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Full Length Article

Influence of oil aging time, pressure and temperature on contact angle measurements of reservoir mineral surfaces

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A R T I C L E I N F O

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A B S T R A C T

Reservoir wettability plays a critical role in determining the fluid interactions, distributions, and flow paths in porous mediums, which is directly reflected in oil production and recovery strategies during a field's production life. Many factors can influence reservoir wettability characterisation on the laboratory scale such as: sample origin, mineralogical composition, cleaning procedures for further analysis, pressure, temperature, and the oil aging process. The best practice to restore the sample's original wettability by aging processes is unclear, impacting the assessment of contact angle (CA) measurements. This work addresses the oil aging time and experimental conditions associated with the pressure and temperature in CA measurements on rock surfaces from a Brazilian pre-salt reservoir with a predominance of calcite, dolomite, and quartz, respectively. For this purpose, all surfaces used were cleaned with solvents and saturated with synthetic formation water under vacuum conditions. To evaluate the influence of oil aging time on the samples' wettability, the CA measurements, by captive bubble method, were performed on unaged surfaces. The CA measurements were then taken again on the same surfaces after 14 and 42 days of oil aging. To evaluate the pressure and temperature effects on the CA values, the experiments were run at 14.7 psi and 22 °C (room conditions), 14.7 psi and 60 °C and 4000 psi and 60 °C. The results showed a mischaracterization of all surfaces' original wettability due to the cleaning process, showing the higher hydrophilic degree and wettability character of neutral and water-wet surfaces. The oil aging procedure was confirmed to be a fundamental process in restoring the wettability of rock surfaces, requiring more than 14 days to be achieved. Pressure and temperature conditions also matter on CA evaluations. The surface's hydrophobic degree increased as the pressure increased from 14.7 psi to 4000 psi, and the temperature from 22 °C to 60 °C. Furthermore, a higher influence of temperature than pressure was observed to impact CA values. Oil aging and experimental conditions presented a larger effect on increasing the hydrophobic degree of samples with calcite and dolomite predominance than those with quartz predominance.

1. Introduction

Carbonate reservoirs are recognized by their permo-porosity heterogeneities and mineralogical features [1–4], which are linked to the rock-fluid interactions and, consequently, reservoir wettability.

The main minerals content in reservoir rocks comprises carbonates (calcite and dolomite) and quartz [5]. Calcite, dolomite, and quartz are related as predominant minerals identified by scanning electron microscopy-based image analysis (SEM-MLA) coupled with X ray diffraction of rock samples from a Brazilian pre-salt reservoir [6]. Each mineral can interact differently with the formation fluids, such as oil and formation water, resulting in wettability heterogeneities and therefore making the composite rock's wetting behaviour challenging to describe [5,6]. The local wettability evaluation by contact angle (CA) measurements of target reservoir rocks, considering surfaces with high concentrations of predominant minerals, can help understand the mineral-fluid

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behavior and its wettability tendency [6]. Arif et al. [7] emphasised that using an analogous mineral sample to represent the carbonate rock reservoir is not entirely justified for contact angle measurements.

CA is a quantitative method for determining the wettability of rock samples [8]. However, the wetting properties can be influenced by pressure, temperature, salinity, pH, oil composition, rock type, surface mineralogy, sample roughness, and sample handling, such as the cleaning method and oil aging conditions [7,9–11]. Moreover, the droplet size can influence the CA measurements [12–14]. However, the contact angle's droplet size dependence can be negligible when using smooth homogeneous surfaces [14].

The application of solvents to remove the organic and inorganic compounds from rock samples tends to change the original wettability to water wet [8,15,16]. This effect can be emphasized by the water aging process; a procedure recommended to be performed before the oil aging [4,17,18]. Moreover, this water wet behavior can be correlated before the crude oil migration process [5].

After water aging, to restore the rock wettability, an oil aging process must be carried out. The adsorption of organic compounds on rock surfaces is considered a critical issue for proper wettability characterization [11,19–21]. Furthermore, the total oil acid number (AN), characterized by the carboxylic and naphthenic acid content in the crude oil [20], also impacts the wetting nature of the reservoir [22]. Nevertheless, as noted in the literature, there is no consensus about the time required to conduct the aging process. According to some studies, the aging times adopted ranged from 5 min to 60 days [4,17,18,23,24].

The literature points out that carbonate reservoirs can present water, mixed/neutral, or oil wettability [25–27]. However, there is a consensus that this kind of reservoir has mixed or oil-wet wetting properties [19,28].

Some wettability variation is correlated to surface charge variation associated with mineralogical composition [29]. A positive charge on carbonate surfaces [30] can contribute to the adsorption of carboxyl groups during oil aging [31]. Using Zeta potential measurements, Shehata and Nasr-El-Din [32] verified that carbonate samples such as calcite and dolomite present more positive values than sandstone.

Some authors have evaluated the pressure and temperature influence on CA values. Wang and Gupta [23] assessed the wettability of calcite and quartz samples. These samples were classified as water wet. Nevertheless, as the pressure increased (200 psi up to 3000 psi), the hydrophobic degree increased also, while the temperature effect shows an opposite trend. However, it is worth noting that the samples were aged for only 5 min in this work. Such a short period may not have been enough to restore the wettability of the sample.

Hamouda and Gomari [18] evaluated a system consisting of calcite and mineral oil containing fatty acids. The samples were aged for 24 h. According to the results, by increasing the temperature, the calcite surfaces became more hydrophilic. Zeta potential analysis corroborated this result. The increase in temperature led to a reduction in the positive charges on the calcite surface. In this way, the adherence of fatty acids on the mineral surface was less effective. However, in the former work cited [23], the aging time was relatively short.

On carbonate reservoir samples aged for 30 days, Najafi et al. [33] observed that by increasing the pressure (3000 to 4500 psi) at a constant temperature, the hydrophobic degree increased, but without changing the wettability classification. The increase in temperature (25 °C to 110 °C) at constant pressure for two samples presented a hydrophobic effect, which changed the wettability classification (water to neutral). For the sample that showed the highest hydrophilic degree, the authors associated the water wetness with the high resin/asphaltene ratio in the oleic phase.

Arif et al. [7] evaluated the pressure and temperature effects of rock samples' mineralogical composition, ranging from 100% calcite to 100% dolomite. Increasing the pressure from 14.7 psi up to 2175.57psi at 50 °C, it was observed that the hydrophobic degree increased. Considering the temperature effect (25 °C up to 50° at 14.7 psi), the authors reported different hydrophilicity behavior from the samples due to their mineralogical composition. The sample with 100% dolomite showed a higher hydrophobic degree than other samples.

According to works published elsewhere, the pressure influence on CA values is less significant when compared to the temperature influence [7,33]. However, the temperature effect is not evident, so that these differences may be associated with the oil-rock and oil-brine systems [10].

Despite the existence of scientific investigations into the influence of pressure and temperature on the contact angle, including studies on carbonates, little is known about these properties in ultra-deep basin reservoirs, such as the case of the Brazilian pre-salt reservoirs. The peculiarities of these reservoirs include high pressure and temperatures acting in specific mineralogical proportions. In such a scenario, there is a lack of studies with systematic approaches evaluating the oil aging process to restore the sample's original wettability, emphasizing the reservoir's mineral heterogeneity in this process. The characterization of these systems has a considerable influence on the modeling of reservoirs and, consequently, on the reduction of uncertainties related to oil and gas recovery.

This work evaluates the influence of dead oil aging time, temperature, and pressure in the CA values on rock surfaces from a Brazilian pre-salt reservoir, where mineralogical composition was mostly of calcite, dolomite, quartz and mixture of these components.

2. Experimental procedures

In this section, the experimental procedures to evaluate the CA measurements by the captive bubble method of varying the aging time and experimental conditions will be described in detail.

2.1. Test protocol

The experimental test protocol is presented in Fig. 1. The flowchart represents each step taken to develop contact angle (CA) measurements considering distinct oil aging periods, temperature and pressure conditions.

The experiments were carried out using dead oil, synthetic formation water (SFW), and surfaces with a predominance of calcite, dolomite and quartz, and a mixture of them. These surfaces refer to plugs' slices samples from a Brazilian pre-salt carbonate reservoir.

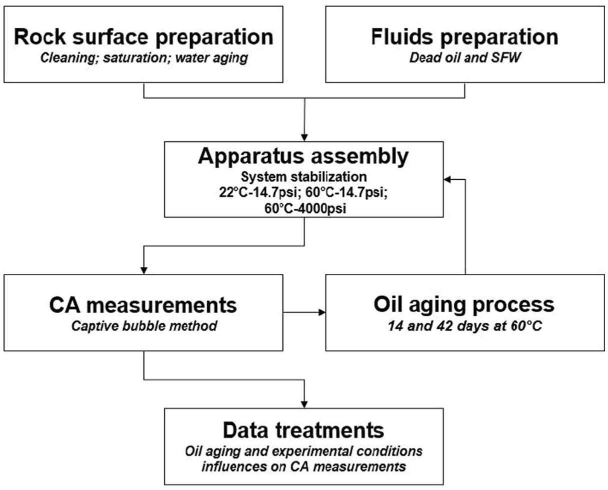

Fig. 1. Test protocol for CA measurements under different aging periods and experimental conditions of temperature and pressure.

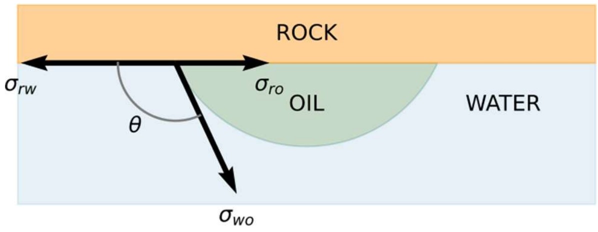

Fig. 2. Contact angle measured from the denser phase as is the usual convention.

Table 1
Synthetic formation water composition and dead oil properties.

SWF Salts	NaCl	$CaCl_2.2H_2O$	$MgCl_2 \cdot 6H_2O$	KCl	Na_2SO_4	$NaHCO_3$
ppm	175,312	27,182	14,002	6,593	58	25

Some phases properties
- SFW solution: pH = 7.7 and density = 1.14 g/cm^3 (22 °C and 14.7psi)
- Dead oil: 29.5°API; Total acid number (TAN) = 0.57 mg KOH/g; Density, viscosity and dead oil-SFW interfacial tension (IFT) are 0.8884 g/cm^3, 25.03 mPa.s and 9.66 mN/m, respectivelly (at 60 °C and 4000 psi)

Source: Adapted from Ferrari *et al.* [6].

2.2. SFW composition and dead oil properties

The external phase in CA measurements was the SWF, the denser phase where the contact angle is, by convention, measured [8] as in Fig. 2, and the droplet was the dead oil. These phases represent the target reservoir fluid formation, and their properties are in Table 1.

2.3. Rock surfaces preparation: Cleaning, saturation, and SFW aging process

The surfaces used to perform the CA measurements were the same ones used by Ferrari et al. [6], i.e., two samples of each mineral predominant, such as calcite (C1 and C2), dolomite (D1 and D2), quartz (Q1 and Q2) and the mixture of them (M1 and M2).

The previous work published [6] focused on the influence of mineralogy on contact angle measurements and wettability determinations. In this previous work, the contact angle was measured on selected surfaces guided by SEM-MLA results. Those same selected surfaces, mineralogically characterized by the SEM-MLA analysis, were used in the contact angle measurements in this present work, now considering an oil aging, temperature and pressure effects. These SEM-MLA results are presented in Table 2. Also, it is important to mention that XRD bulk analyses were carried out in representative aliquots of the bulk sample and represent the mineralogical composition of the whole sample. XRD results contributed to the correct identification of minerals by SEM-MLA since this system identifies only chemical composition but not crystalline phases. Based on the mineral map, we selected the areas for CA analysis.

Each sample presented in Table 2, refers to a smooth surface, due to the polishing process applied to SEM-MLA analysis and, also to minimize the roughness effect on CA measurements. According to the literature, polishing procedures reduced the surface roughness reducing the magnitude of contact angles values [34,35].

The samples surfaces were slightly cleaned with toluene and methanol, followed by the saturation process with SFW under vacuum conditions. After that, the SFW aging process under 60 °C for 24 h was performed. The whole procedure can be observed in detail in Ferrari *et al.* [6].

It is important to clarify that core samples acquired in wells are often contaminated with drilling fluid and other impurities, which completely mischaracterizes the properties of the reservoir fluids. In this way, it is not possible to analyze the core samples without the cleaning process. The provided core samples were previously cleaned using a systematic process common in core sample analytical routines [36]. Then, after that, it is necessary to restore the wettability of samples with an oil aging process [37].

The samples only aged in SFW will be considered as t0, i.e., no-oil aging. After CA measurements at t0, the oil aging process was performed for further measurements.

2.4. Oil aging evaluation

Oil aging was carried out to restore the original wettability of rock sample surfaces. To assess whether the aging time would influence the samples' wettability, the CA measurements were performed in three different periods. The first refers to no oil aged surfaces (t0), and the second and third after 14 (t14) and 42 (t42) days of oil aging, respectively. The process was performed by immersing each surface in dead oil and placing it into an air-bath at 60 °C during the respective period.

2.5. Experimental setup and CA quantification

The High Pressure and High Temperature apparatus (HPHT - up to 10,000 psi and 200 °C) used for CA measurements was the IFT Cell Part No 10 model from Core Lab Instruments, as shown in Fig. 3, which presents a measurement resolution of 0.1°.

Before assembling the system, the excess oil covering each surface was removed with absorbent paper until no more oil was transferred from the rock to the paper. The rock was then immersed in SFW and dried with soft paper. To place the rock samples into the HPHT cell, item 7 of Fig. 3, a double-sided tape was used to secure them in the sample holder, item 8 of Fig. 3.

After the apparatus was assembled, the external fluid (SFW) and the oil droplet were manually pumped into the HPHT cell. The system configuration after the mentioned procedure is present in Fig. 4.

To avoid the droplet size effect on CA measurements, the same needle was used in all experiments, with 0.793 mm ID (inner Diameter) and 1 mm OD (outer diameter), item 9 of Fig. 3, to put the oil droplets on the surfaces.

The experiments were carried out at 14.7 psi and 22 °C (room conditions), 14.7 psi and 60 °C, and at 4000 psi and 60 °C. In this way, it was possible to evaluate the pressure and temperature effects of CA characterization.

To perform the experiments at high pressure (4000 psi), the value was set by SWF injection and back pressure valve adjustments. In the experiments at high temperature (60 °C), the value was set by a digitally-controlled heating blanket. This temperature refers to a maximum value of further USBM (United States Bureau of Mines) experiments (not addressed in this work).

The captive bubble method was used to measure the external CA of the oil droplet on the mineral surface surrounded by SFW. The wettability classification as water-wet, neutral or oil-wet was considered for CA values lower than 75°, ranging from 75°up to 105° and higher than 105°, respectively [8,38].

Table 2
Samples identification and mineralogical composition.

Predominant mineral		Calcite		Dolomite		Quartz		Mixture	
Sample/Area		C1	C2	D1	D2	Q1	Q2	M1	M2
Main mineral content (w/w)	Quartz	3.76%	2.47%	12.83%	7.30%	93.93%	55.06%	16.27%	23.29%
	Dolomite	25.59%	16.05%	85.42%	89.37%	3.47%	25.86%	45.40%	49.99%
	Calcite	69.93%	81.23%	1.52%	0.09%	2.52%	18.67%	37.96%	25.85%

Fig. 3. Scheme of experimental apparatus for CA measurements.

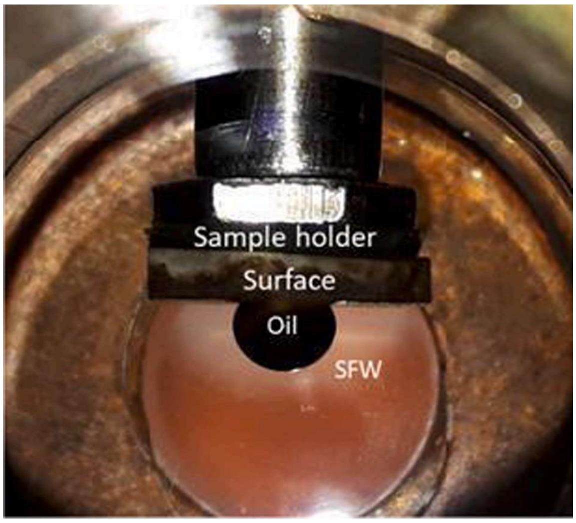

Fig. 4. Picture of SFW/oil droplet/surface/sample holder inside the HPHT cell.

3. Results and discussion

The global results of CA measurements of each surface, aging time, and experimental conditions are summarized in Fig. 5. All points were measured every 60 s until the end of the experiment. CA values reported were the average of at least ten points in the equilibrium.

In Fig. 5, three data for some samples in some conditions (D2 at t00 – 22 °C – 14.7psi; Q2 at t00 – 60 °C – 14.7psi; M2 at t42 – 22 °C – 14.7 psi) were not presented due to problems with the data acquisition during those measurements.

The presented data points out that the aging process and experimental conditions can alter the CA values and the wetting tendency of the samples tested. Increasing the oil aging period, temperature, and pressure tends to increase the CA values for all surfaces. In the following items, both effects on CA values will be presented.

3.1. Oil aging effect

According to the data presented in Fig. 5, lower values of contact angles were observed in each mineral surface with no oil aging (t0). As the CA values were lower than 105°, the surfaces presented neutral and water wetting tendencies. This higher hydrophilic degree is associated with the cleaning process, which modifies the original rock samples' wettability. According to the literature, the application of toluene and methanol on the rock surface alters its wettability to water-wet tendency [8,15,16].

After 14 days of oil aging (t14), Fig. 5 shows a tendency to increase the contact angles for all samples compared to the t0 data. For the quartz surfaces, even with CA increase, the wettability was not altered to oil-wet. However, for samples with a predominance of carbonate minerals, the CA values are concentrated in the oil-wet region (CA > 105°).

Considering 42 days of oil aging (t42), it was observed that the CA increasing tendency stayed the same, increasing the hydrophobic degree of all surfaces tested. This is supported by CA values higher than 105°, except for samples Q1 and M2, which presented water-wetting and neutral-wetting, respectively.

The higher hydrophobic degree may be associated with the oil phase contact on the rock surface during aging processes, and, consequently, the adsorption of organic compounds on the rock surface. This behavior is consistent with the information in the literature [4,11,24,38].

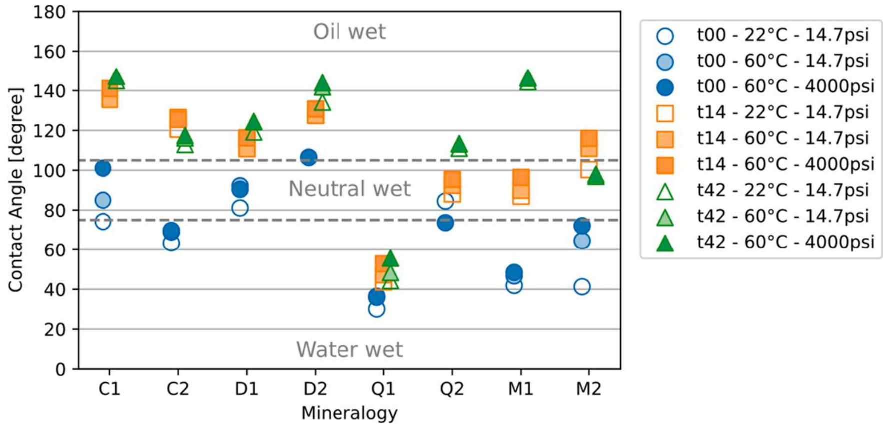

Fig. 5. Contact angle and wettability classification for each surface, aging time, and experimental condition.

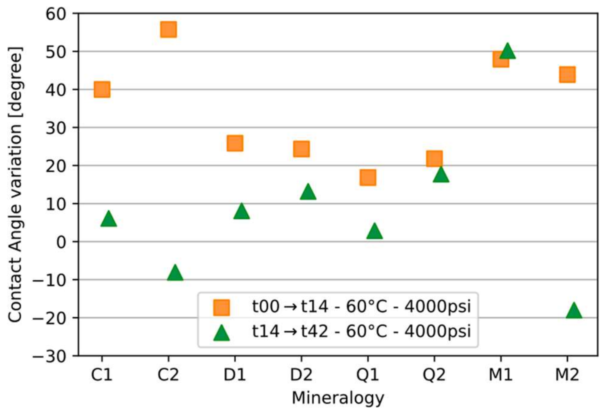

Fig. 6. Aging effect between 0 and 14 and 14–42 days on the contact angle measurements.

Fig. 6 presents the CA variation for each sample at 60 °C and 4000 psi (T60P4000), considering the data between 0 and 14 days and the continuity of the process, i.e., between 14 and 42 days oil aging.

Analyzing the aging process for the initial 14 days and afterwards up to 42 days, the collected data show a CA positive variation increase tendency that ranged from 16.8° to 55.8° and from 2.9° to 50.2°, respectively. In this way, the CA increase tendency after 14 to 42 days of aging was identified for all surfaces, except for samples C2 (-8.1°) and M2 (-18.0°). According to Okasha [39], the dead oil droplet becomes more spread on the rock surface due to asphaltene deposition as the aging time increase. Furthermore, oil aging shows more influence on carbonate samples compared to quartz ones.

Regarding the exceptions observed, the C2 sample has the higher calcite mineral content. As previously mentioned, a more positive charge on carbonate surfaces [30] can contribute to an easier adsorption of carboxyl groups during oil aging [31], in comparison with sandstone [32]. A hypothesis to explain such behavior of sample C2 would be a more favorable adsorption of such compounds during the oil aging process due to its higher calcite content, reducing its positive charge, and promoting a process of adsorption reversal, observed only after 42 days. In fact, the C2 sample presented a higher positive variation of the contact angle after 14 days, indicating higher adsorption of such organic compounds. On the other hand, sample M2 presents the third-highest quartz content in a carbonate-containing mineral bulk, following samples Q1 and Q2. Both this relatively higher quartz content and a bulk containing carbonate minerals would explain the similar behavior of the M2 sample to the C2 sample after 42 days of oil aging.

According to the presented data of the different mineralogies evaluated under the experimental conditions, it can be inferred that the period required to restore the surface's wettability is longer than 14 days for these particular rock samples and fluid samples.

In summary, the cleaning and oil aging process is important in contact angle evaluation. Oil aging is an essential process to restore the original wettability of rock samples. According to Drexler et al. [11], oil aging is one of the main factors influencing the wettability characterization of reservoir rock samples.

3.2. *Temperature and pressure effect on CA values*

The influence of temperature on CA measurements was evaluated from 22 °C to 60 °C at constant pressure (14.7 psi). Pressure influence was evaluated from 14.7 psi to 4000 psi at constant temperature (60 °C). The CA values for these conditions are separatedly presented in Fig. 7, considering all surfaces and aging times.

The presented data, considering all periods of oil aging, shows that as the temperature (Fig. 7(a)) and pressure increase (Fig. 7(b)), the contact angles tend to increase. This trend is stressed in Fig. 8. The plotted values represent the increase in CA values due to the influence of temperature (Fig. 8(a)) and pressure (Fig. 8(b)).

Increasing the temperature from 22° C to 60° C, there was an average increase in the CA values of 10.3°, 2.9° and 3.8° for t0, t14 and t42, respectively. Also, the CA variation was more significant for non-aged surfaces. This behaviour was similar to that reported by Okasha [39], when the author increases the temperature from 26.6 °C to 90 °C at 3000psi using calcite samples and crude oil. Furthermore, the oil droplet on non-aged samples can start the aging process during the contact angle measurements [39].

Increasing the pressure from 14.7 psi to 4000 psi, there was an average increase in the contact angle of 3.4°, 4.1°, and 1.5° for t0, t14 t42, respectively. The experimental condition influenced more on samples with carbonate mineral predominance compared to the samples with quartz predominance.

The presented results agree with those reported by Najafi et al. [33] and Arif et al. [7], in which the hydrophobic degree of surfaces tested increased as the temperature and pressure increased. Furthermore, the higher influence of temperature on CA measurements, compared to the pressure, also fits their results. In some cases (C1, D2, and M2), increasing the temperature from 22 °C to 60 °C changed the wettability classification, unlike the effect of pressure. Even though it showed a tendency to increase the contact angle, the pressure effect did not change the classification of surface wettability.

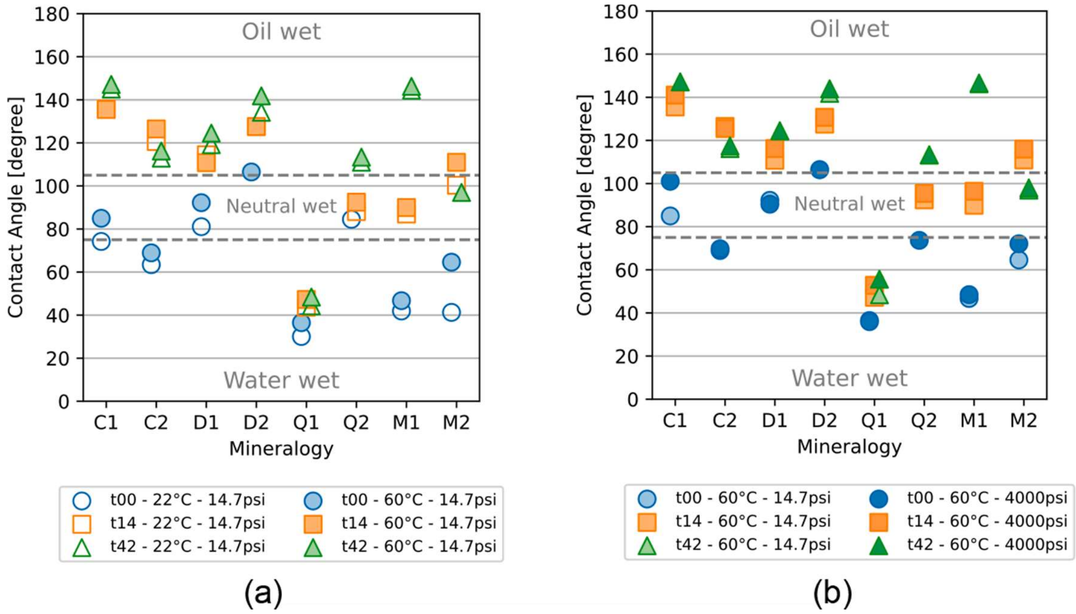

Fig. 7. (a) Temperature effect at constant pressure (14.7 psi) and (b) pressure effect at constant temperature (60 °C), on contact angle measurements.

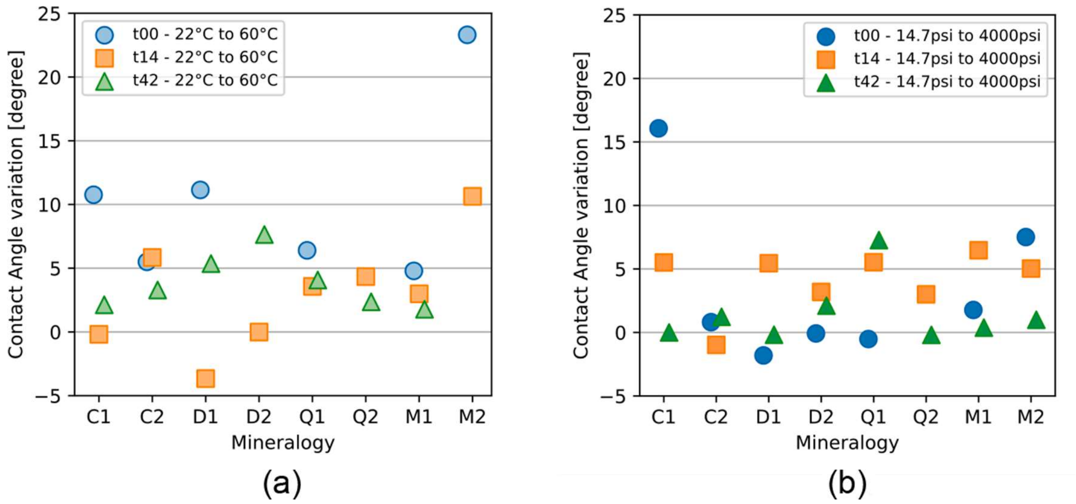

Fig. 8. Temperature (a) and pressure (b) effect on the contact angle measurements.

4. Conclusions

The contact angle measurements of the surfaces of rock samples extracted from plugs from a Brazilian Pre-Salt carbonate reservoir confirmed the mischaracterization of the original wettability of all surfaces due to the cleaning process.

The aging of the surfaces with dead oil was required to restore the original mineral's wettability. The dead oil contact on mineral surfaces decreases the wetness of the water phase for all samples tested.

Considering the system and experimental conditions evaluated, the results showed that the samples required more than 14 days of oil aging to restore wettability. A tendency for the contact angle values measured after 42 days of oil aging to increase was verified. This indicates that the hydrophobic degree of the samples increased after 14 days of oil aging.

Temperature changes showed significant effects on contact angle values following a tendency of CA to increase as the temperature rises. On the other hand, pressure changes showed less intense effects on CA although it followed an overall tendency to oil wetness when the pressure was increased. These results emphasize the need to evaluate wettability under the HPHT conditions found in the reservoirs.

Both the oil aging process and experimental conditions, associated with pressure and temperature, had a larger influence on increasing the hydrophobic degree on samples with carbonate mineral predominance when compared to the samples with quartz predominance.

CRediT authorship contribution statement

Bruno Marco de Oliveira Silveira: Conceptualization, Methodology, Investigation, Validation, Writing – original draft. **Rafael dos Santos Gioria:** Validation, Writing – review & editing. **Jhonatan Jair Arismendi-Florez:** Investigation, Writing – review & editing. **Thais Bortotti Fagundes:** Investigation, Writing – review & editing. **Mayara Antunes da Trindade Silva:** Investigation, Writing – review & editing. **Rodrigo Skinner:** Project administration, Resources, Writing – review

& editing. **Carina Ulsen:** Project administration, Funding acquisition, Methodology, Writing – review & editing. **Cleyton de Carvalho Carneiro:** Project administration, Funding acquisition, Writing – review & editing. **Jean Vicente Ferrari:** Conceptualization, Methodology, Writing – original draft, Supervision.

Declaration of competing interest

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

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