The Development of a Powder-Flotation Protocol for Rapid Screening of Aging- and Cleaning-Induced Wettability Alterations in Sandstone

Anisa Nugrahaningtyas[1,*], *Mohammad* Masoudi[2], *Bård* Johan Arnt Bjørkvik[2], *Carl* Fredrik Berg[1], and *Antje* van der Net[1]

[1] Department of Geosciences, Norwegian University of Science and Technology (NTNU), 7031 Trondheim, Norway
[2] Applied Geoscience Department, SINTEF Industry, 7465 Trondheim, Norway

Abstract. This study develops a powder-flotation workflow for rapid screening of wettability alterations in Bleurswiller sandstone induced by crude-oil aging and subsequent solvent cleaning. Powder (50–74 μm) was aged with three North Sea crude oils and cleaned with five protocols: Isopar-L, hexane filtration-only (Hx+F), subsequent continued hexane cleaning with mechanical agitation (Hx+FS), toluene–methanol filtration-only (TM+F), and subsequent continued toluene–methanol cleaning with mechanical agitation (TM+FS). Wettability is quantified through a Flotation Index (FI), where FI ≈ 0% indicates fully water-wet conditions. Isopar-L and hexane proved insufficient for efficient cleaning towards a fully water wet state, with nearly all FI values remaining above 2%. Toluene–methanol cleaning significantly reduces FI across all crude oils (TM+F: 0.32–1.40%; TM+FS: 0.23–0.82%), approaching fully water-wet conditions (FI ≈ 0%). Repeatability tests confirm robust qualitative classification, although inter-run variability under non-water-wet conditions is high. A mass-sensitivity study could indicate that FI is powder-mass-dependent under oil-wet conditions, while water-wet conditions show little sensitivity to powder mass. The protocol provides a time-efficient, qualitative tool for detecting wettability alteration and evaluating solvent-cleaning effectiveness in sandstone outcrop material. Remaining work includes reducing inter-run variability and constraining powder-mass effects to enable precise quantitative wettability assessment.

1 Introduction

Wettability is one of the most fundamental properties in reservoir engineering, defined as the tendency of a reservoir rock's surface to wet gas, oil, or water [1]. It governs fluid distribution and flow in porous media, which are important for describing multiphase flow like CO_2 storage and hydrocarbon production [2,3]. Wettability strongly influences multiphase reservoir rock (core) analyses, including capillary pressure, relative permeability, waterflood behaviour, and electrical properties [1]. However, wettability remains a difficult reservoir parameter to characterize reliably, reproducibly, and at high throughput.

Common practices use conventional core-scale methods, notably the Amott–Harvey index and the U.S. Bureau of Mines (USBM) method, which provide bulk wettability averages from spontaneous and forced imbibition cycles [4,5]. Nevertheless, these methods are time-consuming, require long experimental durations and a high level of expertise, are affected by pore structure and experimental errors, and are expensive due to equipment setup and experimental time [6]. On the other hand, a quick and well-established wettability measurement method, such as Contact Angle (CA), is available to assess macroscopic wettability on polished mineral surfaces or microscopically, in-situ in cores. Nonetheless, CA are constrained by the use of idealized smooth substrates that poorly represent the mineralogical complexity and surface roughness of real reservoir rock [1,6].

Therefore, the limitations of established methods have long motivated the search for simpler, faster, and more scalable wettability screening tools [8]. Powder flotation, also termed the float–sink or modified flotation technique (MFT), has emerged as a grain-scale alternative to measure the wettability that operates on crushed rock material, and can be performed rapidly in standard laboratory glassware [7,9,10]. The principle is to crush the rock samples into a fine powder, mix it with an immiscible oil–water mixture, and let the system equilibrate. Eventually, if gravity forces can be overcome, oil-wet particles would preferentially associate with the oil phase (float fraction), while water-wet particles would settle into the water phase (sink fraction) [8,9]. A comparative metric of fractional wettability can then be estimated from the mass fractions recovered in each zone, quantifying the proportion of grains preferentially associated with the oil or water phase. This method is particularly attractive due to its straightforward methodology, and it has potential for application to drill cuttings as it does not require intact core plugs, enabling quasi-continuous reservoir wettability characterization along a borehole trajectory [8].

However, despite its conceptual simplicity and practical appeal, the powder-flotation method lacks a standardized protocol. The operating conditions governing flotation, including particle size and powder

* Corresponding author: anisa.nugrahaningtyas@ntnu.no

mass, have not been consistently controlled or reported across studies, fundamentally limiting cross-study comparability. Other variables also require standardization: powder mass controls the powder-to-fluid ratio, and inter-particle clumping affects the accurate weighing of powder mass added; the fluid-grain system is governed by interfacial tension and capillary forces acting on grains at the interface; the mixing procedure determines initial powder–fluid contact; settling time affects the degree of phase equilibration; and phase-separation technique influences how completely each fraction is recovered. According to Ghosh et al. [8], particle size plays a dominant role in controlling phase partitioning: when particles are too large, gravitational settling overrides the surface-energy-driven affinity, rendering the float–sink ratio insensitive to the true wetting state. These observations emphasize the need for systematic method development before the flotation technique can be relied upon for comparative screening.

The present study addresses this gap by developing a standardized powder-flotation workflow applied to sandstone powder. A systematic evaluation of cleaning solvent choice, mixing protocol, settling time, and powder mass were done as part of method development. Three North Sea crude oils with distinct origins (Oil A, Oil B, and Oil C) were used to evaluate the robustness of the protocol across different oil chemistries. This study demonstrates that the measured wettability response reliably reflects the wetting state of the grains, selecting between water and oil wet, rather than being an outcome of the measurement procedure itself, confirming that the results are independent of controllable methodological variables. The validated protocol detects crude-oil-induced wettability alteration and tracks progressive wettability restoration upon solvent cleaning.

2 Materials and Methods

2.1 Rock Material

Bleurswiller sandstone (Vosges, France) was selected as the core sample. Literature data indicates this outcrop sandstone comprises primarily quartz (>66%), 28% feldspar, 4% clays, and 2% mica [12]. Core material was crushed and dry-sieved into a grain fraction of 50–74 µm.

2.2 Crude Oils

Three North Sea crude oils from different fields were selected to evaluate the robustness of the protocol across compositionally distinct crude oils. The densities of all three oils are given in Table 1.

Table 1. Oil densities at atmospheric pressure and temperature 15.0 ºC.

Name	Density (g/ml) @15.0 ºC
Oil A	0.86
Oil B	0.89
Oil C	0.92

2.3 Powder-Aging Protocol

The prepared Bleurswiller powder was submerged in crude oil and aged statically at 80 °C for 5 days. After aging, the powder was transferred to a funnel, and excess bulk crude oil was first removed by vacuum filtration. The powder was then rinsed with Isopar-L (3 × 100 mL) under vacuum filtration to remove remaining loosely bound oil, yielding the 'Aged' powder baseline for subsequent flotation experiments or solvent-cleaning sequences.

2.4 Solvent-Cleaning Procedure

The aged powder was then cleaned using two different solvents:

1) Hexane (mild cleaning) to remove light, non-polar aliphatic oil components while leaving the heavier polar and asphaltenic surface-active compounds intact on the grain surface [11,12].
2) Toluene and methanol (harsh cleaning) to dissolve all oil components, both polar and non-polar.

For each solvent, we employ two different cleaning methods:

1) Filtration only (F): using a solvent of 300 mL volume which is drained through the filtration unit without vacuum by repeated filtration (3 cycles)
2) Filtration + Shaking (FS): filtration-cleaned powder is then subjected to solvent of 45 mL through mechanical agitation using a shaker at 300 RPM for 12 hours.

The detailed procedure is illustrated in Figure 1.

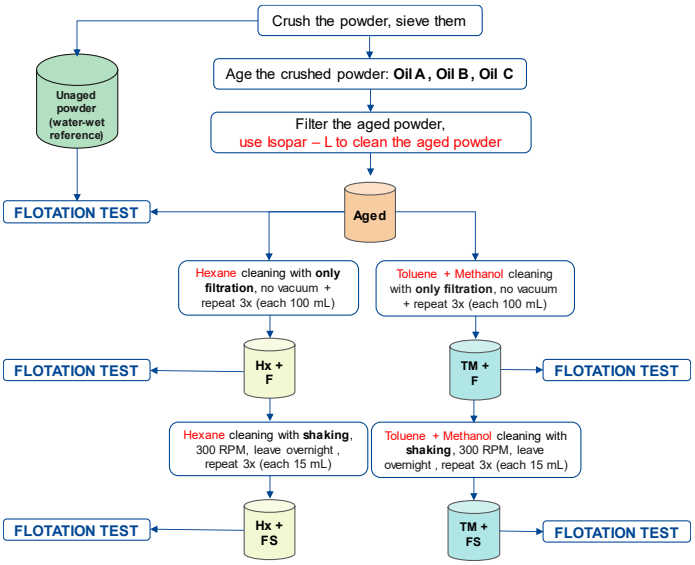

Fig. 1. Experimental plan procedure studying the effect of aging and solvent effectiveness.

2.5 Flotation Protocol

The flotation test was conducted at room temperature in sealed glass jars with an internal diameter of 25 mm, yielding a fixed cross-sectional area of 4.9 cm² and a height of 54 mm. A defined mass of powder (1 gram or 2 grams) was introduced into the jar, followed by the addition of the oil phase (Isopar-L as the model oil phase) and the aqueous phase (deionized water) at a fixed 1:1 oil-to-water volume ratio (5 mL each). The jars were manually inverted and gently swirled to promote homogeneous contact between the powder and both fluid phases, avoiding vigorous agitation that could introduce air bubbles. Afterward, the powder was allowed to settle at room temperature. Visual observations were recorded

at Day 1, Day 2, Day 3, and Week 1. This single-mixing approach differs from that of Mwangi et al. [9], who applied vigorous stirring multiple times every 8–12 hours, followed by a 24-hour settling period. In the present study, gentler mixing was adopted to avoid bubble formation and the emulsion building that could interfere with the gravimetric phase separation. Following an assumed complete phase equilibration at Week 1, the entire oil phase, including any material at the oil–water interface, was extracted using a syringe and transferred to a pre-weighed vessel (float fraction). The remaining water phase was similarly transferred to a separate pre-weighed vessel (sink fraction). Both fractions were dried at 80 °C to constant mass before gravimetric weighing. The Flotation Index (FI) was calculated as the float fraction of the total mass:

$$\mathrm{FI} = \frac{m_{float}}{m_{float}+m_{sink}} \quad (1)$$

where m_{float} is the mass of particles recovered from the oil phase and the oil–water interface, and m_{sink} is the mass of particles recovered from the water phase.

3 Results and Discussion

3.1 Reference Wetting States

A fully water-wet reference condition was established by placing the unaged, uncleaned Bleurswiller powder directly into the flotation jar. This fully water-wet sample yielded light-coloured grey grains that sank rapidly to the bottom of the water phase at Day 1 and remained there through Week 1, with a sharp, flat oil–water interface, a colourless Isopar-L phase, and no floating or interfacial material (see Table 2, row 1).

3.2 Visual Flotation Response Dependent on Equilibration Time and Cleaning Solvent

Visual flotation observations were recorded at Day 1, Day 2, Day 3, and Week 1 for all crude oil–rock combinations and cleaning conditions. The following visual criteria are used to classify the wetting state for each liquid phase.

1) Oil-wet indicators: dark or amber coloration of the Isopar-L phase (residual crude oil leaching from the grain coating into the solvent), powder accumulating at or above the oil–water interface, grain clumping, and a diffuse or irregular oil-water interface.
2) Water-wet indicators: a colourless or near-colourless Isopar-L phase, grains sinking completely into the deionized water, a sharp and flat oil–water interface, and no floating material exist within both phases.

Note that the 'oil-wet indicators' here merely denote a state that is not fully water-wet, as no quantification of wettability, e.g., by contact angle, is measured at this stage.

3.2.1 Isopar-L Cleaning – least water-wetting state

Table 2 show the flotation tubes for Oil A, Oil B, and Oil-C-aged powder cleaned with Isopar-L (2 grams, Day 1 through Week 1).

Table 2. Aged powder flotation results (2 gr).

Crude Oil	Day 1	Day 2	Day 3	Week 1
No oil-Unaged Reference				
Oil A				
Oil B				
Oil C				

For Oil A at Day 1 (Table 2, row 2), the Isopar-L phase showed dark amber coloration, with a dark brown band beneath it. By Day 2, the floating mass has largely consolidated into a persistent dark brown horizontal band at the oil–water interface, with no visible migration downward into the water phase, while the oil phase retains a golden-amber color and the water phase below remains turbid. On Day 3 and Week 1, the dark interfacial band remains present. Grain clumping and incomplete settling persist through Week 1, and the oil–water interface remains poorly defined.

For Oil B at Day 1 (Table 2, row 3), the Oil B jars display the beige floating layer in the Isopar-L phase with a thick, massive aggregate of material sitting at and above the oil–water interface, with a milky-white water phase below. The oil phase at Day 1 is relatively light amber yellow. By Day 2, the large beige floating mass has partially reorganized into a combination of white interfacial material and large, irregular dark-brown clumped aggregates that sit at the interface. From Day 2 onwards, the significant dark-brown clumped masses remained at the interface, with the white material reduced but the interface still heavily populated and irregular. The oil phase remains light amber throughout.

For Oil C on Day 1 and Day 2 (Table 2, row 4), the oil phase is noticeably lighter than both Oil A and Oil B, a pale yellow-amber. For Oil C on Day 1 and Day 2 (Table 2, row 4), the oil phase is noticeably lighter than both Oil A and Oil B, showing a pale yellow-amber color. A band of brown floating material is present at the oil–water interface, and the water phase contains large, clearly visible brown granular particles distributed throughout the water phase rather than all settling to the bottom. From Day 3, this brown granular material becomes mixed with darker material, in which the water phase shows a heterogeneous distribution of dark particles. The overall appearance is less stratified and less cleanly separated than Oil A or Oil B.

The persistence of non-water-wet indicators across all three crude oils is consistent with the established

mechanism of crude oil wettability alteration, in which surface-active components from the crude oil adsorb to mineral surfaces and modify their wetting properties through multiple mechanisms, including polar interactions, acid–base interactions, and ion binding [13,14]. Since Isopar-L is a non-polar, low-aromatic solvent, it cannot disrupt these surface interactions or dissolve the adsorbed compounds responsible for the observed non-water-wet behaviour.

3.2.2 Hexane Cleaning

Effect of hexane solvent exposure method

Table 3 exhibit the flotation results for crude oil aged powder cleaned with hexane by filtration only over Day 1 through Week 1.

Table 3. Crude oil-hexane cleaned with filtration only (Hx+F)-results (2 gr).

Crude Oil	Day 1	Day 2	Day 3	Week 1
Oil A				
Oil B				
Oil C				

For the Oil A-hexane cleaned powder (Table 3) at Day 1 (a), the oil phase is amber orange colored, with a brown powder suspended at the oil–water interface and a dark brown sediment at the bottom, visually similar to the Isopar-L baseline (Table 2, Oil A). At Day 2, the floating particle in the oil-phase gradually accumulated on the interface, making the oil-phase clearer, with the water phase still turbid. By Week 1, the system shows more settled dark granular material both within and just below the oil phase, with a few small visible bubbles.

In Table 3, Oil B shows the most distinctive and persistent interfacial feature of the three crude oils. At Day 1, a large, consolidated dark brown-to-black mass is clearly visible at the oil–water interface, markedly darker and more consolidated than the equivalent Oil A feature, with beige/cream material in the lower water phase. This dark interfacial clump persists visibly through Day 2 and Day 3 and is still clearly present in Week 1. The beige/cream material in the lower water phase is gone by Week 1. The oil phase remains light amber throughout. The persistence and consolidated character of this dark interfacial mass in Oil B compared with Oil A is consistent with the established understanding that the extent to which oil components alter wetting depends strongly on crude oil composition [16].

Oil C shows the most visually distinct response of the three crude oils in Table 3. Across all four timepoints, the oil phase is a distinctly pale yellow, noticeably lighter than either Oil A or Oil B, indicating better removal of the colouring oil fraction by hexane filtration.

Across all three crude oils, none of the observed features approaches the water-wet reference in Table 2, as evidenced by persistent grain accumulation at the oil–water interface, a coloured oil phase, and incomplete grain settling, in contrast to the unaged reference, where all grains settled cleanly to the water phase. The reason is most likely to be related to asphaltene chemistry, which is defined as the fraction of petroleum insoluble in n-alkanes (typically heptane, but also hexane or pentane) but soluble in toluene [11,12]. This is consistent with the observation that toluene-based cleaning achieves significantly lower FI values compared to hexane, suggesting that asphaltenic surface coating remains after hexane cleaning.

Effect of additional mechanical agitation during cleaning

Table 4 show the flotation jars for Oil A (row 1), Oil B (row 2), and Oil C-aged (row 3) powder, cleaned with hexane by filtration, followed by mechanical shaking at 300 RPM from Day 1 through Week 1.

Table 4. Crude oil-hexane cleaned with filtration and shaking-results (Hx+FS) (2 gr).

Crude Oil	Day 1	Day 2	Day 3	Week 1
Oil A				
Oil B				
Oil C				

Under Hx+FS, mechanical agitation produced visible changes relative to Hx+F across all three crude oils. For Oil A in Table 4, the water phase was consistently cleaner and more settled, though dark interfacial material persisted through Week 1. Oil B in Table 4 showed the most striking response: a very large, consolidated dark brown-black mass floated within the oil phase at Day 1, progressively consolidating into a well-defined, rounded mass at the base of the oil phase by Week 1, with the water phase becoming translucent and clean. Oil C in Table 4 showed the largest change from Table 3, with the water phase noticeably cleaner at Week 1 compared with Hx+F, and most of the grains had settled to the bottom.

Across all three crude oils, mechanical agitation redistributed loosely associated material but did not visually restore water-wet conditions, suggesting that hexane's cleaning effectiveness is governed by its chemical nature as a solvent for non-asphaltene components rather than by the mode of application. The mechanisms by which crude oil components adsorb on mineral surfaces including polar, acid/base, and ion-binding interactions that can further alter surface wetting

[13,14], are therefore likely not disrupted by hexane, regardless of whether it is applied with or without agitation. The non-water-wet character of the grain surfaces therefore persists after Hx+FS in all three crude oil systems, and none of the three crude oils approaches the water-wet reference of Table 2, row 1.

3.2.3 Toluene and Methanol Cleaning

Effect of toluene and methanol solvent exposure method

Table 5 show the flotation jars for Oil A (row 1), Oil B (row 2), and Oil C-aged (row 3) powder cleaned with Toluene and Methanol by filtration only (TM+F) over Day 1 through Week 1.

Table 5. Crude oil-toluene and methanol cleaned with filtration only (TM+F)- results (2 gr).

Crude Oil	Day 1	Day 2	Day 3	Week 1
Oil A				
Oil B				
Oil C				

For the Oil A at day 1 in Table 5, the overall appearance is already different from hexane conditions, as the jar shows a warm yellow-brown oil phase in the upper portion, with most of the powder having settled to a large dark brown zone in the lower half. The oil–water interface is seen more as a gradient rather than a clean line, suggesting the system is still equilibrating. By day 2, a clearly defined, thin, dark brown band is visible at the oil–water interface, which is darker and more distinct than on day 1, while the lower zone lightens to a warm golden-amber. By Day 3, the oil phase above is becoming more transparent, and the thin, dark interfacial band persists, while the lower water phase becomes more translucent, and the powder has settled further to a compact, dark layer at the bottom. After one week, the system shows that most of the powder has already settled: a warm amber oil phase, a thin but still-visible dark residual band at the oil–water interface, a pale cream-coloured water phase below, and a compact dark brown powder sediment at the bottom.

Oil B in Table 5 shows a similar visual response as oil A at day 1, where a translucent condition is seen in the oil phase. Below it, the lower half is occupied by a dark brown zone. By day 2, some of the floated mass has largely dissipated, leaving a small amount of powder at the interface, and the system begins to separate into a clearer oil phase above and a dark interfacial band below. At the end of the week, the system has greatly settled, showing a translucent oil phase, and a clear pale amber-yellow water phase below.

From Table 5, Oil C at day 1, the oil phase is almost colourless, the lightest oil phase of all three crude oils and all cleaning conditions so far. The water phase below the interface is dark-coloured, with a compact, dark-brown powder layer at the bottom. At day 2, the oil phase remains translucent with only a small amount of beiger/cream material visible around the interface. By day 3, dark brown material is visible hanging downward from the oil–water interface in elongated structures extending into the water phase. By week 1, these structures persist.

Effect of additional mechanical agitation during cleaning

Similar as for cleaning with hexane, the effect of mechanical agitation under toluene–methanol cleaning was tested. Visual comparison of Table 5 and 6 shows smaller changes than for hexane, indicating a smaller effect of mechanical agitation.

Table 6. Crude oil-toluene and methanol cleaned with filtration and shaking (TM+FS)- results (2 gr)

Crude Oil	Day 1	Day 2	Day 3	Week 1
Oil A				
Oil B				
Oil C				

For Oil A and Oil B in Table 6, the visual appearance at week 1 is largely similar to TM+F, with a near-colourless oil phase, essentially complete grain settling, and only a very narrow residual interfacial band remaining. The most notable difference is observed for Oil C : the particles that hang downward from the oil-water interface in fibrous structures under TM+F in Table 5 are completely absent under TM+FS in Table 6, confirming that the mechanical agitation step mobilized and settled these loosely bound grains that resides in the oil-water interface. Overall, TM+FS produced the closest visual approximation to the fully water-wet reference across all three crude oils, with a clear water phase, near-colourless oil phase, and compact dark sediment at the bottom by week 1.

Comparison across all toluene and methanol conditions reveals a distinct change in cleaning effectiveness: all three crude oils show substantially improved phase separation relative to Hx+F and Hx+FS. This is consistent with the established roles of these two solvents in cleaning crude-oil-aged rock material, in which toluene can remove the hydrocarbons and non-polar/weakly polar organics [17]. At the same time, methanol is commonly paired with toluene in laboratory practice to remove polar organic and inorganic salts from the rock, which also can make it easier for toluene to

access the organics on the rock surface [15,16]. Furthermore, toluene is more effective here precisely because, in contrast to hexane, asphaltenes are soluble in aromatic solvents such as toluene [11,12].

3.3 Quantitative Flotation Index (FI)

So far, the tests have been analyzed qualitatively. But despite the visual criteria, irregular liquid-liquid interfaces and emulsion formation limit accurate comparison of the samples.

Table 7 and Figure 2 summarize for the cases described in Section 3.2 the quantification, providing the Flotation Index (FI) values for all cleaning conditions and crude oils derived from gravimetric measurements of the float and sink fractions after one week, calculated according to Equation 1.

In addition to the FI, we also consider the grain recovery factor RF as given by:

$$RF = \frac{m_{float}+m_{sink}}{m_{initial}} \quad (2)$$

Here $m_{initial}$ is the mass added to fluids at the start of the flotation experiments.

The error to the FI, arising from incomplete mass recovery, is estimated by considering the two bounding cases for the unrecovered mass Δm, defined as:

$$\Delta m = m_{initial} - (m_{sink} + m_{float}) \quad (3)$$

If all unrecovered mass is assigned to the sink fraction, the FI value is minimized, and if it assigned to the float fraction, FI value is maximized. The bounds are therefore:

$$FI^{-} = \frac{m_{float}}{m_{initial}} \quad (4)$$

$$FI^{+} = \frac{m_{float+\Delta m}}{m_{initial}} \quad (5)$$

Note that this approach leads to an uneven error distribution around the calculated FI. When the float fraction is smaller than the sink fraction, the upper boundary is relative (and absolutely) further from FI than the lower boundary. The error itself therefore needs to be used with caution. If considering the repeatability of the experiments, the deviation generally shows a larger spread as discussed in the next section, though this has not been tested for all data.

Table 7. Summary of FI values (%) of 2 grams sample at week 1 for all conditions, with the upper and lower estimation of FI using Equations 4 and 5 in brackets.

Metric	Oil A		Oil B		Oil C	
	FI (%)	RF (%)	FI (%)	RF (%)	FI (%)	RF (%)
Isopar-L (Aged)	**8.58** (6.92-26.36)	80.6	**11.50** (9.21-29.18)	80	**9.08** (7.59-24.02)	83.6
Hx+F	**7.43** (7.23-9.97)	97.3	**5.44** (5.33-7.38)	98	**16.83** (16.54-18.25)	98.3
Hx+FS	**6.97** (6.77-9.63)	97.1	**11.89** (11.58-14.22)	97.4	**1.61** (1.56-5.10)	96.5
TM+F	**0.32** (0.31-2.63)	97.7	**1.08** (1.06-3.17)	97.9	**1.40** (1.37-3.62)	97.8
TM+FS	**0.23** (0.23-1.39)	98.8	**0.43** (0.43-1.71)	98.7	**0.82** (0.81-2.39)	98.4

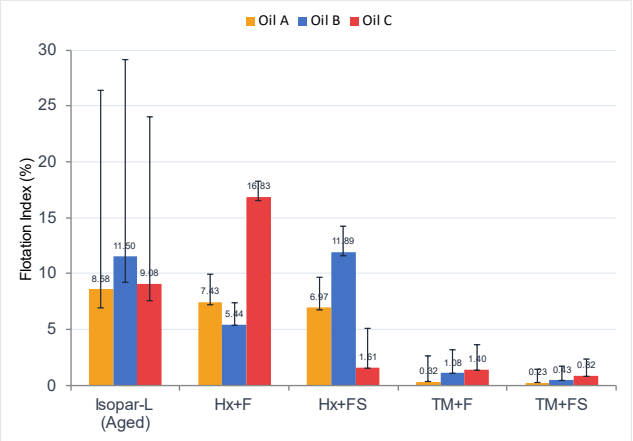

Fig. 2. Summary of FI values (%) of 2 grams sample at week 1 for all conditions.

3.3.1 Isopar-L (aged baseline)

Under Isopar-L cleaning, all three-crude oil–rock pairs at 2 grams yield FI values of 8.58–11.50%, substantially above the unaged fully water-wet reference (FI ≈ 0%, as established in Section 3.1), confirming a wetting state different from the fully water-wet state across all three crude oils. The lower RF values under Isopar-L (RF ≈ 80%) compared to all other conditions (RF > 96%) reflect residual crude oil retained on the grain surfaces after Isopar-L rinsing, which contributes to the initial powder mass but is lost upon drying of the recovered fractions. However, the exact mass of retained oil was not independently quantified in this study, and the magnitude of this mass loss is non-negligible and eventually drives the high upper uncertainty bounds (upper FI uncertainty up to ~29%) reported for Isopar-L conditions. This level of uncertainty limits the reliability of FI as a quantitative wettability indicator under Isopar-L conditions and reducing mass loss through improved cleaning or drying protocols, and removing fine particles that require extended sedimentation times, are therefore important steps toward more precise FI measurements.

The relatively low float fraction, with at most 12% of grains partitioning to the oil phase at 2 grams even under the most non-water-wet conditions, could reflect either (i) a mixed-wet character of the grain population, (ii) the competition between surface energy and gravitational settling at the 50–74 µm grain size, or the potential preferential wetting of the clay mineral sub-fraction rather than the bulk powder. The relatively low float fraction, with at most 12% of grains partitioning to the oil phase at 2 grams even under the most non-water-wet conditions, could reflect either (i) a mixed-wet character of the grain population, with expected contact angles still below the ~90° threshold for preferred water-phase partitioning, (ii) the competition between surface energy and gravitational settling at the 50–74 µm grain size, or the potential preferential wetting of the clay mineral sub-fraction rather than the bulk powder. Given the overlapping error ranges (Oil A: 8.58 (6.92-26.36) %; Oil B: 11.50 (9.21-29.18) %); Oil C: 9.08 (7.59-24.02) %, no significant ranking of crude oil wettability alteration potential can be established from these FI values alone.

These relative high FI values are consistent with the visual observations (Table 2), which showed that at week 1 all three jars, showed a dark or amber-coloured oil phase, grain clumping and grain accumulation at the oil–water interface, and no clean grain settling comparable to the known water-wet reference (Table 2) criteria, identified therefore as non-water-wet indicators. The relatively narrow spread across the three crude oils after Isopar L cleaning (2.92%) suggests that the Isopar-L pre-rinse removes a similar proportion of loosely bound bulk oil regardless of crude oil composition. However, given the overlapping error ranges and the theoretical threshold contact angle above which grains preferentially partition to the oil phase, the FI alone cannot fully resolve gradations in wettability between the three oils, a limitation that could be addressed by grouping grains according to multiple critical contact angle thresholds tied to specific mineral wettabilities.

3.3.2 Hexane – Filtration only (Hx+F)

Under Hx+F at 2 grams, the FI values diverge substantially across the three crude oils: Oil A decreases to 7.43% (-1.15 percentage points from the Isopar-L aged baseline), Oil B decreases to 5.44% (−6.06 %), while Oil C increases from 9.08% to 16.83%, an 85% increase relative to its Isopar-L baseline.

For Oil A and Oil B, the FI decreases are consistent with the partial removal of the loosely bound fraction of the crude oil. However, both values remain above 5%, and the visual appearance confirms that both systems remain in a state that is not fully water-wet: dark asphaltic chunks persisted at the oil–water interface through week 1 for Oil A in Table 3, and a large consolidated dark clump persisted prominently at the interface for Oil B in Table 3. The Oil B FI decrease of 6.06 %, therefore, reflects the removal of the larger initial, loosely bound non-asphaltene mass in Oil B crude.

The Oil C Hx+F result, which yielded FI 16.83%, is higher than the Isopar-L baseline, which is the most anomalous result in the entire dataset. No clear explanation can be given at this stage and further investigation is needed.

3.3.3 Hexane – Filtration and Shaking (Hx+FS)

Under Hx+FS, the FI values show divergent behavior: Oil A decreases to 6.97% (−0.46 % from Hx+F), Oil C decreases sharply to 1.61% (−15.22 % from Hx+F), while Oil B increases to 11.89% (+6.45 % from Hx+F).

From the visual observations in Table 4, the marginal FI decrease in Oil A is matched by only a slight visual improvement, a cleaner water phase, but persistent dark interfacial material, confirming incomplete and very limited cleaning.

In the case of Oil B, the rise in FI despite mechanical agitation is reflected visually in Table 4 by the continued presence of a large, consolidated, dark mass at the interface throughout week 1. Agitation with a non-polar solvent could have redistributed the material rather than removing more oil coating, however it can also reflect the uncertainty in the FI determination.

Meanwhile, for Oil C, the sharp FI drop, both compared to Isopar L or Hx+F, indicates a more effective removal of loosely bound non-asphaltenic components through the mechanical agitation during the Hx+FS cleaning step, which reduced the interfacial powder accumulation observed under Hx+F. Nevertheless, the oil phase remains pale yellow throughout (consistent with the visual appearance in Table 4) and does not approach the near-colourless state of the water-wet reference, confirming that the asphaltenic surface coating remains intact and water-wet conditions are not restored.

3.3.4 Toluene and Methanol — Filtration only (TM+F)

TM+F produces a reduction in FI across all three crude oils: Oil A drops from 7.43% (Hx+F) to 0.32% (a reduction of 7.12 %, approximately 23-fold), Oil B drops from 5.44% to 1.08% (a reduction of 4.36%, approximately 5-fold), and Oil C drops from 16.83% to 1.40% (a reduction of 15.43%, approximately 12-fold). All three values fall below 1.5%, approaching the water-wet reference, with the maximum FI decreasing from 16.83% to 1.40% across the three crude oils, confirming the effectiveness of harsh cleaning (toluene and methanol) in shaping the FI response.

The visual observations (Table 5) support these near-zero FI values. For all three crude oils, most grains settled to the water phase, and the oil phase became creamy beige to pale. However, a residual dark band was still visible at the interface for all three crude oils after week 1, which from the visual observations in Table 5 was found to be thinnest for Oil A, more prominent for Oil B, and most persistent for Oil C. This residual band is consistent with the non-zero FI values, indicating that a small fraction of surface-active material was not fully removed by filtration alone, producing a small residual float fraction.

3.3.5 Toluene and Methanol — Filtration and Shaking (TM+FS)

TM+FS yields a further reduction in FI relative to TM+F: Oil A 0.23% (−0.09 %), Oil B 0.43% (−0.65 %), Oil C 0.82% (−0.58 %). The relative spread in FI values across the three crude oils stands at a ratio of 3.57 under TM+FS (0.82/0.23), the smallest of all conditions tested.

Visually (Table 6), TM+FS produced the closest approximation to the water-wet reference (shown in Table 2) across all conditions. The oil phase was nearly colourless for all three crude oils, grain settling was essentially complete, and the residual dark interfacial band observed under TM+F was reduced to a very narrow line. The residual FI values are below 1%, corresponding to these very slight residual visual features, confirming that near-water-wet conditions are achieved under TM+FS. This is consistent with the finding that strong solvents used in core cleaning can remove material from the rock phase, leading to more water-wet behaviour, but not all adsorbed polar organic compounds are removed [14,15].

3.4 Experimental Repeatability

For the purpose of assessing the consistency of the flotation response across repeat experiments, identical

runs were performed with 1-gram samples applying the same protocol conditions. Repeatability was tested for Isopar L treated grains and Toluene-Methanol with filtration only (TM+F). Table 8 presents the results on selected conditions at 1 gram, where so far 2 grams data was presented. The effect of sample weight is addressed in Section 3.5.

3.4.1 Visual Observations

Repeatability tests closely reproduced the original visual features for all three crude oils under Isopar-L conditions, as shown in Table 8 and Figure 3.

Table 8. Repeatability test of crude oil-aged flotation results (1 gr) and crude oil-toluene and methanol cleaned with filtration only.

A. Isopar L					
Oil A		**Oil B**		**Oil C**	
Exp 1	**Exp 2**	**Exp 1**	**Exp 2**	**Exp 1**	**Exp 2**
					
B. TM+F					
Oil A		**Oil B**		**Oil C**	
Exp 1	**Exp 2**	**Exp 1**	**Exp 2**	**Exp 1**	**Exp 2**
					

From Table 8A, Oil A showed the same branching, dispersed dark material in the water phase; Oil B reproduced the large consolidated dark brown-black interfacial mass in both size and persistence; and Oil C showed the same pale-yellow oil phase with granular white material distributed in the water phase, confirming visual reproducibility across all three systems.

For TM+F repeatability, all three crude oils (Table 8B) reproduced the near-water-wet appearance observed in the original experiments, confirming the consistency of the TM+F condition.

3.4.2 Quantitative FI

Table 9 and Figure 3 summarize FI values for the samples shown in repeatability test. The FI average is the mean of the 2 runs, with the deviation based on the data spread around the average.

Table 9. FI values (%) in Week 1 for repeatability for 1 gram sample, for Isopar-L cleaning and TM+F cleaning.

Repeatability Test				
Condition	**Run**	**Oil A (%)**	**Oil B (%)**	**Oil C (%)**
Isopar-L (Aged)	Run 1	**3.02** (2.45-21.21)	**28.12** (22.05-43.64)	**12.06** (10.36-24.44)
	Run 2	**2.54** (2.11-19.01)	**11.16** (9.27-26.24)	**13.18** (11.16-26.5)
	FI Average ± max dev	2.78 ± 0.24	19.64 ± 8.48	12.62 ± 0.56
TM+F	Run 1	**0.15** (0.15-1.29)	**0.63** (0.62-1.98)	**0.87** (0.86-1.45)
	Run 2	**0.23** (0.22-2.54)	**0.76** (0.74-2.99)	**1.18** (1.15-3.45)
	FI Average ± dev	0.19 ± 0.04	0.69 ± 0.06	1.02 ± 0.16

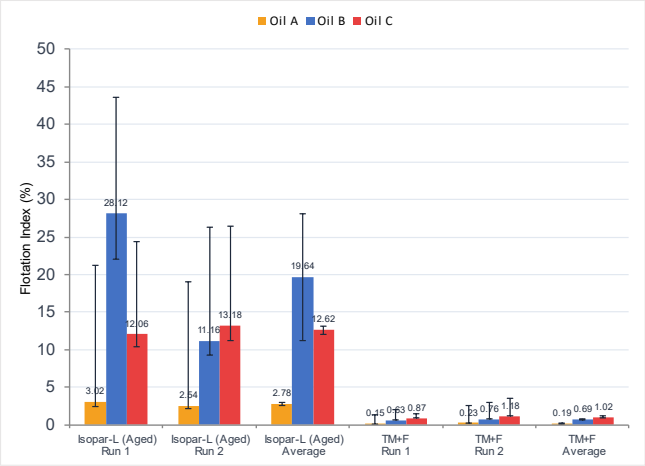

Fig. 3. Comparing two runs of FI values (%) after week 1 for 1 gram sample

For the Isopar-L aged condition, Oil A and Oil C show good consistency: Oil A yields 3.02 (2.45-21.21) % and 2.54 (2.11-19.01) %; and Oil C yields 12.06 (10.36-24.44) % and 13.18 (11.16-26.5) %, with averages of 2.78 ± 0.24 and 12.62 ± 0.56 respectively. Oil B, however, shows a large spread of 28.12% and 11.16%. This likely reflects the uncertainty due to presence of a consolidated dark clump, which may have been unevenly distributed between the float and sink fractions during extraction. This highlights the importance of repeat runs and a consistent extraction methodology to ensure reproducibility, particularly for conditions where large interfacial aggregates are present.

For TM+F, all three crude oils reproduce similar values, with absolute FI differences below 0.32% (Oil A: 0.15% vs 0.23%; Oil B: 0.63% vs 0.76%; Oil C: 0.87% vs 1.18%). Although the deviations between runs are non-negligible relative to the FI values themselves (Oil A: 0.19 ± 0.04%; Oil B: 0.69 ± 0.06%; Oil C: 1.02 ± 0.16%), all values remain below 1.5% and approach the unaged fully water-wet reference (FI ≈ 0%), confirming that the method reliably classifies the wetting state as restored toward water-wet conditions.

The variability in FI across repeat runs also raises a broader question regarding the powder's homogeneity. Bleurswiller sandstone consists of more than two-thirds quartz, close to one-third with feldspar, and few percentages of clay minerals, and mica. Despite the relatively low clay content, clay minerals exhibit disproportionately greater surface reactivity toward polar crude oil components than quartz and feldspar. Consequently, the float fraction, which represents only 8–12% of total grain mass, may be selectively enriched in clay minerals rather than representing a homogeneous

subsample of the bulk powder. If that is the case, run-to-run variability in FI may partly reflect variability in clay mineral distribution within the small mass of powder used (1 gram), rather than a purely procedural issue. This further underscores the need to characterize float and sink fractions by XRD or SEM in future work.

3.5 Sensitivity Study

3.5.1 Mass Sensitivity

Under the assumption that gravitational forces are limited, experiments using different sample sizes shall show repeatable results. However, if sedimented particles are stacked in the oil phase, the exerted gravitational forces could counteract the interfacial forces holding oil-preferring particles in the oil phase. As the area of the containers remain constant, the column of stacked particles increases with increasing mass, causing gravitational forces to increase, potentially leading to a higher percentage of grains in the water phase. Therefore, a mass sensitivity study was conducted to assess how reducing the powder mass from 2 grams to 1 gram would affect the flotation response.

Three representative cleaning conditions were studied, which consist of Isopar-L, Oil C Hexane and filtration (Hx+F), and Toluene and Methanol filtration only (TM+F). Their visual observations are illustrated in Table 10.

Table 10. Visual comparison of flotation results of (A) aged powder and (B) hexane with filtration (Hx+F) and (C) toluene and methanol with filtration only after 1 week for 1 and 2 grams.

A. Isopar-L			
Mass	**Oil A**	**Oil B**	**Oil C**
1 g			
2 g			
B. Hx+F			
1 g	-	-	
2 g	-	-	
C. TM+F			
Mass	**Oil A**	**Oil B**	**Oil C**
1 g			

2 g				

The corresponding estimated FI for the 1 and 2-grams sensitivity study is shown in the Table 11 and Figure 4.

Table 11. FI values (%) at week 1 for mass sensitivity experiments, with errors determined according to Equation 4 and 5.

Condition	**Mass (g)**	**Oil A (%)**	**Oil B (%)**	**Oil C (%)**
Isopar-L (Aged)	1	**2.78** (2.54-3.02)	**19.64** (11.16-28.12)	**12.62** (12.06-13.18)
	2	**8.58** (6.92-26.36)	**11.50** (9.21-29.18)	**9.08** (7.59-24.02)
	FI Average ± max dev	5.68 ± 2.90	15.57 ± 4.07	10.85 ± 1.77
Hx+F	1	-	-	**26.31** (24.87-30.35)
	2	-	-	**16.83** (16.54-18.25)
	FI Average ± dev	-	-	21.57 ± 4.74
Hx+FS	1	**6.20** (6.09-7.9)	**11.44** (11.25-12.95)	**2.02** (1.97-4.37)
	2	**6.97** (6.77-9.63)	**11.89** (11.58-14.22)	**1.61** (1.56-5.10)
	FI Average ± dev	6.59 ± 0.38	11.67 ± 0.22	1.82 ± 0.20
TM+F	1	**0.19** (0.15-0.23)	**0.69** (0.63-0.75)	**1.02** (0.86-1.18)
	2	**0.32** (0.31-2.63)	**1.08** (1.06-3.17)	**1.40** (1.37-3.62)
	FI Average ± dev	0.26 ± 0.07	0.89 ± 0.20	1.21 ± 0.19

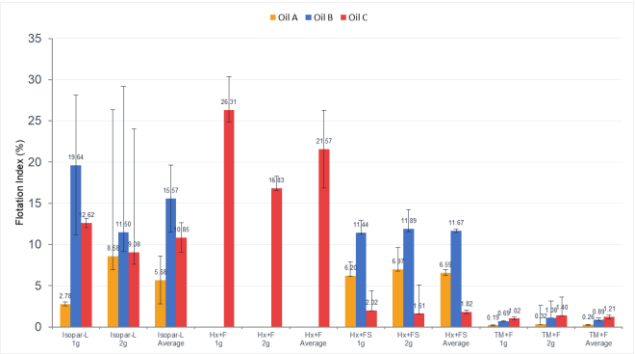

Fig. 4. Summary of FI values (%) in mass sensitivity study.

For the Isopar-L condition, FI for Oil A decreases from 8.58% at 2 grams to 2.78% at 1 gram, a reduction of 5.80 %. For Oil B the FI increases from 11.50% at 2 grams to 19.64% at 1 gram, a difference of 8.14 %, making this the most substantial mass-dependent FI change observed across all conditions tested. Meanwhile, for Oil C, FI changes from 9.08% at 2 grams to 12.62% at 1 gram, an increase of 3.54.

In Hx+F case, mass sensitivity data is available only for Oil C, yielding FI values of 26.31 (24.87-30.35) % at 1 gram and 16.83 (16.54-18.25) % at 2 grams, with an average of 21.57 ± 4.74%. The increase in FI from 2 grams to 1 gram (9.48%) follows the same trend observed under Isopar-L-aged conditions, where reducing the powder mass increases the proportion of grains reporting to the oil phase under non-water-wet conditions (with the

exception of Oil A). This is consistent with the gravitational argument outlined above: at lower powder mass, the reduced column height decreases the gravitational load on oil-preferring particles, allowing a greater fraction to be retained in the oil phase.

For Hx-FS, the 1-gram measured yield FI values of 6.20 (6.09-7.9) %, 11.44 (11.25-12.95) %, and 2.02 (1.97-4.37) % for Oil A, Oil B, and Oil C respectively, compared to 6.97 (6.77-9.63) %, 11.89 (11.58-14.22) %, and 1.61 (1.56-5.10) % at 2 grams, yielding averages of 6.59 ± 0.38 %, 11.67 ± 0.22 %, and 1.82 ± 0.20 %. Mass recovery exceeds 97.6% for all three oils at both masses. For HX-FS we observe small mass-dependent differences, all within 0.77 percentage points. This is in contrast to the strong mass dependence observed under Isopar-L aged conditions.

Lastly, considering the TM-F, the 1-gram system for oil A at week 1 is less cleanly settled and shows a more complex distribution of residual material, compared to the 2-gram equivalent. The FI decreases from 0.32% at 2 grams to 0.19% at 1 gram, a reduction of 0.13%. For Oil B at week 1, the 1 gram sample shows a lighter color in the water phase compared to the 2 gram sample. The FI changes from 1.08% at 2 grams to 0.69% at 1 gram, a reduction of 0.39%. Compared with TM+F at 2 grams, the water phase became clearer from day 2, suggesting slightly more complete dissolution of the asphaltic coating at lower powder mass. FI changes from 1.40% at 2 grams to 1.02% at 1 gram, a reduction of 0.38 %. Under TM+F conditions, both 1-gram and 2-gram samples produce FI values approaching the unaged fully water-wet reference (all below 1.5%), with a maximum difference of ≤0.39% across all three crude oils. Since this difference is smaller than the inter-run variability observed under the same conditions, the FI can be considered mass-insensitive in this regime.

Summarizing, the variability observed under the tested mass changes are in the same order as the variability observed from repeatability, except for the Isopar-L samples. There might be indications that FI is reduced with increased mass under oil-wet conditions (Isopar-L aged), consistent with an explanation that the interface cannot hold the additional mass due to the gravitational pull. Under more water-wet conditions (Hx+FS and TM+F), mass-dependent differences are substantially smaller.

These results suggest that under oil-wet conditions, gravitational forces are not negligible relative to the interfacial forces that hold oil-preferring particles in the oil phase. Thus, this represents a limitation of the method under non-water-wet conditions where FI does not completely reflect the surface affinity when gravitational settling competes with capillary retention. Hence, powder mass must be standardized and reported for any comparative interpretation of results.

Here only two masses were tested. More tests are needed to find the optimal sample mass, where it needs to be noted that also particle size affects the influence of gravitational forces, which was here not a variable.

3.5.2 Fluid Addition Order

As explained in the Methodology Section 2.5, when setting up the flotation experiment, the powder was first introduced to Isopar L and subsequently deionized water was added before agitation. The effect of changing the fluid addition order were tested on a 1 gram sample by reversing the standard sequence: the powder was introduced to deionized water first, followed by Isopar-L.

Table 12 shows a visual comparison of the test after 1 week for Isopar L, and Table 13 provides the FI for Isopar L.

Table 12. Visual observations of the fluid addition order test of crude oil-aged flotation results (1 gr), after one week, with W-O representing introduction to water (W) first and thereafter oil (O) and reverse for O-W.

Condition	Fluid Order	Oil A	Oil B	Oil C
Isopar-L	W→O			
	O→W			

Table 13. Fluid addition order test of crude oil-aged flotation results (1 gr), after one week, with W=water, O=oil.

Fluid Addition Order Test				
Condition	**Fluid Order**	**Oil A (%)**	**Oil B (%)**	**Oil C (%)**
Isopar-L (Aged)	W→O	**3.66** (3.07-19.27)	**12.94** (10.79-27.38)	**14.47** (11.88-29.8)
	O→W	**2.54** (2.11-19.01)	**11.16** (9.27-26.24)	**13.18** (11.16-26.5)
	FI Average ± dev	3.10 ± 0.56	12.05 ± 0.89	13.83 ± 0.65

For the fluid-addition order experiments, considering the W-O sequence, Oil A and Oil B maintained their qualitative non-water-wet visual character regardless of which phase contacted the powder first. Oil C showed a slight difference in the water phase; the W-O sequence is cleaner and with only a thin compact sediment layer, suggesting that prior contact with water may have altered the distribution of fine material, although the system remains clearly non-water wet.

For all three crude oils, FI values are largely independent of fluid addition order, as evidenced by the small deviations between W→O and O→W protocols: 1.12% for Oil A (3.10 ± 0.56%), 1.78% for Oil B (12.05 ± 0.89%), and 1.29% for Oil C (13.83 ± 0.65%). While the visibly cleaner water phase is observed for Oil C under the water-first protocol (Table 12), given the non-significant FI difference, this visual difference cannot be confirmed as a systematic effect and requires further investigation.

Given the wide and overlapping error ranges across all three oils, and comparable to the repeatability experiments for Isopar L in Table 9, none of these differences is considered statistically significant. The

fluid-addition order is therefore concluded to have a limited effect on FI, confirming that the standard protocol is robust to the sequence in which the two immiscible phases contact the powder.

4 Conclusions

This work presents a study for development of a standardized powder-flotation protocol for wettability screening of crude-oil-aged sandstone and demonstrated its sensitivity to solvent-cleaning effectiveness. Four conclusions are drawn accordingly:

Method execution

1) The method separates grains into oil- and water-wet fractions based on their surface wetting preferences. The float fraction under Isopar-L aged conditions, expected to be the most non water wet state, represents only 8–12% of total grain mass at 2 grams, Either this is suggesting that despite their low bulk abundance, the disproportionately surface-reactive clay minerals may selectively enrich the oil-wet float fraction rather than representing a homogeneous subsample of the bulk powder, or that that this wetting state still prefers the water phase over the oil phase.
2) Repeated tests indicate significant uncertainty, especially for the less water-wet samples. This uncertainty arises due to several causes: (i) not all grains partition into their preferred wetting phase as some grains may remain trapped at the interface, or sink due to gravitational forces exceeding the capillary forces; (ii) emulsion existence at the oil–water interface could trap grains regardless of their wetting preference; (iii) mass determination of the fractions is affected by oil dissolved in the Isopar-L, since the start weight is recorded before drying, as well as by particle loss during transfer. However, despite this uncertainty, the FI value is able to distinguish between different wetting states for the tested samples.
3) For less water-wet samples, our 1- and 2-gram measurements could indicate a trend of lower FI for higher mass, however, this trend is obscured by the variability observed with repeated tests. Thus, powder mass must be standardized between tests and reported.
4) Fluid addition orders appear within the measurement error not to have a significant influence.
5) Visual oil- and water-wet indicators contribute to identification of the wetting preference.

Cleaning procedure

1) Non-polar solvents are insufficient for restoration to fully water wet conditions: Isopar-L and hexane, whether applied by filtration alone or combined with mechanical shaking, left nearly all FI values above 5% and showed persistent oil-wet visual indicators through week 1.
2) Toluene–methanol is the only protocol that approaches water-wet conditions, reducing FI by approximately one order of magnitude across all crude oils and yielding residual FI values below 1%, confirming near water-wet conditions.
3) Adding mechanical agitation with only 15 mL additional solvent compared to the vacuum filtration protocol shows that the method of exposure matters. This is most clear for ‘mild’ cleaning solvents like Isopar and hexane, while less clear for hard solvents like the toluene-methanol combination where results are close to water-wet.
4) The crude oil composition of the three used oils governs distinct flotation responses: Oil B produced the most prominent and random occurring variable grain clumping, indicative of a higher concentration of surface-active components; Oil C produced an anomalous response under hexane cleaning where insufficient removal of surface-active components by hexane left grains in partially wetted state, causing them to stabilize more at the oil-water interface; and Oil A demonstrated the most reproducible and mass-insensitive behavior across all conditions.

Future work recommendation

The present study demonstrates that FI distinguishes non-water-wet from near-water-wet states, but does not establish a reliable quantitative relationship between FI and other established wettability measurements, such as CA. Future work should therefore focus on three interconnected directions:

1) A direct calibration of FI against contact angle should be established. Homogenous material with known established wettabilities based on artificial coating can be used as wettability references to identify the critical contact angle threshold above which grains preferentially partition to the oil phase and to assess whether FI can resolve intermediate wetting states within the same wettability class. Further benchmarking against established wettability indices such as Amott and USBM is recommended to further support the interpretation of FI in terms of wettability variations.
2) The method should be extended to mixed-wet systems, where grains of different wettabilities are combined in controlled proportions. This would allow assessment of whether FI can quantify fractional wettability in a mixed grain population and establish a link between the float fraction and the proportion of oil-wet grains.
3) Characterization of the float and sink fractions by XRD or SEM-EDS is recommended to determine whether the oil-wet fraction in Bleurswiller sandstone is selectively enriched in clay minerals, which would clarify whether FI reflects bulk grain wettability or the wettability of a mineralogically selective sub-fraction.

Acknowledgements

This work was produced with support from the project “TeSCAL: Testing the foundations of special core analysis” project under grant number 352883 funded by the Research Council of Norway and industry partners (Equinor and AkerBP). The authors would like to

sincerely thank Jørgen Furuset Lie and Ole Støren for their support in the Reservoir Laboratory in SINTEF Industry.

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