Evaluation of relative permeability using the multi-flow method in carbonate rocks with highly heterogeneity considering the preservation of wettability through a mild cleaning process

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Abstract. This study presents a mild cleaning procedure developed for carbonate rocks from the Brazilian Pre-salt, whose representative facies were used in relative oil/water permeability tests. Traditionally, core cleaning for flow property characterization is performed using solvents such as toluene and methanol, primarily to eliminate contaminants introduced during coring operations. While alternative procedures for wettability restoration have been proposed mostly applied to outcrop rocks these methods often show limited effectiveness when used on actual reservoir samples, particularly regarding preserving initial wettability. This limitation underscores the need for improved methodologies to generate reliable data in reservoir characterization workflows. The proposed mild cleaning method aims to preserve the wetting properties of carbonates reservoir samples during relative permeability measurements. The procedure's effectiveness was evaluated by analyzing the effluents collected during fluid injection stages and by comparing the relative oil permeability values at different points throughout the restoration and cleaning process. The results showed that, regardless of the aging method employed, the relative oil permeability remained consistent. This consistency is attributed to the successful preservation of wettability achieved using the proposed cleaning approach.

1 INTRODUCTION

Conventional rock cleaning is typically carried out following the API RP-40 standard [3], often using solvents such as toluene and methanol. Several studies have proposed methods to prepare and restore core samples for forced displacement tests, oil recovery, and, in practical terms, for formation characterization [7, 8, 4]. However, the treatment of reservoir rocks becomes even more complex due to the intricate mineralogy and high heterogeneity among the various facies that may be found within reservoirs. Wettability, in addition to being highly relevant for reservoir characterization, is one of the key factors influencing the recoverable oil volumes during exploration campaigns [5].

Wettability in carbonate reservoirs has been one of the main topics of research in recent years. Numerous approaches have been proposed to address wettability restoration and its alteration when different fluids are used in oil recovery experiments [12]. However, in terms of wettability preservation, few studies are available, since during routine characterization workflows, it has not traditionally been considered a relevant parameter for relative permeability evaluation or for understanding wettability-related effects [8].

During formation characterization, experimental data is of critical importance. These data help define production behavior throughout a reservoir's lifecycle and support timely predictive modeling. One such key dataset is the generation of relative permeability curves in the laboratory [6,9]. There are various methods to obtain such data, but when it comes to the Brazilian pre-salt carbonate rocks, this has been one of the most challenging endeavors [2, 10,11].

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This work proposes an alternative to the traditionally used solvents in the rock cleaning process, replacing them with low-aromatic hydrocarbon compounds such as pentane, hexane, heptane, and aviation kerosene for the removal of organic components [7], and water with controlled ionic composition for the removal of inorganic components, implementing what is referred to as the Mild Cleaning approach. For this purpose, outcrop samples of Indiana Limestone were used to assess possible chemical reactions that may alter pore structures, while also aiming to develop faster and more cost-effective cleaning techniques, and compare their impacts with those of conventional methods.

The experimental campaign was divided into two main stages: in the first stage, outcrop samples were saturated with formation water from a pre-salt reservoir, followed by injection of multiple pore volumes of formation water diluted 1:1500. The effluents were collected for ion chromatographic analysis to track the evolution of the cleaning process, as well as potential increases in ion concentrations that could be linked to chemical reactions within the porous system. After gathering this data, petrophysical properties of the same cores were measured to assess any variation resulting from the procedure. This methodology was repeated a second time to validate the initial results.

The next step involved preparing the rock samples at initial water saturation using pre-salt formation water and crude oil. All samples were aged in a cell for 30 days at the reservoir temperature of 60°C. Oil recovery was measured by coreflooding, after which each core underwent a new cleaning cycle. In this stage, the same fluid as previously used was applied for removing the inorganic phase. For removal of the oil phase, n-hexane was injected until no further fluorescence was detected, indicating the absence of residual hydrocarbons. The mild cleaning process concluded with differential pressure measurements at three different flow rates to determine the relative permeability of the oil phase.

After validating the methodology using outcrop samples, two representative reservoir rock types—Stromatolite and Grainstone—were selected and prepared using the same procedure, as they represent typical facies of Brazilian pre-salt reservoirs. The results obtained from the outcrop material indicated no chemical reaction due to the injection of diluted formation water. Additionally, the relative variation in calcium and magnesium concentrations—key constituents of the rock matrix showed low reactivity. This finding was supported by porosity and permeability measurements at each stage of the process.

For the reservoir samples, results indicated the need for a higher volume of diluted formation water injection. This could be attributed to the high heterogeneity of these samples, which may lead to the formation of preferential flow paths during solvent injection. For these cores, effluent pH was also monitored throughout the injection of several pore volumes to track clay activity and identify potential variations in chemical reactivity or pressure drop behavior.

2 Material and Methods

The mild cleaning process was designed to preserve the wettability of reservoir samples while avoiding alterations to their petrophysical properties. Two critical parameters guided the design: the rock's reactivity to different fluid compositions (e.g., seawater, diluted formation water) and the compatibility of solvents used for organic phase removal.

To achieve these goals, two fluids were selected. For the aqueous phase removal, formation water diluted 1500 times with deionized water was used. For the organic phase, n-hexane was chosen due to its ability to extract hydrocarbons while minimizing reactivity with the rock matrix.

Initially, a calibration curve was developed to correlate the electrical conductivity of the diluted formation water with its concentration (Fig.1). This curve was used during the mild cleaning process to monitor salinity reduction in the effluents over time, thereby confirming the flushing efficiency.

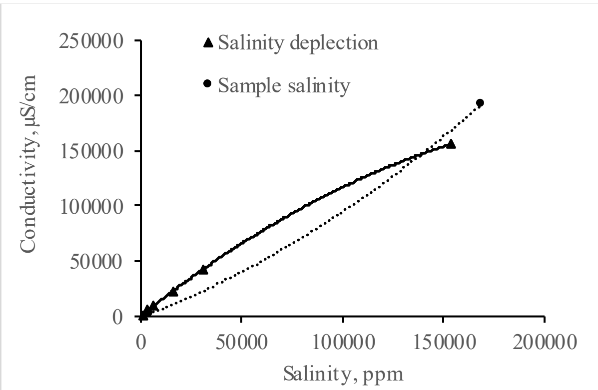

Figure 1 – Calibration curve for fluid effluents

2.1 Preparation of Formation Water and Desulphated Seawater

Based on the ionic composition of formation water and seawater, the required quantities of each salt were calculated to reproduce the fluids in the laboratory. Table 1 presents the ionic composition used in the experiments.

Table 1 – Ionic composition for formation water and seawater desulphated, (ppm)

	FW	SWd
NaCl	154,954	31,467
CaCl2x2H2O	84,108	5,402
MgCl2x6H2O	35,581	0,276
KCl	6,046	0,848
BaCl2x2H2O	0,898	-
SrCl2x6H2O	17,289	-
LiCl2	0,547	-
KBr	2,085	0,107
NaHCO3	0,324	0,059

2.2 Oil Characterization and viscosity adjustment

For the oil characterization stage, the procedures described in the ASTM D7752-18 standard were utilized. The first activity involved collecting a representative sample, for which a container was heated to a temperature of 58°C for 8 hours. After this period, smaller samples were collected in 1000 ml containers, which were used for characterization tests and other activities.

2.2.1 Density and Viscosity

Table 2 presents the values of viscosity and density as a function of temperature, where the results validate the data provided by Petrobras. With this data, it is possible to identify that the heating process did not produce significant variations in the density of the sampled oil.

Table 2 – Oil Properties

Temperature	15.6	20.0	60.0	**°C**
Viscosity	47.0	33.5	6.3	**mPa.s**
Density	0.88	0.87	0.84	**g/cm3**

2.2.2 Physicochemical Properties

The assessment of oil properties was fundamental to understanding the adsorption dynamics of polar components in the evaluated rocks. To this end, Table 3 presents the acid and base indices of the oil, which are the most relevant factors for an aging process. A representative oil sample was collected, and the measured values for the fractions of saturates, aromatics, paraffins, and asphaltenes (SARA) are also presented in Table 3.

Table 3 - Characterization of SARA fractions and polar compounds of crude oil

	Sat	Arom	Res	Asph
Oil	57.4	24.8	16.2	0.2
TAN	0.23			
TBN	3.16			

2.3 Sample Selection and preparation

For this study, two types of samples were selected. The first group consisted of outcrop rocks of the Indiana limestone type, and the second group comprised rocks whose facies are representative of the Brazilian pre-salt reservoirs. The first group was used to validate the method and to gather relevant information regarding rock–fluid interactions. Following this initial round of testing, reservoir rocks were used.

Figures 2 and 3 present, respectively, the cumulative and incremental pore space distributions against the pore throat radius and cumulative permeability distributions as a function of pore throat radius and for the grainstone and stromatolite samples revealing significant differences in pore connectivity between the two rock facies analyzed.

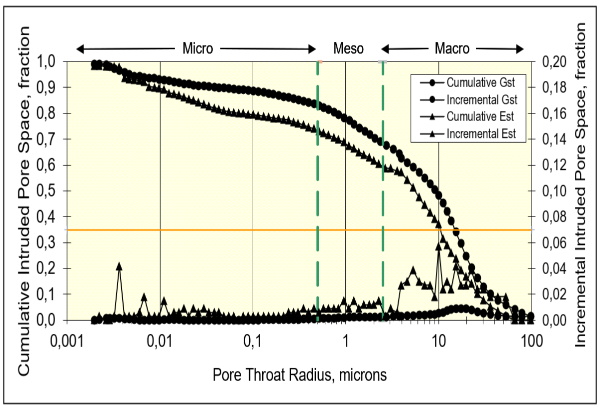

Figure 2 - Pore throat distribution for stromatolite and grainstone facies from Brazilian Pré-salt reservoir

In Figure 2, the cumulative permeability curves indicate that pore throats smaller than approximately 10 microns contribute marginally to total permeability. The main contribution is associated with throats in the range between 10 and 100 microns. The curve for the grainstone sample is shifted toward larger pore throats compared with the stromatolite, indicating that grainstone is supported predominantly by meso- and macropores, enhancing fluid flow capacity. Conversely, stromatolite presents a more restricted pore network dominated by smaller throats, resulting in a sharper decline in cumulative permeability.

Figure 3 complements this observation by showing that the cumulative pore space distribution for grainstone reflects a progressive filling dominated by meso- and macropore throats. The incremental curves clearly exhibit distinct peaks within these ranges, highlighting the dominance of well-connected and effective pore structures. In contrast, the stromatolite sample shows both cumulative and incremental distributions shifted toward smaller pore throats, reinforcing the presence of a less efficient pore network in terms of fluid flow.

The pore classification into micro-, meso-, and macropores, as depicted in Figure 3, highlights that micropores contribute minimally to both permeability and effective storage, while meso- and macropores play a critical role in maintaining effective permeability. Those differences between grainstone and stromatolite may be attributed to diagenetic processes which can be inherent lithological heterogeneity. These results underscore the importance of preserving larger pore structures to ensure efficient flow in carbonate rock samples, particularly in highly heterogeneous reservoirs.

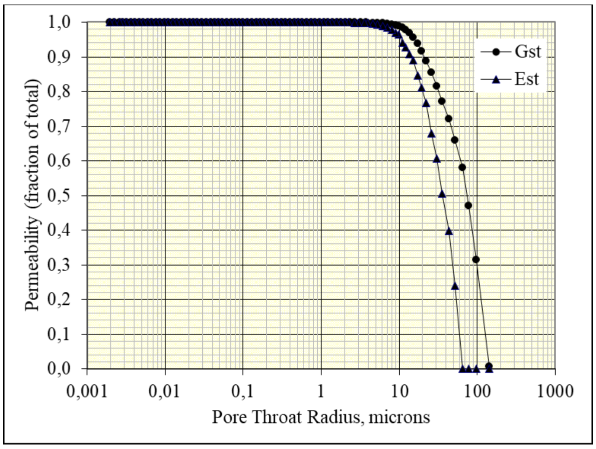

Figure 3 - cumulative permeability distribution for grainstone and stromatolite samples

The first step was to measure the petrophysical properties of the sample set using a DV-4000 pore-permeameter under a confining pressure of 1000 psi. Table 4 presents the samples classified into two main lithotypes stromatolites and grainstones along with their respective face identifications. The same table also displays the saturation values obtained during the preparation of the initial oil saturation, which was performed using an ACES-300 ultracentrifuge capable of generating capillary pressures of up to 120 psi. In this process, formation water was used as the wetting phase to establish the initial water saturation (Swi). To determine Swi through centrifugation, the samples previously saturated with formation water were subjected to a centrifugal force in the presence of EMCA PLUS 7015 mineral oil.

Table 4 - Petrophysical properties obtained from the samples studied

Sample	L, cm	D, cm	K, mD	Φ, %	VP, ml	Swi, %
Est1	6.2	3.8	52.3	13.0	9.2	22.5
Est 2	5.3	3.7	108.6	17.4	10.3	23.7
Gst1	5.5	3.7	61.1	18.9	11.7	21.6
Gst2	4.5	3.7	107.7	16.3	8.2	19.7

2.4 Mild cleaning validation with Outcrop

To validate the mild cleaning method, activities were initiated with the selection of two groups of carbonate outcrop samples. These samples were properly characterized in their petrophysical properties. Subsequently, they were saturated with 100% formation water and placed in a holder to inject 1500 times diluted formation water at a flow rate of 0.1 cc/min for 15 consecutive days. Figure 4 presents the results of the collected effluents, which were characterized using ion chromatography, and the pH of each sample was measured to assess potential dissolutions and reactions between the injection fluid and the rock.

2.5 Relative permeability using multistep

The relative permeability evaluation method was adapted from the work of Lenormand et al. 2016 [9]. The same flow rates suggested in that study were initially employed. However, adaptation was required for the samples used in this work, as it was not possible to obtain stable differential pressure values at the recommended flow rates. Relative permeability curvas were obtained from processing by CYDAREX software and LET method.

3 Results

Once the outcrop samples were saturated with formation water, they were placed in the forced displacement bench. A confining pressure of 1000 psi was applied, and formation water diluted 1500 times was injected at a flow rate of 0.1 mL/min for 14 consecutive days. During the injection, effluents were collected and analyzed using ion chromatography. To confirm that the cleaning process was complete, the electrical conductivity of the effluent was measured. Based on the analysis of the collected effluents, the variation of the main ions of interest with the injected pore volume was plotted. Figure 4 shows this variation for the ions present in the formation water. This allowed for the monitoring of key ions and the identification of undesirable effects such as deposition phenomena, chemical reactions, and potential precipitations, which could lead to significant changes in the petrophysical properties of the samples under study.

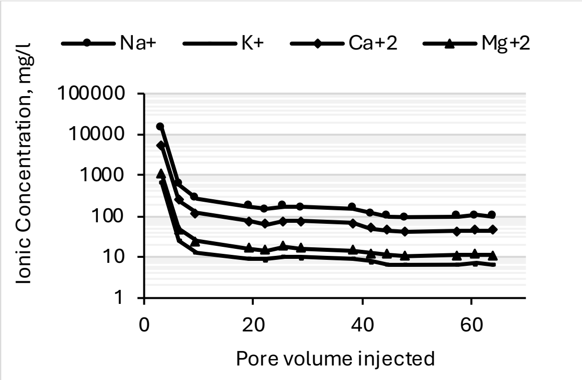

Figure 4 – Variation of ionic concentrations along the mild cleaning

The validation method process consisted of the systematic injection of formation water diluted 1500 times into outcrop samples of Indiana limestone. As previously described, effluents were collected throughout the injection process to monitor variations in ionic composition. This procedure was repeated twice. The samples were initially characterized in terms of their petrophysical properties prior to the first cleaning cycle, after which they were subjected to the injection protocol. Upon completion, the samples were placed in an oven until a constant mass was achieved and were subsequently recharacterized. The samples were then re-saturated with formation water and submitted to the same cleaning procedure. Following the second mild cleaning cycle, a third set of petrophysical measurements was conducted.

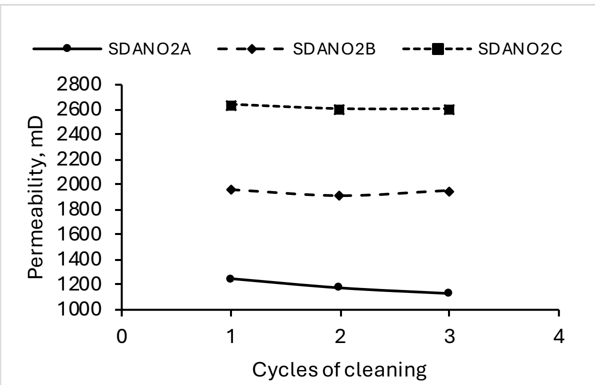

Figure 5 – Permeability variation of outcrop after mild cleaning

The results obtained during this stage are presented in Figures 5 and 6 illustrating the variation in permeability and porosity of the samples used in this study. As observed, no significant changes in both, porosity and permeability occurred during the cleaning cycles, suggesting low reactivity between the rock and the injected fluid. This finding is further supported by the porosity data shown in Figure 6, which similarly indicates minimal variation. Together, these results confirm the effectiveness and non-invasive nature of the mild cleaning process.

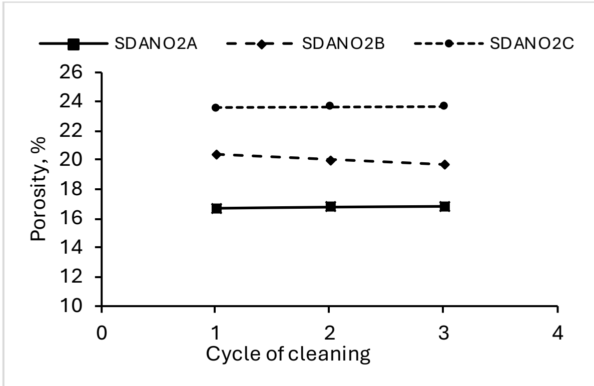

Figure 6 – Porosity variation of outcrop after mild cleaning

To further evaluate the mild cleaning process, a reservoir rock sample was subjected to the same procedure, aiming to verify whether similar behaviors are observed in actual reservoir rocks. For this phase, pH measurements of the collected effluents were also incorporated. Figure 7 presents the results obtained during the mild cleaning of the reservoir sample, which corresponds to a stromatolite—one of the main facies found in Brazilian Pre-salt reservoirs. This type of rock may contain quartz and clay mineral variations ranging from 8 to 12%.

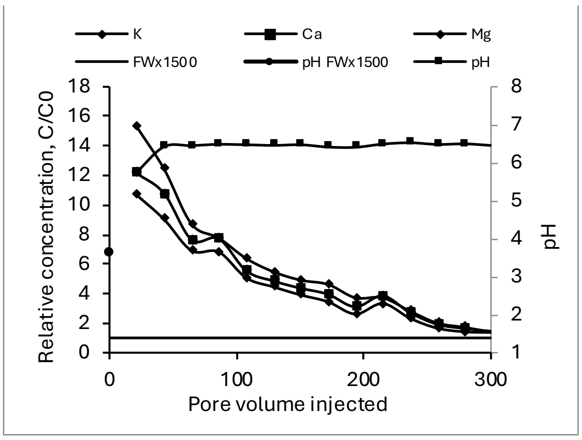

Figure 7 – Variation of ionic concentration of reservoir samples along the mild cleaning

In Figure 7, the relative concentration of key ions is plotted against the injected pore volume.

Additional relevant information is included, such as the pH of the injected solution and a reference line corresponding to the concentration of the injection water. As shown, the relative concentrations of the ions of interest decrease rapidly with the progression of injected pore volumes. However, the overall cleaning process required five times longer than it did for the outcrop rocks.

Regarding pH variation, Figure 7 also presents the pH values of the effluents collected during the injection of diluted seawater. These results suggest minimal to no reactivity of the minerals present in the reservoir sample an observation that is consistent with the stable behavior of ion concentrations throughout the injection process.

3.1 Analysis of Kro values at Swi condition based on wettability restoration and cleaning process evolution

The following figures illustrate the evolution of the relative permeability to oil at irreducible water saturation (Kro@Swi) over time, considering different stages of fluid exposure and injected, respectively. Initially, a high Kro@Swi values are observed during light oil injection, reflecting a condition of high oil mobility typical of a strongly water-wet system. After the following exposure to crude oil, there is a sharp decrease in Kro, which subsequently stabilizes at significantly lower values. This behavior indicates a substantial shift in the rock's wettability state, progressively transitioning towards a non-water wetness condition. In this configuration, a considerable portion of the oil adheres to the pore walls, reducing the efficiency of the main flow paths and consequently lowering Kro. Subsequent injection of model oil does not significantly alter the Kro values, which remain stable, reinforcing the establishment of an oil-wet or mixed-wet condition as a result of the prolonged interaction with crude oil.

After performing the cleaning procedure, a slight increase in Kro@Swi is observed, although it does not fully recover to the initial levels recorded during the light oil injection phase. This indicates that the restoration process was effective in preserving the wettability. These results highlight the sensitivity of this methodology proposed by the direct indicator wettability preservation porous media. Moreover, it emphasizes the importance of robust cleaning and restoration protocols to ensure that laboratory measurements accurately represent the original wettability conditions of the reservoir.

The mild cleaning process on reservoir rocks was further extended by preparing four rock samples: two stromatolites and two Grainstones. The samples, at irreducible water saturation conditions, were aged using both dynamic and static methods.

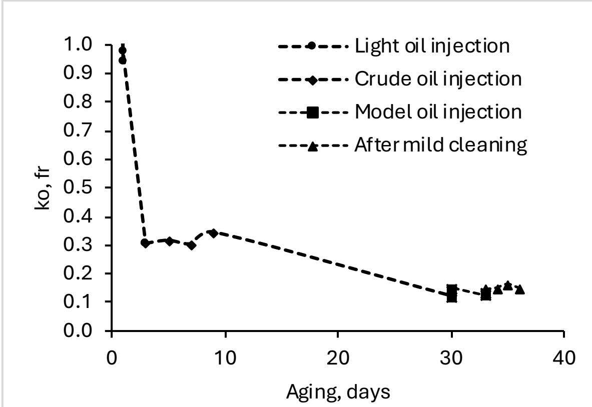

Figure 8 – Relative permeability fraction in all stage of static aging for Grainstone sample

To evaluate the effectiveness of the mild cleaning process, the relative permeability fraction to oil (k_{ro}) was measured under irreducible water saturation within a formation water mineral oil system. This configuration implies that the system is preferentially water-wet, thereby enabling the assessment of wettability preservation throughout the cleaning procedure.

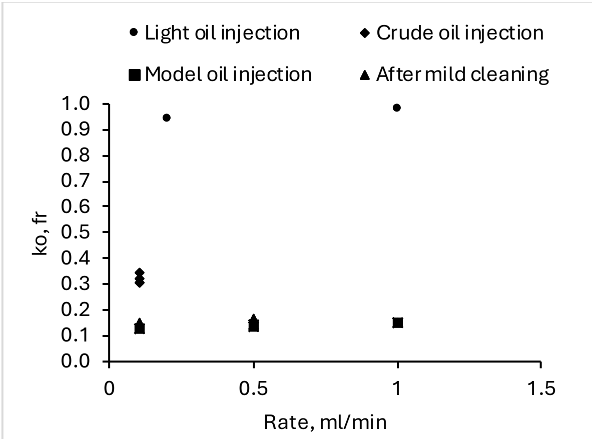

Figure 9 – Relative permeability fraction with different rate for static aging for Stromatolite sample

Figures 8 and 10 present the measured values throughout the wettability restoration process using static aging. As shown in the figures, there was a significant reduction in kro once the mineral oil was replaced with dead oil. This indicates that the polar components present in the oil interact with the sample surface, altering its wettability in the initial phase. After the static aging process was completed, the samples were placed in an oven and then injected with a model oil mixture—composed of a solvent and crude oil formulated to adjust the viscosity ratio.

Following the injection of the model oil, kro measurements were performed at different flow rates to assess the effectiveness of the wettability restoration process. Once it was confirmed that kro values remained stable across the evaluated flow rates, a multistep relative permeability test was conducted. Upon completion of the test, the mild cleaning process was initiated by injecting diluted formation water and collecting effluents until electrical conductivity values reflected those of the injected fluid.

With this initial cleaning phase concluded, hexane was injected at the same flow rate, and effluents were monitored using fluorescence to identify the removal of hydrocarbons from the sample. To verify the preservation of wettability, a kro measurement using the solvent was carried out and compared with results obtained throughout all stages of the wettability restoration process.

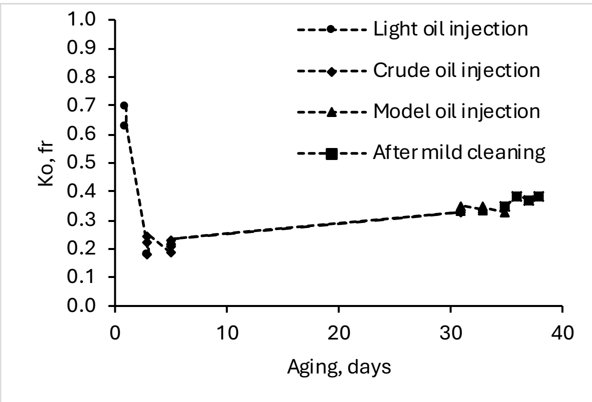

Figure 10 – Relative permeability fraction in all stages of static aging for Stromatolite sample

Figures 9 and 11 present the results obtained during the different flow rate evaluations in each stage. As observed in both figures, the kro values measured for the solvent were like those obtained for the model oil, indicating that both the wettability restoration and mild cleaning processes successfully preserved the original wettability conditions. These findings support the conclusion that the implemented methodologies were effective in maintaining the original wetting state of the rock samples, even after multiple stages of treatment and fluid injection.

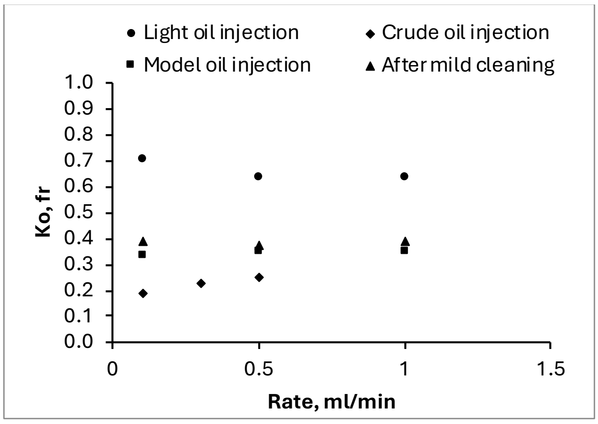

Figure 11 – Relative permeability fraction with different rates for static aging for Grainstone sample

For the dynamic aging process, kro was measured under irreducible water saturation using mineral oil at three different flow rates, as shown in Figures 12 and 13. Following these initial measurements, the mineral oil was replaced with dead oil, and a controlled volume of 2.0 pore volumes of dead oil was injected at a flow rate of 0.1 ml/min every 48 hours, totaling 12 injections. As observed in both figures, once the dead oil was introduced, a nearly stable trend in wettability restoration was established using this technique. Upon completion of the dynamic aging process, kro was re-evaluated at different flow rates, and similar values were obtained across the tested rates, indicating consistency. The relative permeability test was then carried out.

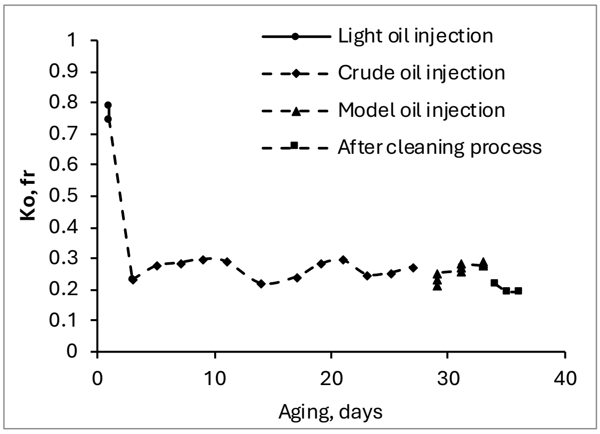

Figure 12 – Relative permeability fraction in all stages of dynamic aging for Grainstone sample

The mild cleaning process for these samples was conducted as previously described. The kro values obtained with the solvent indicated a slight decrease in the Grainstone sample, possibly due to differences in the adsorption dynamics of polar components between the studied facies. In contrast, for the stromatolite, the values obtained throughout both the wettability restoration and mild cleaning procedures confirmed effective preservation of wettability.

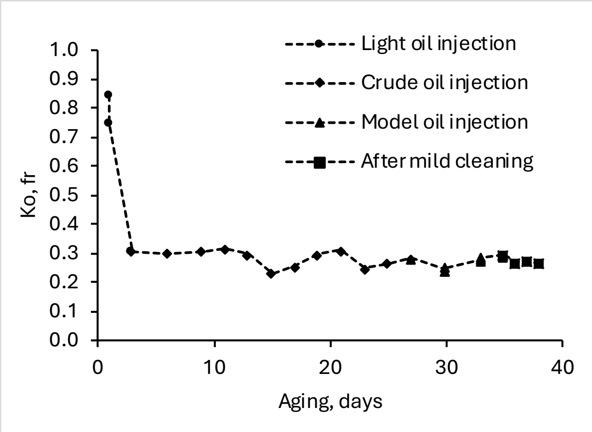

Figure 13 – Relative permeability fraction in all stages of dynamic aging for Stromatolite sample

3.2 Oil Recovery from multistep method

The differential pressure results for the grainstone sample, also shown in Figure 14, exhibited a different trend compared to the stromatolite sample. Once the mineral oil was replaced and the first dead oil injection was performed, the differential pressure progressively decreased with each subsequent injection, a trend that continued until the tenth injection. Therefore, for these facies, at least 20 pore volumes or approximately 30 days of dynamic aging are required to reach wettability restoration. Two additional injections were conducted to confirm that the differential pressure values had stabilized and would not show significant variations. When correlating these results with the polar component indices throughout the injections, it is evident that the largest pressure variations occurred during the initial oil injections, indicating strong interactions between the polar components and the rock surface, with stabilization occurring toward the end of the injection period.

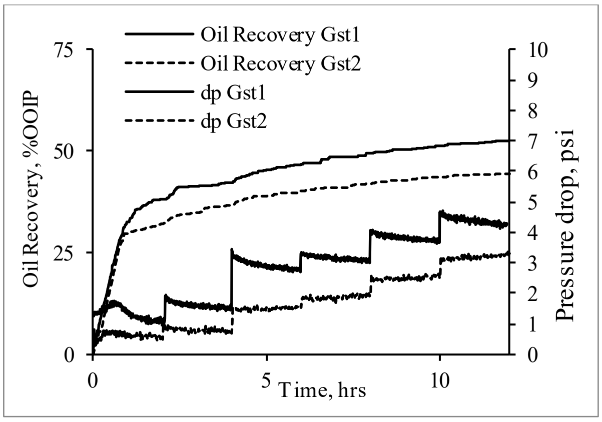

Figure 14 – Oil recovery and pressure drop for Grainstone sample

Regarding the oil recovery behavior resulting from the evaluated aging procedures, it can be stated that for the stromatolites (Figure 15), there were no significant differences in the recovery levels or in the differential pressures throughout the applied flow rates. Additionally, the figure shows that it was necessary to extend the final flow rates for a longer period, which led to the production of additional oil volumes. This may represent an experimental artifact, since each sample must be individually monitored in terms of both differential pressure and oil volume recovered per applied flow rate. On the other hand, for the Grainstone samples (Figure 14), the oil recovery behavior showed significantly different recovered volumes, which may be attributed to the dynamic interaction of polar components in each aging process characteristic of these facies.

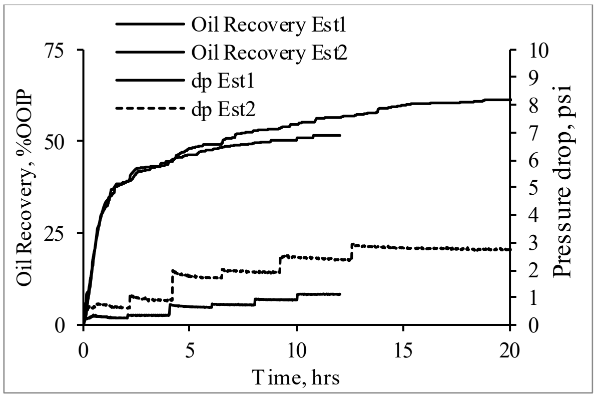

Figure 15 – Oil recovery and pressure drop for Stromatolite sample

3.3 Relative permeability using a multistep method

To determine the relative permeability curves from the multistep tests, the LET method was applied, in which several parameters are considered. The data were fitted using experimental data adjustment through the bi-Exp multi-step method for all datasets used in each simulation. Figures 16 and 17 present the relative permeability curves obtained for each evaluated facies, representing the behavior of each test and the aging process applied to each sample.

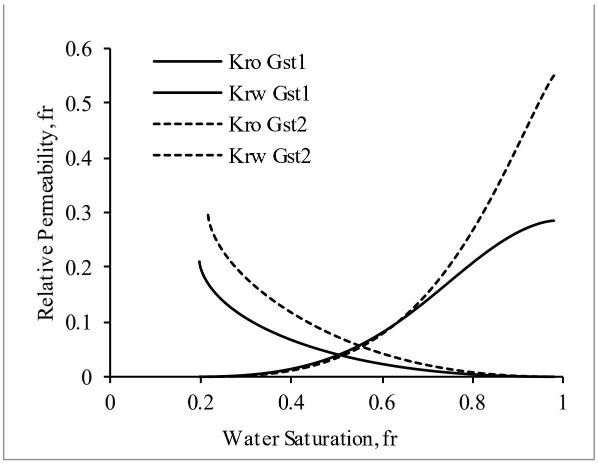

Figure 16 – Relative permeability obtained from both aging processes for Grainstone sample

In both figures, it is noticeable that the wettability indicated by the shape of the curves shows a slight oil-wet preference, which is a result of the aging process applied. It is important to highlight that for the Grainstone samples (Figure 16), the obtained curves exhibit a slight variation, which contrasts with the data obtained during the evaluation of the relative permeability fractions throughout the aging process. In the case of the stromatolite samples (Figure 17), the curve behavior shows fewer differences among them, which suggests a different interaction of polar components during each aging process.

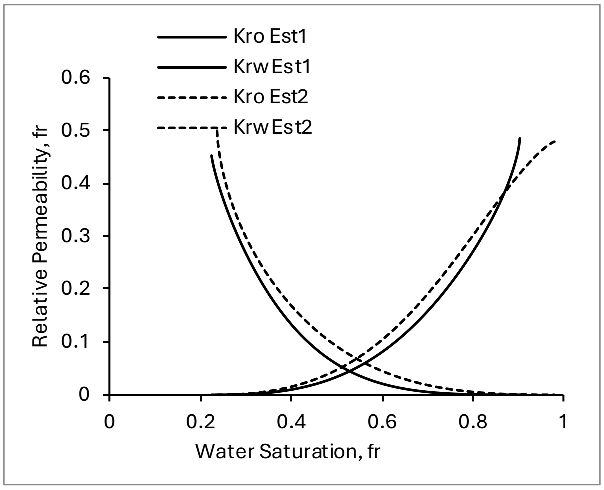

Figure 17 - Relative permeability obtained from both aging processes for Stromatolite sample

4 Conclusion

A mild cleaning process was developed for outcrop samples using fluids with low reactivity in a rock/oil/water system, demonstrating that it is possible to remove salts present in the samples without the need for aggressive solvents. No evidence of alterations in petrophysical properties was observed during any of the cleaning stages performed.

For the mild cleaning process applied to reservoir samples, an increase in the volume of injected fluid was observed, serving the same purpose. In the case of hexane use, the results indicated that it was possible to remove the hydrocarbons present in the samples in a controlled manner.

The indicators used to assess the effectiveness of mild cleaning directly demonstrated that it is possible to preserve wettability for relative permeability tests aimed at formation characterization. These findings were confirmed when the samples were evaluated under different flow rates.

The relative permeability curves obtained from the multistep tests highlighted the importance of the wettability restoration process. The results were consistent with an oil-wet preference in the stromatolite samples. In contrast, the Grainstone rocks exhibited two distinct trends, indicating the presence of different wettability states.

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