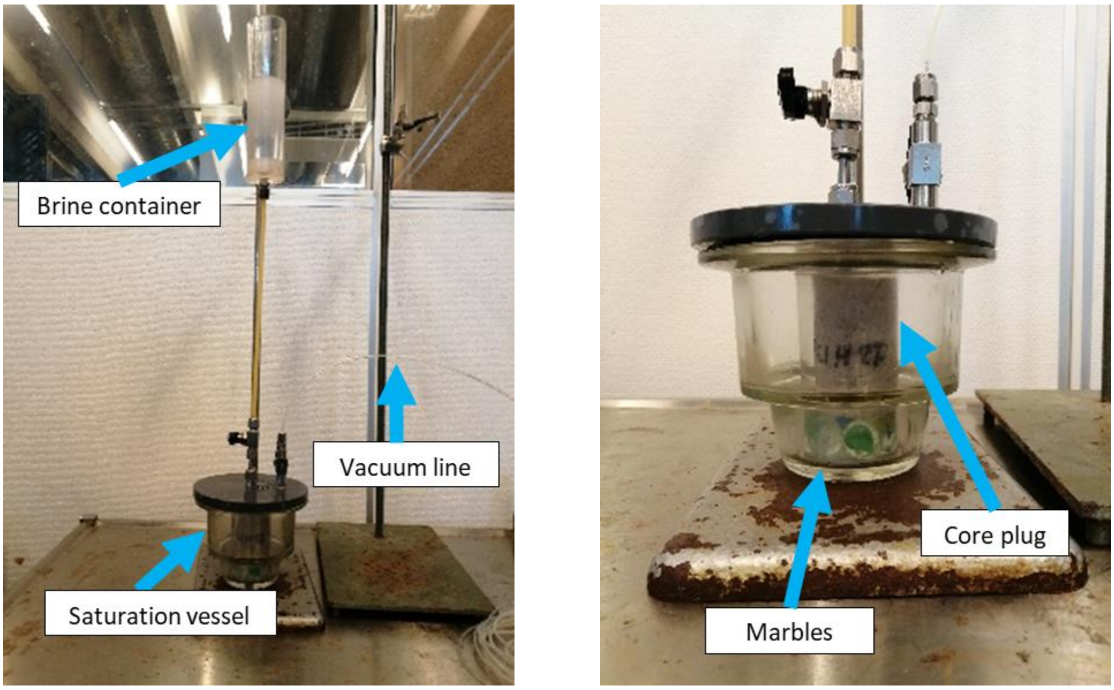


Figure 13. Comparison of porous plate (pp) vs desiccator technique (dc).

The results showed that similar S_{wi}-values were established, which also resulted in similar core wettabilities (Fathi et al. 2010, RezaeiDoust et al. 2011, Puntervold et al. 2015). However, the desiccator technique is more time efficient and the water saturation between different cores is reproducible and highly uniform (Springer et al. 2003). When using the desiccator, a first stage of saturation under vacuum to assure that complete fluid invasion is performed. Ideally, the core should be surrounded by the fluid, for that reason spheres of an inert material can be used to support the core in the saturation vessel, this is shown in the photos included in **Figure 14**. The brine used is a diluted formation water, and the dilution factor is calculated relative to the desired initial water saturation.



(a) (b)

Figure 14. Brine saturation prior to using the desiccator. (a) Saturation set with vacuum. (b) Core plug being saturated with brine.

After saturation with the diluted brine, the core is put into a desiccator chamber, and at this step the water molecules are evaporated until reaching the target weight to establish initial water saturation. Then, the core is left for equilibration for at least three days to assure an even concentration of ions across the core plug, **Figure 15**. By the end of this process the predetermined S_{wi} has been reached, and the composition of the remaining water has attained similar composition as the original (undiluted) formation water.

(a) (b) (c)

Figure 15. Various photos of a desiccator. (a) Desiccator showing the absorptive material, in this case silica gel. (b) Desiccator with supportive plate covering the absorptive material. (c) Core plug being dried in the desiccator.

Oil saturation

The oil saturation method and conditions have a direct impact on wettability. Crude oil exposure will increase surface active components on the surfaces affecting wettability. Recent studies on both carbonates and sandstones have shown that POC adsorption takes place as soon as the flooded crude oil encounters the porous media. Furthermore, if crude oil volume is increased a reduction in the water-wetness degree can be observed (Fulcher et al. 1985, Abeysinghe et al. 2012, Guo et al. 2015, Hopkins et al. 2016a, Mjos et al. 2018, Klewiah et al. 2019, Mamonov et al. 2019).

Crude oil

The use of non-contaminated live oil at reservoir conditions is often recommended. However, if live conditions cannot be recreated at the laboratory, stabilized crude oil can be a good alternative. It is not recommended to use non-polar oil or so called "white-oils" in core restoration processes that aim to study wettability processes. Their properties such as the interfacial tension between the oil and water phases, or their capacity to wet the rock differ substantially from that of natural crude oils. Thus, their use should be limited to testing the strength of the capillary forces of the rock in spontaneous imbibition.

Reservoir crude oils are complex mixtures composed of organic components. They contain polar molecules like nitrogen, sulfur and oxygen (NSO)-containing components that contribute with acidic and basic POC which influence the rock surface wettability (Buckley and Morrow 1990). The content of crude oil acids and bases can be determined by measuring the acid number (AN) and base number (BN), both given in mg KOH per gram of oil. It is strongly recommended to quantify these values for the crude oil to be used.

Effect of crude oil exposure

The amount of crude oil injected during core restoration can play a very important role on the resulting wettability. This is not yet fully understood in industry and academia, due to the still accepted method of establishing initial water saturation by oil displacement. As mentioned above oil displacement may provide scattered initial water saturations in a set of cores (McPhee et al. 2015), and can also provoke side errors as non-measured volumes of injection. Crude oil adsorption occurs rapidly as soon as the POC present in the crude oil enter in contact with the rock surface. A previous study by Hopkins et al. (2017) indicated that the amount of crude oil flooded can have a significant impact on chalk wettability. Different amounts of the same crude oil were flooded into outcrop chalk plugs, but the ageing time was kept constant for all cores. Therefore, crude oil exposure dictates the initial wetting more than the ageing process itself, according to the results in **Figure 16.** Here spontaneous imbibition and viscous flooding of formation water were performed in core P3 which had been flooded with 5 PV of crude oil, in core P4 that had been flooded with 15 PV of crude oil and in core Q2, which is a reference core saturated with heptane and that had not been exposed to crude oil.

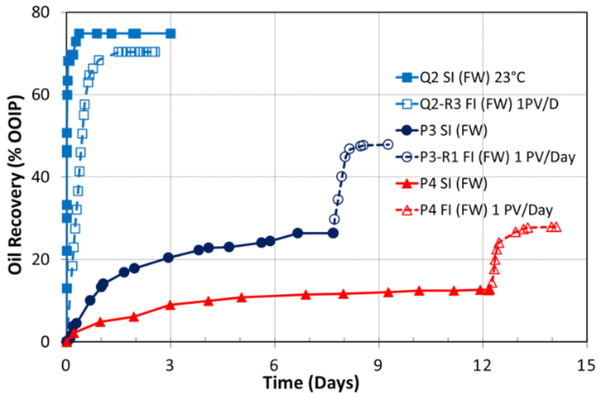
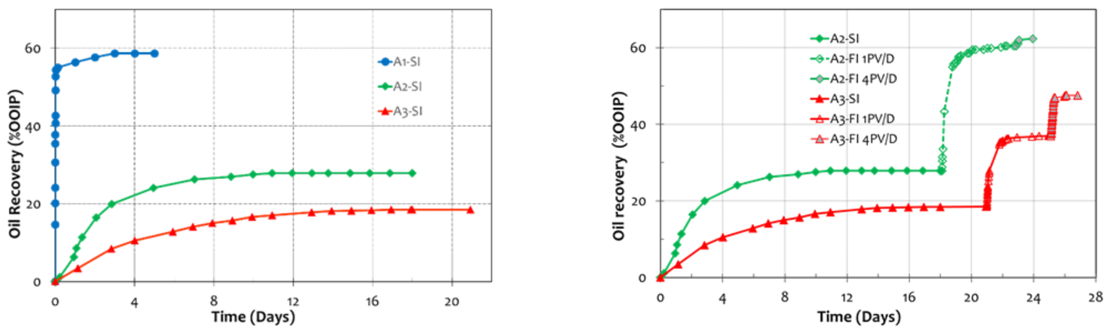


Figure 16. Core restoration of 3 SK chalk cores with Swi = 10%, Q2 – Heptane, P3 – Flooded with 5 PV Crude oil (AN 0.34), P4- core flooded with 15 PV Crude oil (AN 0.34), SI (Spontaneous imbibition, FI (Forced Imbibition, viscous flooding). Tests on cores P3 and P4 at 50 °C and Q2 at 23°C.

The findings were also validated in Aalborg chalk by Konstantinopoulos et al. (2019). This study showed that as the amount of crude oil injected increased, less water-wet conditions were obtained. It was also observed that lower water wetness yielded lower ultimate recovery during forced imbibition, results that are in line with previously published work on Stevns Klint chalk, **Figure 17**.



(a) (b)

Figure 17. Oil recovery tests on Aalborg chalk cores restored with S_{wi} = 0.1 and various oils giving different initial wetting. Core A1 was exposed to heptane, while core A2 and core A3 were exposed to 5 PV and 8 PV crude oil, respectively. (a) SI test with VB0S (FW) as imbibing brine, followed by (b) forced imbibition (FI) with VB0S at a rate of 1PV/D and 4 PV/D. The tests on core A2 and core A3 were carried out at 50 °C, test on core A1 was performed at room temperature.

Both results showed that initial wettability has a profound effect on oil recovery processes, whether it is spontaneous imbibition or forced displacement the effect appears to be consistent and showed a trend. The cores with the most water-wet state, showed significantly higher ultimate oil recovery than the less water-wet cores. These observations are in contradiction to the work presented by Jadhunandan and Morrow (1995), reporting the highest ultimate oil recovery or lowest S_{or} at slightly water-wet conditions.

The effect of reduced water wetness by increased volume of crude oil flooded during core restoration was validated in the study performed by Piñerez et al. (2020), which provided a basis for achieving wettability reproduction between core restorations. In this work, completely water-wet Stevns Klint chalk was initially restored with 5 PV of crude oil. In the second core restoration the same crude oil amount was used. Wettability was measured by spontaneous imbibition before and after the second core restoration. It was found that injection of 5 PV of crude oil into a mildly cleaned core in the second restoration induced a reduction on ultimate oil recovery by spontaneous imbibition compared to the first restoration, **Figure 18**.

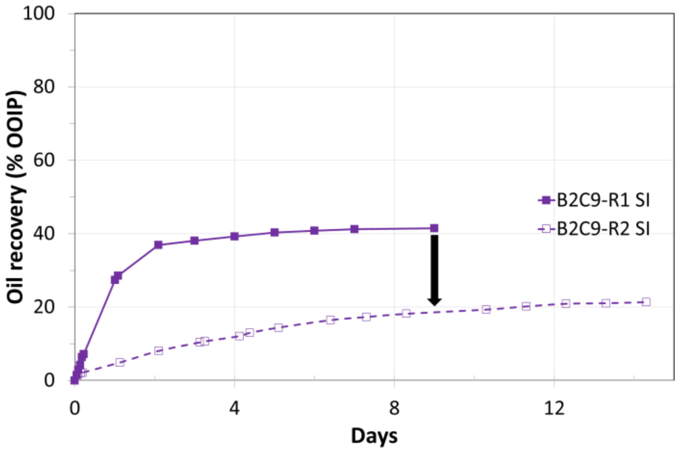


Figure 18. SI tests at 50 °C after initial restoration (R1) and second restoration (R2) in core B29. During R1 the core was exposed to 5 PV Oil B with AN = 0.36 mgKOH/g. After mild cleaning the core was exposed to another 5 PV of Oil B in R2.

The same observation was seen on reservoir sandstones core by Aslanidis et al. (2022). In this study 11 PV of crude oil exposure was used. After the first restoration (R1), the core P6–R1 behaved quite water-wet with an ultimate recovery of 30 %OOIP in SI. After the second core restoration, the core P6–R2 behaved significantly less water-wet, producing 21 %OOIP after 19 days, **Figure 19**.

The study states that a reduction of 30% in water-wetness from restoration 1 to restoration 2 was observed, confirming that the injected volume of crude oil significantly affects core wettability in sandstone cores. Furthermore, it is a parameter that can be optimized as in the work presented by Piñerez et al. (2020).

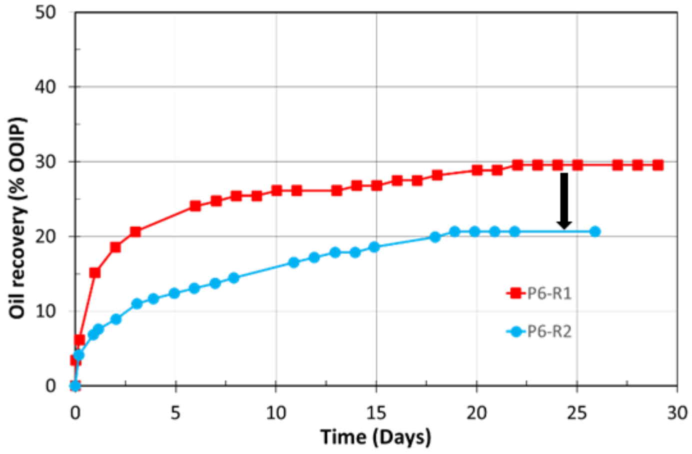


Figure 19. Oil recovery by spontaneous imbibition at 60 °C from core P6, which was cleaned with kerosene/heptane and exposed to 11 PV of crude oil in both restorations R1 and R2.

Effect of adsorption

Dynamic adsorption tests have been performed on carbonates and sandstone outcrop material (Mamonov et al. 2019, Piñerez Torrijos et al. 2020). The main observation was that the acid number (AN) of crude oil is one of the most important parameters influencing carbonates' wettability. In sandstones, the adsorption of basic polar components quantified by the basic number (BN) was more significant than the acid adsorption, the evidence shows that the BN of a crude oil is a more relevant wetting parameter than the AN in clastic reservoirs. Additionally, instantaneous adsorption of POC was observed for both sandstones and carbonates during oil flooding. These results confirmed the work carried out by Hopkins et al. (Hopkins 2016, Hopkins et al. 2016b) on carbonates. Adsorption was controlled by the chemistry of the crude oil-brine-rock system.

Additional adsorption studies in carbonates with mixed mineralogy, were carrried out with outcrop Aalborg chalk; an outcrop chalk with different mineralogical characteristics. This material contains Opal-CT lepispheres made of silicate (Klewiah et al. 2019). The two chalk core systems have similar physical characteristics, except for the large variation in specific surface area due to the Opal-CT contribution. Opal-CT is negatively charged, reducing the overall positive charge density in this material, and limiting the ionic interactions between the negatively charged carboxylates and the Aalborg mineral surface. The presence of negatively charged silicates, creates a competitive adsorption between the acidic and basic components affecting the initial wetting conditions of this material.

In pure carbonate surface, like Stevns Klint, the acids in the crude oil dictated the initial wetting when crude oils with increasing BN were used, as shown by Puntervold et al. (2007b). Base adsorption was explained by co-adsorption with carboxylic acids. The results from Aalborg chalk with silica minerals, shows that the BN adsorption is independent of AN adsorption. Thus, the Opal-CT present in Aalborg chalk, brings on a new surface chemistry in the adsorption process that induces basic component adsorption.

A base-acid adsorption ratio was used to compare the intensity of adsorption between acidic and basic species, and the ratio (BN_{ads}/AN_{ads}) was higher for Aalborg chalk than that for Stevns Klint, confirming that the silicates contributed in a large extent to the available mineral surface in Aalborg chalk (Klewiah et al. 2019). These results can help to understand crude oil adsorption processes in carbonate formation with presence of clays or other silicates that are active in the pore surface.

On sandstones it was shown that adsorption of polar components was dominated by the basic material (Mamonov et al. 2019). By running a mass balance calculation, it was found that about 7 % of the acidic polar components present in the crude oil was adsorbed onto the sandstone surface. On the other hand, a total of 32 % of organic basic material was adsorbed during crude oil injection. The results validate the idea that in sandstones, the adsorption of basic polar components quantified by the base number (BN) was more significant than the acid adsorption. The evidence shows that the BN of a crude oil is a more relevant wetting parameter than the AN in clastic reservoirs.

Ageing

Ageing is carried out in a closed container made of stainless steel. The crude oil exposed cores are placed on top of marble balls and the ageing cell is filled with the same crude oil as that used during oil saturation. In several cases, there is no barrier between the saturated core and the bulk crude oil present in the cell. In such situations non-representative adsorption of POC can take place during the ageing process affecting the core wettability. Therefore, before introducing the core plug into the ageing cell, it is recommended to wrap it in Teflon tape after oil saturation. The Teflon tape is used to avoid additional and unrepresentative adsorption of active POC onto the core material, reducing the likelihood of establishing a non-representative wettability of the core (Standnes and Austad 2000).

It was observed in previous experiments that the adsorption of POC on carbonates occurred rapidly during oil injection, it was also shown that the amount of crude oil injected had more impact on wettability that the ageing time (Hopkins et al. 2016a). Despite this evidence, ageing time is recommended to assure chemical equilibrium of the reservoir system evaluated. An effective measure is to run the ageing process for longer time than the experimental time of the planned test. Thus, 2 to 4 weeks should be enough to obtain an equilibrated system. The ageing temperature should also be the same as the test temperature, this is especially important in the cases where oil saturation has been performed at a different test temperature.

Wettability reproduction

A successful strategy for wettability reproduction, is a combination of different lessons learned. From one side the cleaning methods play an important role. It was seen that mild cleaning was better at preserving POC components onto the rock surface and that a harsh cleaning solvates and removes adsorbed POC to a larger extent. Nevertheless, harsh cleaning also removes asphaltene fractions and precipitated salts that may be of importance in the establishment of initial reservoir wettability.

Crude oil adsorption processes are influenced by the mineral composition and distribution. Adsorption of predominantly crude oil acidic material is higher in carbonates than in sandstones, whereas in sandstones the basic material adsorbs more readily than the acidic material. Additionally, it was observed that reproducibility of experimental procedures is of utmost importance, especially during fluid saturation, where fluid variations can lead to highly scattered wettability results and misleading studies and conclusions. Focus was put on the effect of crude oil exposure during core restoration, since increased crude oil exposure reduce the water wetness of cores, which impacts recovery processes and results.

Wettability reproduction in outcrop chalk

In a recent core restoration study on chalk, it was found that core wettability could be reproduced in core restorations using the mild cleaning scheme with kerosene and heptane

(Piñerez et al. 2020). The study established S_{wi} by the desiccator technique as described by Springer et al. (2003). The amount of crude oil needed to reproduce the wettability was limited to (1–S_{wi}) PV. Larger volumes of crude oil injected gave significantly less water-wet cores. Spontaneous imbibition tests at 50 °C resulted in reproducible oil production profiles and ultimate oil recoveries before and after using the optimized cleaning and restoration procedure. The observed differences in ultimate oil recovery between restorations were in the range of +1.5 - 3.6% OOIP, **Figure 20**.

The study suggested that the cleaning and restoration procedure described, could be used for reservoir wettability studies in carbonate cores. The method can lead to more reliable SCAL analyses, which are critical for reservoir characterization and production performance studies.

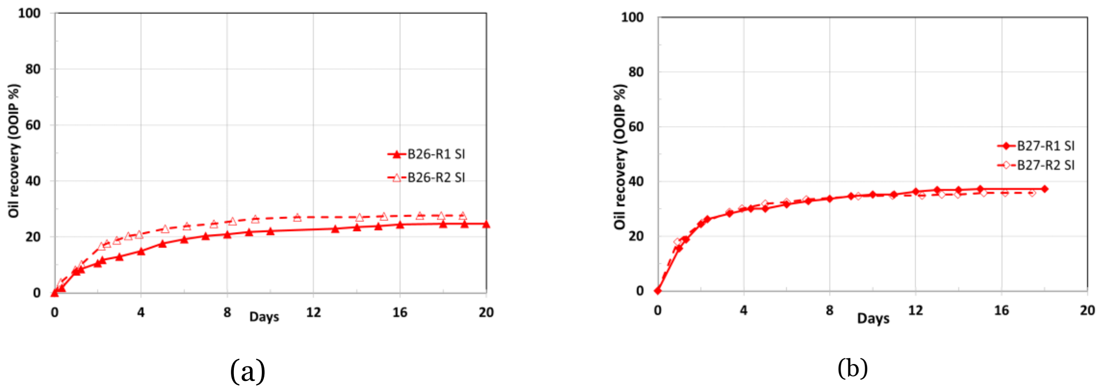
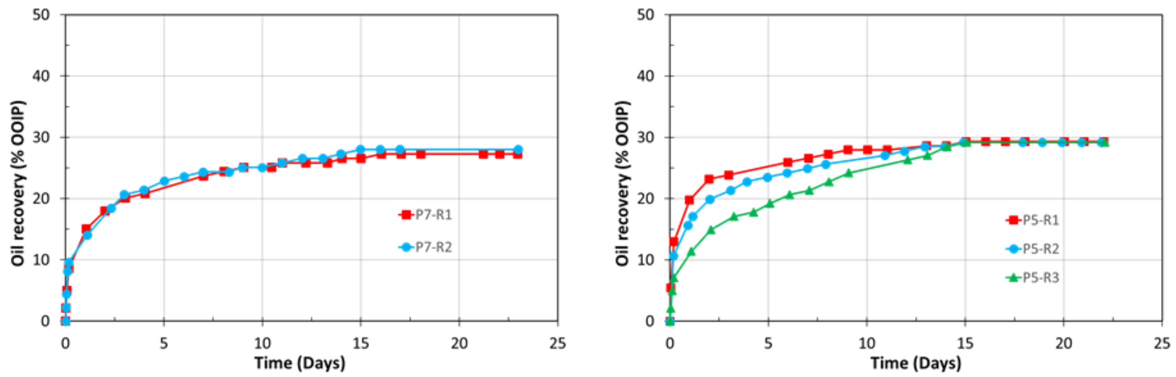


Figure 20. Oil recovery by spontaneous imbibition at T_{test}=50 °C after initial restoration (R1) and second restoration (R2) using the optimized cleaning and restoration process. Stevns Klint chalk Core B26 (a) and Core B27 (b) were both restored with S_{wi} = 0.1. and exposed to crude oil with AN = 0.5 mgKOH/g.

Reproducing wettability in sandstone reservoir cores

Following the procedure outlined above for chalk wettability restoration, Aslanidis et al. (2022) adapted similar methodology to reproduce wettability in sandstone reservoir (Varg) cores. Mild cleaning with kerosene and heptane was performed to maintain most of the POC adsorbed in the preserved reservoir core. Crude oil exposure was limited to (1–S_{wi}) PV in the restoration process. Spontaneous imbibition tests were performed for wettability evaluation. The sandstone core P7 reproduced the wettability between restorations 1 and 2 by a difference of only 1 %OOIP and imbibition rates, and ultimate oil recovery were similar, **Figure 21**.

In a second core, P5, wettability reproduction based on ultimate recovery was achieved in 3 consecutive restorations, and the imbibition profiles were similar in restoration 1 and 2. However, in restoration 3, the core was already exposed to a total of 3 PV of crude oil, which may explain why the production profile was not fully reproduced in the third restoration. Nonetheless, the same ultimate oil recovery of 29 %OOIP was achieved, indicating that wettability and fluid flow paths in the porous media were not significantly altered.



(a) **(b)**

Figure 21. Oil recovery by spontaneous imbibition at 60°C of the Varg reservoir sandstone cores P7 (a) and P5 (b) cleaned with kerosene and heptane and exposed to $(1–S_{wi})$PV of crude oil.

Evaluation of reservoir wettability

Effects of varying core preparation procedures on wettability have been demonstrated in both chalk and sandstone cores. Reliable wettability characterization is fundamental for waterflooding operations or for evaluating efficiency of EOR/IOR-methods. A good example is in the studies of Smart Water or low salinity EOR-potential, where the increased oil recovery is a result of wettability alteration toward more water-wet conditions (Austad et al. 2010, Aghaeifar et al. 2015, Pinerez Torrijos et al. 2016, Mamonov et al. 2019, Puntervold et al. 2021). It has been found that at strongly water-wet conditions in the reservoir, the EOR-potential by wettability alteration to more water-wet conditions will be rather low (Aghaeifar et al. 2015). Therefore, it is of high importance to be aware of how core restoration processes impact the wettability condition of the core material during SCAL or EOR studies. The experimental examples provided here contribute to the optimization of laboratory routines for core restoration, that in turn can help to obtain reproducible results and perhaps wettability measurements that are closer to the original reservoir wettability.

Mud contamination can represent a major challenge for special core analyses. However, as shown above the preserved reservoir cores that are used in laboratory experiments imbibe water after their first restoration, meaning that their capillary forces have not been seriously affected or that mud contamination did not take place. In **Figure 22** oil recovery results from Varg reservoir sandstone cores after the first restoration process are shown (Aslanidis et al. 2022).

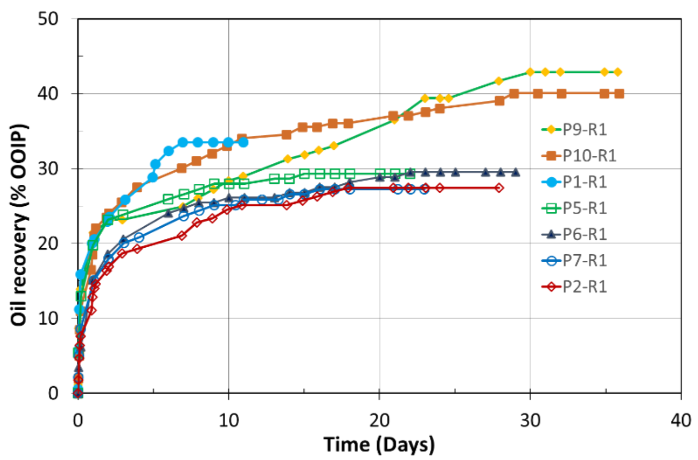


Figure 22. SI experiments of all cores after the first restoration.

The spontaneous imbibition tests show ultimate oil recovery values from water imbibition that ranged from 27 %OOIP to 45 %OOIP, meaning that all these cores are not very much affected by the drilling fluid. Water-wet preserved reservoir core material has been frequently observed in the EOR-laboratory at the University of Stavanger. NORCE reports that contamination of reservoir core plugs both by OBM and WBM have been observed in several studies. Unpublished results at UiS have also shown that initially water-wet reservoir cores have been seriously affected by extensive mud filtrate exposure to become oil-wet. In conclusion, not all reservoir cores are suffering from mud invasion and contamination. The potential for mud invasion and contamination clearly depends on the mud content, as discussed above, and properties of the reservoir rock and its wettability.

The oil recovery results from Varg reservoir sandstone cores presented in **Figure 22** displayed high variability. Even twin cores sharing core restoration methods displayed differences in the spontaneous imbibition processes. This natural variability in wettability is probably derived from mineral heterogeneity, i.e., mineralogy and porous size distribution (Aslanidis et al. 2022). Therefore, to be able to observe and understand these differences in wettability, reliable and reproducible core restoration procedures are needed. Furthermore, these results indicate that comparison between different cores can be challenging and, in many cases, not convenient. Thus, it should be beneficial and recommended to perform comparative tests on the same core. This is one of the most important reasons for why using protocols for wettability reproduction is a strategic point in SCAL evaluation.

Conclusions and recommendations

This recommended practice contains core preparation procedures for obtaining reproducible core wettability in the laboratory. Only relevant procedures after the cores have been received in the laboratory are considered. Reproducible wettability in core material is essential for correctly evaluating Smart Water EOR potential (See Recommended Practice "Smart Water flooding: Part1-Laboratory workflow for screening EOR potential").

When preserved rock samples arrive in the laboratory, several control measures for mud invasion should be taken. Mud composition, brine composition, oil and gas properties and the core mineralogy should be known to select the optimum cleaning methods and analytical techniques. Mud filtrate invasion can be determined by analyzing brine effluents for WBM with diverse techniques like, ion chromatography, ICP-OES or pH measurements. For investigating possible OBM contamination, interfacial tension measurements can detect contamination by surface active components from OBM. Imaging techniques such as SEM together with EDS can help to identify mud particles left by drilling fluids. If mud contamination is confirmed, it is advisable to not the cores for wettability studies.

Laboratory experiments for determination of wettability, SCAL and EOR potentials should be carried out with preserved reservoir rock and ideally with live fluids at reservoir conditions. However, stabilized stock tank oil could also be used if reservoir conditions cannot be obtained in the laboratory, as the components wetting reservoir rocks are mostly contained in the liquid oil. It should be confirmed that the received stabilized crude oil samples, live oil samples, and formation water composition are not contaminated by mud. In the case of FW composition, synthetic brines with equilibrated composition can be used.

The choice of cleaning solvents and procedures affects the wettability of the rock. Mild cleaning with kerosene and heptane aims to preserve wettability, while harsh cleaning aims to completely clean the core to water-wet conditions before restoring wettability. Mild cleaning with low solvent volumes only removes a small fraction of the initial adsorbed polar components that created the initial wettability. Then, in the core restoration process, less crude oil can be used in the wettability restoration, and the newly restored core will most likely be closer to the initial wettability prior to laboratory experiments.

Reproducibility of experimental conditions is of high importance. Therefore, desiccator is a preferred method for establishing S_{wi}, compared to the porous plate or dynamic oil displacement to achieve repeatable values of initial water and oil saturations. Not only is the desiccator method very fast, but reproducible and predetermined S_{wi}-values are obtained. In addition, the extent of crude oil exposure during oil saturation is of extreme importance for the final wettability state of the core. Therefore, to establish initial water and oil saturations by crude oil injection will lead to potentially large variation in S_{wi}, crude oil exposure and in consequence, the core wettability. After mild cleaning, and establishment of S_{wi} by desiccator, a minimum amount of crude oil should be used to get closer to initial wettability.

Experimental evidence questions the need for ageing the core in crude oil after crude oil injection to render cores less water-wet or mixed-wet. Adsorption of POC onto mineral surfaces is a rather fast process; however, ageing should be performed to assure chemical equilibrium. Typically, the ageing time should be set to last longer than the experimental test. Wrapping of the core in Teflon tape during ageing is also recommended because it has been found to reduce non-representative adsorption of POC onto chalk surfaces during the ageing phase.

Initial wettability was successfully reproduced in a second core restoration process for both outcrop chalk and reservoir sandstone material. Imbibition rate was successfully reproduced, and ultimate oil recoveries varied by less than 4 %OOIP. Wettability reproduction was achieved by following the recommended workflow presented in **Figure 23**.

Challenging conditions for core restoration can originate from the sampling phase, and limited communication between the drilling and core analyses teams. Mud invasion and possible contamination is dependent on e. g. drilling mud chemicals, reservoir minerals and wettability. More work should be done to understand the interactions between these and possible effects of OBM or WBM on wettability, and how mud invasion can be best avoided. Thus, inter-disciplinary integration and in-depth collaboration among the project participants should be the main point to improve in a SCAL project. In this way, problems like severe mud contamination and its consequences can be minimized.

Furthermore, standard industry cleaning to complete water-wet conditions appears to be a methodology that reduces the accuracy of wettability measurements and should be challenged. The mild cleaning methodology proposed in this recommended practice should be validated in collaboration with service companies. Therefore, more collaboration in form of applied research projects should be created among academia, service companies and oil operators to improve reservoir wettability estimates. Testing and validation of new alternatives to the established protocols are vital for the industry to not become obsolete in this field.

The effects of the oil phase during core restoration on wettability and oil recovery processes should be further investigated. The work in this report pointed out knowledge gaps in the understanding of wettability processes. At their experimental conditions, Jadhunandan & Morrow (1995) suggested that maximum oil recovery is achieved at neutral, slightly water-wet conditions. Those observations are in contradiction to the findings of this work, that suggest that higher recovery is reached at more water-wet conditions instead of at mixed-wet state.

Repeatability of experiments is also of major importance in core restoration procedures, and the use of methods like the centrifuge, porous plate, and establishment of initial water saturation by oil displacement only add more variability to the experimental results affecting seriously the precision of the methods. Scattered results from the SCAL program will be the main consequence, reducing the value of the experimental data. Thus, the operators should create a quality control framework or system to guarantee the repeatability of experimental results.

When these challenges are addressed, application of optimized core restoration procedures can contribute to improved reservoir characterization methodologies and understanding of hydrocarbon recovery processes, allowing the use of laboratory results with enhanced precision and accuracy for simulation studies, EOR/IOR projects or fluid flow understanding beyond hydrocarbon recovery, e.g., CO_2, and H_2 storage.

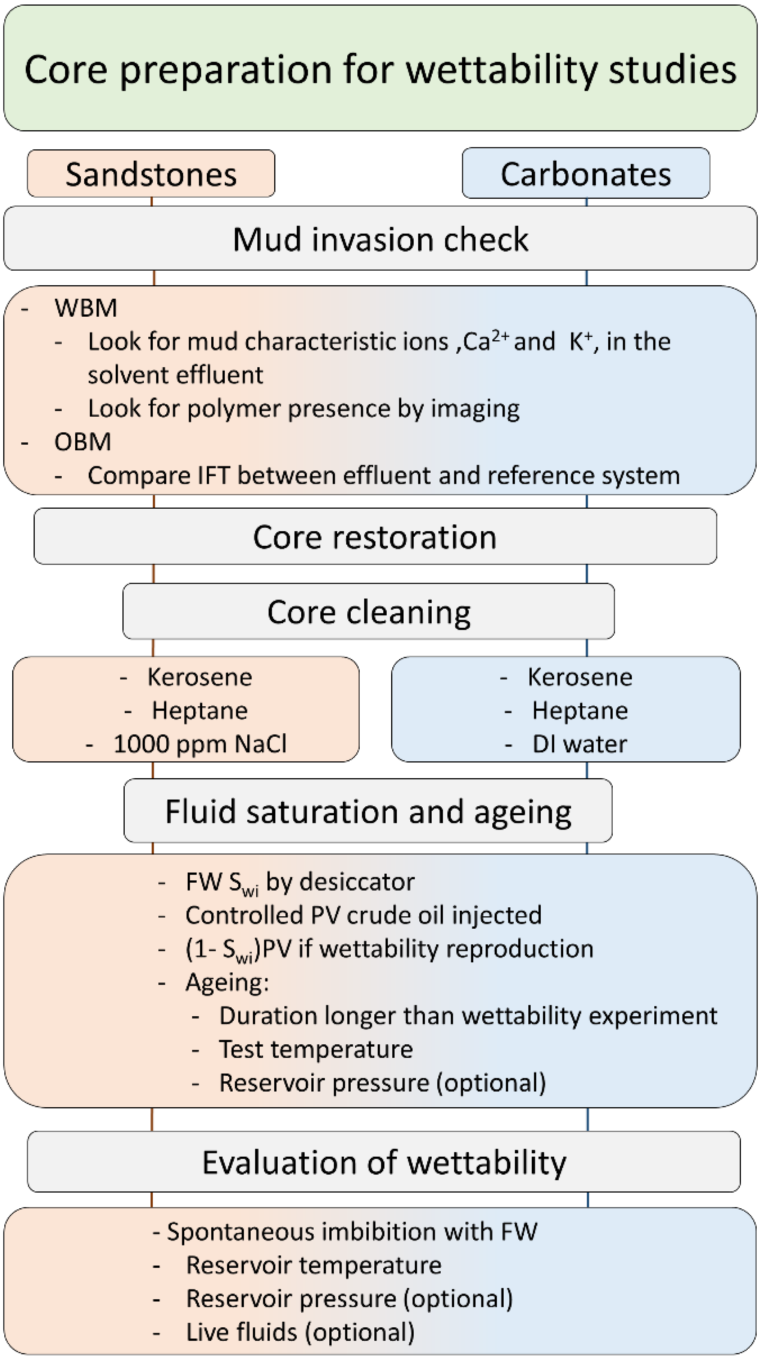


Figure 23: Smart Water practice for core preparation for wettability studies

References

Abeysinghe, K. P., Fjelde, I. and Lohne, A. 2012. Dependency of remaining oil saturation on wettability and capillary number Paper SPE-160883 presented at the 2012 SPE Technical symposium and Exhibition, Al-Khobar, Saudi Arabia, 8-11 April.

Aghaeifar, Z., Puntervold, T., Strand, S., Austad, T., Maghsoudi, B. and Ferreira, J. d. C. 2018. Low Salinity EOR Effects After Seawater Flooding in a High Temperature and High Salinity Offshore Sandstone Reservoir. SPE Norway One Day Seminar, Bergen, Norway, 2018/4/18/.

Aghaeifar, Z., Strand, S., Austad, T., Puntervold, T., Aksulu, H., Navratil, K., Storås, S. and Håmsø, D. 2015. Influence of Formation Water Salinity/Composition on the Low-Salinity Enhanced Oil Recovery Effect in High-Temperature Sandstone Reservoirs. *Energy & Fuels* **29**(8): 4747-4754.

Anderson, W. G. 1986. Wettability Literature Survey - Part 1: Rock/Oil/Brine interactions and the effects of core handling on wettability. *Journal of Petroleum Technology* **October**: 1125-1144.

API. 1998. Recommended Practices for Core Analysis, API Publishing Services.

Aslanidis, P., Strand, S., Pinerez Torrijos, I. D. and Puntervold, T. 2022. Reproducing wettability in sandstone reservoir core material in laboratory core restorations. *Journal of Petroleum Science and Engineering* **208**: 109531.

Austad, T., RezaeiDoust, A. and Puntervold, T. 2010. Chemical mechanism of low salinity water flooding in sandstone reservoirs. Paper SPE 129767 prepared for presentation at the 2010 SPE Improved Oil Recovery Symposium, Tulsa, Oklahoma, USA, 24-28 April.

Austad, T., Shariatpanahi, S. F., Strand, S., Aksulu, H. and Puntervold, T. 2015. Low salinity EOR effects in limestone reservoir cores containing anhydrite: a discussion of the chemical mechanism. *Energy & Fuels* **29**(11): 6903-6911.

Bobek, J. E., Mattax, C. C. and Denekas, M. O. 1958. Reservoir Rock Wettability - Its Significance and Evaluation. *Transactions of the AIME* **213**(01): 155-160.

Buckley, J. S. (1994). Chemistry of the crude oil/brine interface. Proceedings of the 3rd International Symposium on Evaluation of Reservoir Wettability and Its Effect on Oil Recovery, Laramie, WY, USA.

Buckley, J. S. and Fan, T. 2007. Crude oil/brine interfacial tensions. *Petrophysics (Houston, Tex.)* **48**(3): 175-185.

Buckley, J. S. and Liu, Y. 1998. Some mechanisms of crude oil/brine/solid interactions. *Journal of Petroleum Science and Engineering* **20**: 155-160.

Buckley, J. S., Liu, Y. and Monsterleet, S. 1998. Mechanisms of wetting alteration by crude oils. *SPE Journal* **March**: 54-61.

Buckley, J. S. and Morrow, N. R. 1990. Characterization of crude oil wetting behavior by adhesion tests. Paper SPE/DOE 20263 presented at the SPE/DOE Seventh Symposium on Enhanced Oil Recovery, Tulsa, Oklahoma, April 22-25.

Cuiec, L. E. 1975. Restoration of the natural state of core samples - SPE 5634. The 50th annual fall meeting of the Society of Petroleum Engineers of AIME, Dallas, TX, USA, Sep 28 - Oct 1.

ESSO. 1966. Reservoir Engineering Manual. United States of America.

Fathi, S. J., Austad, T. and Strand, S. 2010. "Smart Water" as wettability modifier in chalk: The effect of salinity and ionic composition. *Energy & Fuels* **24**: 2514-2519.

Fjelde, I. 2017. Effect of Mud Invasion on the Determined Low Salinity Water Flooding Potential. **2017**(1): 1-14.

Fjelde, I. (2021). Effects of mud exposure on established wettability conditions. IOR NORWAY 2021-14th International sympoisum on reservoir wettability and its effects on oil recovery, Stavanger, Norway.

Fjelde, I., Voke, O. A. and Wetrhus Minde, M. 2015. Removal of mud components from reservoir sandstone rocks. International Symposium of the Society of Core Analysts held in St. John's Newfoundland and Labrador, Canada, 16-21 August, 2015 St. John's Newfoundland and Labrador, Canada.

Fulcher, R. A., Jr., Ertekin, T. and Stahl, C. D. 1985. Effect of capillary number and its constituents on two-phase relative permeability curves. *Journal of Petroleum Technology* **37**(2): 249-260.

Gant, P. L. and Anderson, W. G. 1988. Core Cleaning for Restoration of Native Wettability. *SPE Formation Evaluation* **3**(01): 131-138.

Grist, D. M., Langley, G. O. and Neustadter, F. L. 1975. The Dependence of Water Permeability On Core Cleaning Methods In the Case of Some Sandstone Samples. *Journal of Canadian Petroleum Technology* **14**(02).

Guo, H., Dou, M., Hanqing, W., Wang, F., Yuanyuan, G., Yu, Z., Yansheng, W. and Li, Y. 2015. Review of capillary number in chemical enhanced oil Recovery. Paper SPE 175172 presented at the 2015 Oil and Gas Show and Conference, Mishref, Kuwait, 2015/10/11/.

Hopkins, Walrond, K., Strand, S., Puntervold, T., Austad, T. and Wakwaya, A. 2016a. Adsoprtion of acidic crude oil components onto outcrop chalk at different wetting conditions during both dynamic adsorption and aging processes. *Energy & Fuels* **30**(9): 7229-7235.

Hopkins, P. A. (2016). Water-Based EOR and Initial Wettability in Carbonates, University of Stavanger.

Hopkins, P. A., Omland, I., Layti, F., Strand, S., Puntervold, T. and Austad, T. 2017. Crude Oil Quantity and Its Effect on Chalk Surface Wetting. *Energy & Fuels* **31**(5): 4663-4669.

Hopkins, P. A., Strand, S., Puntervold, T., Austad, T., Dizaj, S. R., Waldeland, J. O. and Simonsen, J. C. 2016b. The adsorption of polar components onto carbonate surfaces and the effect on wetting. *Journal of Petroleum Science and Engineering* **147**: 381-387.

Jadhunandan, P. P. and Morrow, N. R. 1995. Effect of wettability on waterflood recovery for crude-oil/brine/rock systems. *SPE Reservoir Engineering* **February**: 40-46.

Jennings, H. Y., Jr. 1957. Effect of Laboratory Core Cleaning on Water-Oil Relative Permeability.

Klewiah, I., Piñerez Torrijos, I. D., Strand, S., Puntervold, T. and Konstantinopoulos, M. 2019. Adsorption of Crude Oil Polar Components onto Silica-Rich Chalk and its Impact on Wetting. SPE Norway One Day Seminar, Bergen, Norway, 2019/5/13/.

Konstantinopoulos, M., Piñerez Torrijos, I. D., Klewiah, I., Strand, S. and Puntervold, T. 2019. Effect of Mineralogy on Initial Wettability and Oil Recovery from Silica-containing Chalk. **2019**(1): 1-5.

Lopez-Salinas, J. L., Hirasaki, G. and Millar, C. A. 2011. Determination of anhydrite in reservoirs for EOR. Paper SPE 141420 presented at the SPE International Symposium on Oilfield Chemistry, The Woodlands, Texas, 11-13 April.

Mamonov, A., Kvandal, O. A., Strand, S. and Puntervold, T. 2019. Adsorption of Polar Organic Components onto Sandstone Rock Minerals and Its Effect on Wettability and Enhanced Oil Recovery Potential by Smart Water. *Energy & Fuels* **33**(7): 5954-5960.

McPhee, C., Reed, J. and Zubizarreta, I. 2015. Core analysis: a best practice guide. Amsterdam, Netherlands, Elsevier.

Mjos, J. E. S., Strand, S., Puntervold, T. and Gaybaliyev, H. 2018. Effect of Initial Wetting on Smart Water Potential in Carbonates. Paper presented at the 2018 SPE EOR Conference at Oil and Gas West Asia, Muscat, Oman, 2018/3/26/.

Piñerez, I., Puntervold, T., Strand, S., Hopkins, P., Aslanidis, P., Yang, H. S. and Kinn, M. S. 2020. Core wettability reproduction: A new solvent cleaning and core restoration strategy for chalk cores. *Journal of Petroleum Science and Engineering* **195**: 107654.

Piñerez Torrijos, I. D., Mamonov, A., Strand, S. and Puntervold, T. 2020. The role of polar organic components in dynamic crude oil adsorption on sandstones and carbonates. *CT&F - Ciencia, Tecnología y Futuro* **10**(2): 5-16.

Pinerez Torrijos, I. D., Puntervold, T., Strand, S., Austad, T., Abdullah, H. I. and Olsen, K. 2016. An experimental study of the response time of the low salinity EOR effect during secondary and tertiary low salinity waterflooding. IEA-EOR – 37th Annual Workshop & Symposium, Rueil Malmaison, 2016-09-18 - 2016-09-22.

Pinerez Torrijos, I. D., Risanger, M., Puntervold, T., Strand, S. and Austad, T. 2017. Impact of anhydrite on the low salinity EOR effect in sandstone material with high clay content.

IOR NORWAY 2017 - 19th European Symposium on Improved Oil Recovery, Stavanger, 2017-04-24 - 2017-04-27.

Puntervold, T. 2008. Waterflooding of carbonate reservoirs - EOR by wettability alteration, University of Stavanger, Norway. ISBN 978-82-7644-347-9.

Puntervold, T., Mamonov, A., Piñerez Torrijos, I. D. and Strand, S. 2021. Adsorption of Crude Oil Components onto Carbonate and Sandstone Outcrop Rocks and Its Effect on Wettability. *Energy & Fuels*.

Puntervold, T., Strand, S. and Austad, T. 2007a. New method to prepare outcrop chalk cores for wettability and oil recovery studies at low initial water saturation. *Energy & Fuels* **21**(6): 3425-3430.

Puntervold, T., Strand, S. and Austad, T. 2007b. Water Flooding of Carbonate Reservoirs: Effects of a Model Base and Natural Crude Oil Bases on Chalk Wettability. *Energy & Fuels* **21**(3): 1606-1616.

Puntervold, T., Strand, S., Ellouz, R. and Austad, T. 2015. Modified seawater as a smart EOR fluid in chalk. *Journal of Petroleum Science and Engineering* **133**: 440-443.

RezaeiDoust, A., Puntervold, T. and Austad, T. 2011. Chemical verification of the EOR mechanism by using low saline/smart water in sandstone. *Energy & Fuels* **25**(5): 2151-2162.

Richardson, J. G., Holstein, E. D., Rathmell, J. J. and Warner, H. R., Jr. 1997. Validation of As-Received Oil-Based-Core Water Saturations From Prudhoe Bay. *SPE Reservoir Engineering* **12**(01): 31-36.

Ringen, J. H., C; Lehne, K.A; Rueslaatten, H; Holand, H. 2001. Reservoir Water Saturations Measured on Cores; Case Histories and Recommendations. 6th Nordic Symposium on Petrophysics, 15-16 May.

Shariatpanahi, S. F., Strand, S., Austad, T. and Aksulu, H. 2012. Wettability restoration of limestone cores using core material from the aqueous zone. *Petroleum Science and Technology* **30**(1-9).

Shimoyama, A. and Johns, W. D. 1971. Catalytic Conversion of Fatty Acids to Petroleum-like Paraffins and their Maturation. *Nature Physical Science* **232**(33): 140-144.

Shimoyama, A. and Johns, W. D. 1972. Formation of alkanes from fatty acids in the presence of $CaCO_3$. *Geochimica et Cosmochimica Acta* **36**(1): 87-91.

Springer, N., Korsbech, U. and Aage, H. K. 2003. Resistivity index measurement without the porous plate: A desaturation technique based on evaporation produces uniform water saturation profiles and more reliable results for tight North Sea chalk. Paper presented at the International Symposium of the Society of Core Analysts Pau, France, 21-24 Sept.

Standnes, D. C. and Austad, T. 2000. Wettability alteration in chalk: 1. Preparation of core material and oil properties. *Journal of Petroleum Science and Engineering* **28**(3): 111-121.

Treiber, L. E. and Owens, W. W. 1972. A laboratory evaluation of the wettability of fifty oil producing reservoirs. *SPE Journal* **12**(6): 531-540.

Wendell, D. J., Anderson, W. G. and Meyers, J. D. 1987. Restored-State Core Analysis for the Hutton Reservoir. *SPE Formation Evaluation* **2**(04): 509-517.