An Acad Bras Cienc (2021) 93(4): e20190534 DOI 10.1590/0001-3765202120190534
Anais da Academia Brasileira de Ciências | *Annals of the Brazilian Academy of Sciences*
Printed ISSN 0001-3765 I Online ISSN 1678-2690
www.scielo.br/aabc | www.fb.com/aabcjournal

ENGINEERING SCIENCES

Investigations on the adsorption, wettability and zeta potential of anionic surfactant in limestone

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Abstract: The understanding of the mechanisms that affect oil production in carbonaceous reservoirs has become increasingly necessary, particularly in limestone, which mostly features oil wettability properties that diminishes petroleum recovery. The objective of this work was to investigate the ability of anionic surfactant (coconut oil derived soap) to adsorb in limestone in order to promote wettability change. The finite-bath technique was employed with changes in temperature, mass of adsorbent material (limestone), contact time and surfactant concentration. Contact angle and zeta potential measurements were also made. The surfactant could be significantly adsorbed on the rock, possibly due to ions that are charged oppositely to the species on the rock surface. A temperature rise from 30 °C to 50 °C was unfavourable to the adsorption capacity. The oil-wettable *in-natura* limestone had its wettability reduced after the treatment with surfactant. The zeta potential measurements showed that electrostatic attractions play an important role in the adsorption process.

Key words: Surfactant, adsorption, wettability, limestone, zeta potential.

INTRODUCTION

Wettability is one important characteristic of reservoirs which decisively affects oil production and recovery, and therefore impacts the economical viability of the projects. Carbonaceous rocks are mostly oil wettable, which partly accounts for their relatively low productivity. Investigations have been developed with the purpose of reversing the wettability of this type of rock and enhance productivity (Alyafei & Blunt 2016, Amirpour et al. 2015, Andersen et al. 1991, El-Mofty & Shokir Emel 2003, Jackson & Vinogradov 2012, Lopez-Chavez et al. 2014, Mousavi et al. 2013, Strand & Hognesen 2006, Thomas et al. 1993, Zhang et al. 2007).

It is known that enhanced oil recovery methods have been implemented to maximize oil production from several types of reservoirs. Among these methods, chemical techniques involving the injection of surfactants can be developed. The use of surfactants in carbonaceous reservoirs aims to reverse their wettability, thereby increasing oil flow and recovery.

Adsorption is a phenomenon that may increase the effectiveness of surfactants because of their amphiphilic nature. They tend to adsorb on interfaces to extents that depend on their structures and the main effect is the reduction in the interfacial free energy. Researches with carbonaceous rock are therefore very important to elucidate the mechanisms implicated in the enhancement of oil production.

Legens et al. (1998) show the adsorption of benzoic acid on calcium carbonate by thermogravimetry and wettability assays from contact angle measurements. Good adsorption was observed with strong interactions between benzoic acid and rock, and monolayers were formed on the carbonate surface. The contact angle data indicated that the surface was less hydrophilic with the acid adsorption.

Standnes & Austad (2003) propose that it is possible to enhance oil recovery from carbonaceous reservoirs which have oil or neutral wettability by spontaneous imbibition with water. Low-cost, nontoxic primary amines ($R\text{-}NH_2$) were used as the chemicals that altered the wettability. The oil recovery percentages ranged between 50 % and 75 % of the original oil *in place*. The contact angle data confirmed the potential of a C_{10} amine solution to render the surface strongly water wettable at $pH = 6.5$ and temperatures in the range of 20 °C to 90 °C.

Xue et al. (2006) examine the adsorption of nonionic Triton X-100 (TX) on three types of inorganic surfaces (silica powder, γ-alumina and calcite). It was found that TX could not show appreciable adsorption rates on γ-alumina within the pH range $3.8 < pH < 12.1$, or on calcite for $7.7 < pH < 11.7$. Nevertheless, considerable reversible adsorption was observed for silica powder suspensions. The adsorption isotherms for TX with silica powder were better fitted with the Freundlich model. The adsorption of TX on silica powder diminished with increasing temperature from 15 °C to 45 °C, indicating that the adsorption was exothermic.

Jarrahian et al. (2012) use different analytical tools to investigate the effects of surfactants on the wettability of dolomitic rocks. The effects of cationic DTAB (dodecyltrimethyl ammonium bromide), nonionic TX and anionic SDS (sodium dodecylsulfate) were assessed. According to the data, DTAB was more efficient in changing wettability by means of ionic interactions, rendering the surface more water wettable. The nonionic TX adsorbed on the surface by electron polarization and ion exchange, and the surface of the dolomite was more weakly water wettable when compared with DTAB. The anionic SDS could be adsorbed by hydrophobic interactions between the surfactant tail and the oil found in the rock, and the surface wettability changed to neutral.

Ma et al. (2013) show the adsorption of a cationic (cetyl-pyridinium chloride – CPC) and an anionic (SDS) surfactant on carbonaceous materials. It was observed that CPC was inexpressively adsorbed on synthetic calcite when compared with SDS. However, CPC can be reasonably adsorbed on natural carbonaceous surfaces, such as dolomite and limestone. The authors demonstrated that the adsorption of CPC on carbonates strongly depends on the amount of silicon in the composition of the samples, since there are strong electrostatic interactions between carbonate impurities and CPC, due to the negative sites of silica and/or clay.

Wang et al. (2015) propose a novel methodology to determine the static adsorption of dodecylhydroxypropyl sulfobetaine (DSB) on limestone by HPLC. The results indicated that increasing concentration of sodium chloride in the medium impairs the DSB adsorption on the limestone surface due to increasing zeta potential. By contrast, the increasing concentration of calcium chloride initially impairs and then enhances the adsorption phenomenon, which indicates that the zeta potential tends to change on the limestone surface.

Karimi et al. (2016) show the alteration on the wettability of carbonate rocks by combining brine with surfactant during water flooding experiments. Different brine formulations were tested by contact angle assays and spontaneous imbibition experiments in oil-wet limestone core samples, both in the presence and absence of surfactant (DTAB). Contact angle measurements revealed that all low-salinity brine solutions changed the wettability of oil-wet surface towards more water-wet conditions. In the presence of cationic surfactants, the presence of positive and negative charges affected the wettability and rendered the surfaces more water-wet. The spontaneous imbibition results showed that the combination of the effects of ionic charges (Mg^{2+} and SO_4^{2-}) along with the cationic surfactant can remarkably enhance oil production.

Coconut oil was used to synthesize the anionic surfactant used in the present work. The surfactant is actually a solid mixture of sodium carboxylates obtained after neutralization with sodium hydroxide of the organic acids that comprise the natural oil (Santos et al. 2009). It has been named as Saponified Coconut Oil (SCO), and its adsorption on limestone has been investigated by applying different adsorption models with the finite-bath technique. Measurements of the zeta potential of the rock-surfactant solution interfaces were made aiming to provide another useful tool to elucidate the adsorption mechanism. The capacity of the surfactant to change the rock wettability was finally assessed by contact angle assays.

MATERIALS AND METHODS

Fluids and rock

The ionic surfactant, saponified coconut oil (SCO), was synthesized in laboratory. Table I shows the mean composition in fatty acids present in the vegetable oil that was used to obtain the ionic surfactant. Petroleum samples were collected from the *Ubarana* field (Brazil) and kindly supplied by Petrobras (0.8344 g/mL oil density and 2.90 cP viscosity - Haake Mars rheometer; 30 °C; shear rate from 1 to 1000 s^{-1}). Calcitic limestone samples were extracted from the Jandaíra Formation, in the State of Rio Grande do Norte (Brazil). The brine solution used in the experiments was a 2 % (w/v) KCl aqueous solution. This saline solution was then used to dissolve the surfactant to a final SCO concentration of 3.0 g L^{-1}.

Table I. Mean composition in fatty acids present in the coconut oil used.

Acid	Mass content
Capric ($C_{10}H_{20}O_2$)	6%
Lauric ($C_{12}H_{24}O_2$)	47%
Myristic ($C_{14}H_{28}O_2$)	18%
Palmitic ($C_{16}H_{32}O_2$)	9%
Stearic ($C_{18}H_{36}O_2$)	3%
Oleic ($C_{18}H_{34}O_2$)	6%
Linoleic ($C_{18}H_{32}O_2$)	2%
Ricinoleic ($C_{18}H_{34}O_3$)	–

Adsorption assays

Limestone samples were calcined at 250 °C for 6 h, crushed and sieved to be used in the adsorption assays, with fractions collected on mesh sieves between 48 and 100. Adsorption analyses were made with the finite-bath technique in a Dubnoff waterbath, described as follows.

First, the mass of adsorbent (limestone) in the SCO solution (3.0 g L^{-1}) in 2 % KCl (80 mL) was varied in samples of 0.4 g, 0.6 g, 0.8 g and 1.0 g, which were allowed to stir constantly for 2 h at 30 °C and 50 °C. Then these samples were filtered out (Castro Dantas et al. 2001, Krivova et al. 2013, Marsalek et al. 2011) and the surface tension of the filtrate was assessed as a function of surfactant concentration. Then it was possible to establish the equilibrium mass for the adsorption phenomenon. The surface tension experiments were conducted in a *SensaDyne* tensiometer.

Subsequently, the equilibrium mass was determined for the surfactant solution by varying the contact time in intervals of 5, 10, 20, 30, 60, 90, 120, 150, 180 and 240 min, under constant stirring, at 30 °C and 50 °C. For each experiment carried out at a different time interval, all samples were filtered out and the surface tension of the filtrate was assessed to determine its surfactant concentration. Then it was possible to establish the equilibrium time for the adsorption phenomenon.

Finally, the concentration of SCO was varied in 1, 2, 3, 4, 5, 6, 7 and 8 g L^{-1}, with fixed adsorbend mass and contact time, again under constant stirring at 30 °C and 50 °C. For each experiment carried out with a different SCO concentration, all samples were filtered out and the surface tension of the filtrate was assessed to determine its surfactant concentration. All procedures and analyses were carried out in duplicate.

It was then possible to determine the equilibrium surfactant concentration in each sample, by plotting the analytical curve for the surface tension (γ) *versus* surfactant concentration (ln C) data (Castro Dantas et al. 2002).

The adsorption capacity (*q*, in mg/g) has been calculated with Eq. (1):

$$q = [V (C_0 - C_e)] / m \quad (1)$$

where C_0 is the initial surfactant concentration (mg mL^{-1}), C_e is the concentration of surfactant in the filtrate (mg mL^{-1}); V is the solution volume (mL); and m is the mass of adsorbent (g).

The experimental data were adjusted with the Langmuir and Freundlich isotherms in order to verify which adsorption model provides the best fit. These theoretical models obey the linearized Eq. (2) and Eq. (3), respectively.

$$C_e/q = 1 / (K_L q_m) + C_e/q_m \quad (2)$$

$$\ln (q) = \ln (K_F) + (1/n) \ln (C_e) \quad (3)$$

where q_m (mg g^{-1}) is the adsorption capacity; K_L (mL mg^{-1}) is the equilibrium constant; and K_F and n are empirical constants which indicate the adsorption capacity and the intensity of the adsorption energy, respectively.

Contact angle measurements

Solid, cylindrical tablets were prepared with the limestone samples (retained on the filter paper during the adsorption assay) treated with SCO (3.0 g L^{-1}) and *in-natura*. The natural limestone sample was dried in an oven at 60 °C for 24 h. Six tablets were then obtained, two of which treated at 30 °C, two treated at 50 °C and two *in-natura*. The cylindrical tablets were prepared with a hydraulic press with the aim of reducing rugosity, which consists of surface irregularities on a small scale on the limestone surface. Contact angle measurements could then be made with the tablets using crude oil (density of 0.8344 g mL^{-1} and viscosity of 2.90 cP) in a Krüss goniometer, model DSA100.

Zeta potencial measurements

To analyze the electrical charges of the limestone particles dispersed in the solutions, zeta potential measurements were made in a Brookhaven ZetaPlus equipment after adsorption in a finite bath for 2 h at 30 °C, with brine (2 % KCl solution) and aqueous SCO solution with a concentration of 3 g L^{-1}. The pH of the solutions was taken prior to the zeta potential measurements. Each sample was analyzed with 10 repetitive assays and the final zeta potential was the average of these data.

RESULTS AND DISCUSSION

Adsorption

It was determined the equilibrium surfactant concentration in each sample, plotting the analytical curve for the surface tension (γ) *versus* surfactant concentration (ln C) data. The best-fit equation (below of critical micellar concentration) was $\gamma = -12.42 (\ln C) - 42.435$, with $R^2 = 0.8791$ (Fig. 1).

Equilibrium mass

In order to investigate the adsorption of SCO on the rock, the surface tension of the filtrates obtained with the finite-bath technique was determined. The surface tension of the original SCO solution with a surfactant concentration of 3 g L^{-1}, before being subjected to the finite-bath procedure, was 38.1 mN m^{-1}. In Table II, the average values of surface tensions of the SCO filtrates are presented as a function of temperature and mass of limestone.

A gradual increase in the value of the surface tension can be seen from the data in Table II, from values close to 38.1 mN m^{-1}, which is that of the original SCO solution, to values that are close to that of pure water, when 1.0 g of limestone is used. It can also be inferred that 0.8 g of limestone may be the equilibrium mass, at 30 ºC, from which no important effect on the surface tension is caused. These results indicate that the surfactant adsorption phenomenon occurs to a good extent, since the surface tension of the filtrate increases with increasing amount of adsorbent in contact with the SCO solution. This obviously means that the final solute concentration is lower the initial one, showing that more surfactant molecules could be adsorbed on the rock, reaching a saturation point. This good adsorption capacity may be due to the negative charge of the surfactant headgroup, opposed to the ionic charges on the solid limestone surface. In this case, electrostatic interactions between the surfactant ions and

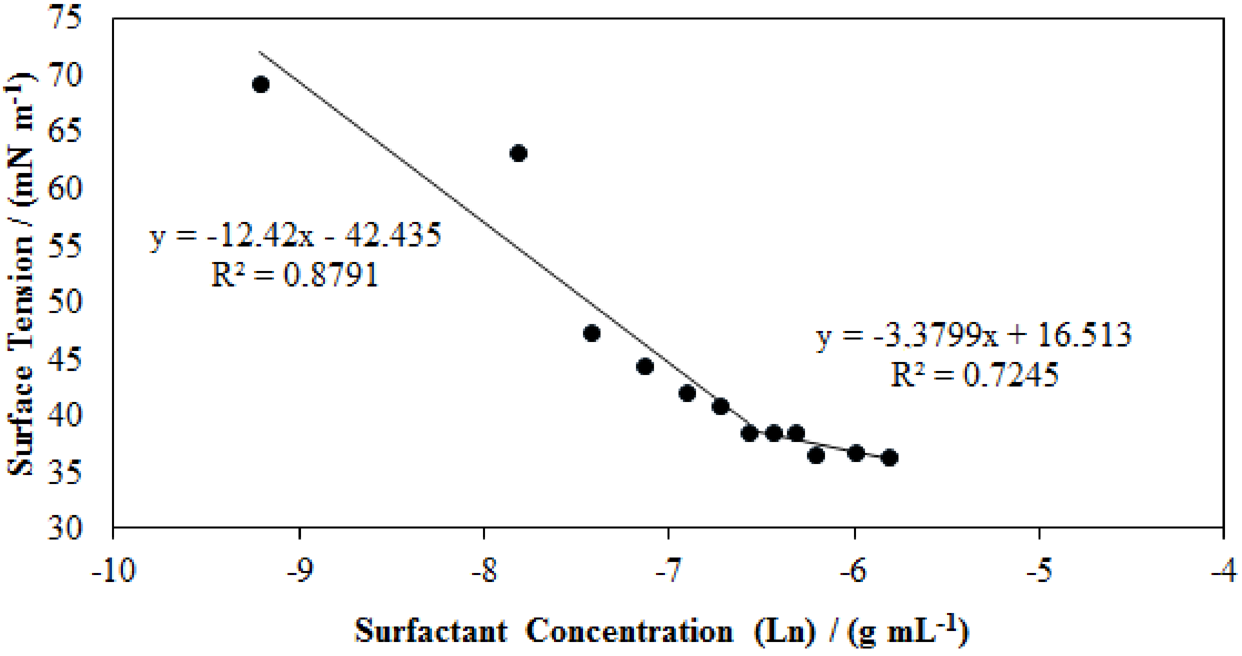

Figure 1. Variation of surface tension as a function of ln C for anionic surfactant SCO, at 27 °C.

Table II. **Average surface tensions of the SCO filtrates obtained after the finite-bath assays at 30 °C and 50 °C.**

Mass of limestone / g	Surface tension / (mN m^{-1})	
	30 °C	**50** °C
0.4	40.6	41.3
0.6	52.6	49.1
0.8	71.5	63.4
1.0	72.1	69.9

the substrate generate strong bonds among the species.

The adsorption curves for SCO on limestone as a function of temperature can be seen in Fig. 2 (30 ºC) and Fig. 3 (50 ºC). The maximal adsorption capacity was 0.36 g surfactant / g limestone, although the increase in temperature could only slightly decrease the adsorption rate in these experiments, with the exception of one point, whose adsorption values were similar (0.36 g surfactant / g limestone at 50 ºC and 0.35 g surfactant / g limestone at 30 ºC). This can be explained by the fact that this point has a lowest mass of adsorbent (0.4 g), possibly leading to saturation of the surface by surfactant molecules. In relation to decrease of adsorption with the increase of temperature, Gupta (1998) explains that molecules may be dessorbed from the surface because of increasing vibrational energies caused by increasing temperature, thereby reducing the adsorption efficiency.

The Langmuir and Freundlich adsorption isotherms were tested in the fitting of the experimental data. Table III shows the fitted equations and correlation coefficients as a function of temperature. It can be seen that the Langmuir model provided the best fit for both temperatures. Figure 3 shows plots of the experimental adsorption capacities for the Langmuir and Freundlich models at 30 °C.

It can also be seen in Fig. 2 that the Langmuir model is actually the best fit for the experimental data, which is an indication that the adsorption occurs in monolayers. In this adsorption model, a continuous monolayer of

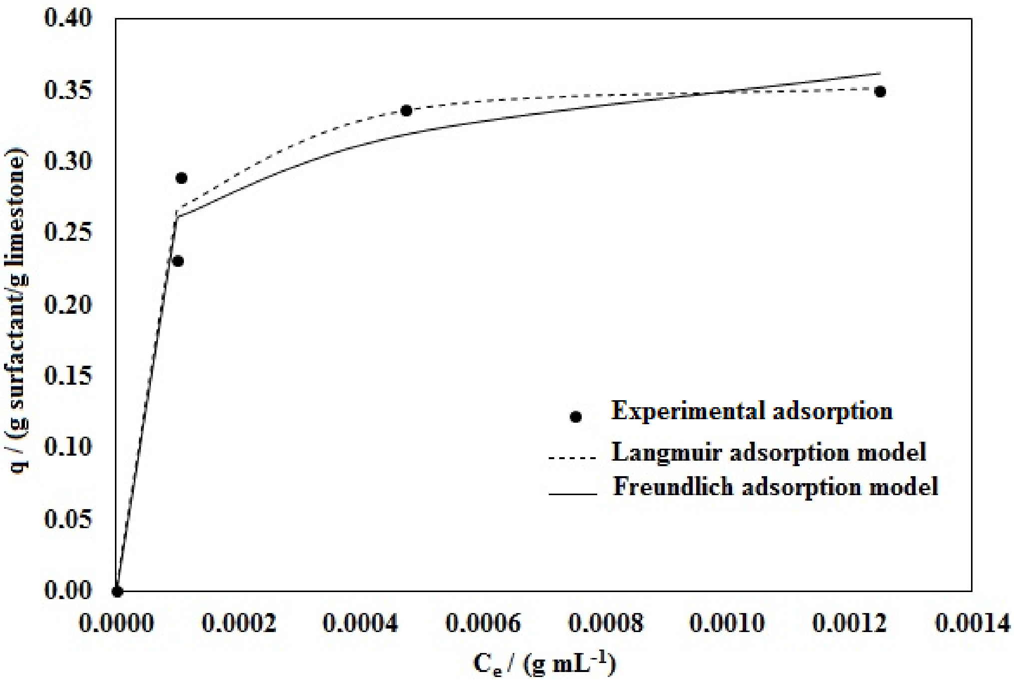

Figure 2. **Langmuir and Freundlich adsorption isotherms for the SCO solution (3 g L^{-1}) at 30 °C.**

adsorbate molecules is formed surrounding a homogeneous solid surface. In Figure 3, the same results are shown for the assays carried out at 50 °C. It is clear that the increase in temperature was again favourable to the Langmuir model.

Equilibrium time

The study on the effect of contact time on the adsoprtion capacity was carried out by fixing the mass of adsorbent at 0.8 g (see discussion given in Equilibrium mass referring to Table II) and the concentration of SCO in solution at 3 g L^{-1}. Figure 4 shows the results. At 30 °C, the adsorption capacity was initially low and reached a maximum at approximately 0.35 g surfactant / g limestone after 120 min. A slight decrease was then observed until the end of the experiment (240 min). A similar behaviour was observed at 50 °C, although the maximal adsorption capacity was reached with approximately 0.27 g surfactant / g limestone after 150 min, tending to slightly decrease and remain constant from 180 min to 240 min. It could also be demonstrated that the adsorption capacity decreased with increasing temperature.

Variation of SCO concentration

In order to expand the isotherms studies, the concentration of SCO in the aqueous solution was varied in experiments conducted at fixed contact time (2 h), mass of adsorbent (0.8 g) and temperature (30 °C). The results in Fig. 5 indicate that increasing SCO concentration enhanced the adsorption phenomenon. In Equilibrium time, it was shown that maximal adsorption capacity could be obtained with 0.35 g surfactant / g limestone after 120 min when a 3 g L^{-1} SCO solution was used. Between 3 g L^{-1} and 5 g L^{-1}, the adsorption is stabilized but can be further enhanced up to 0.70 g surfactant / g limestone with 8 g L^{-1}. The shape of the curve shown in Figure 5 indicates a tendency to the increase SCO adsorption with increasing concentration to values greater than 8 g L^{-1}, which may occur due to surfactant adsorption in multilayer.

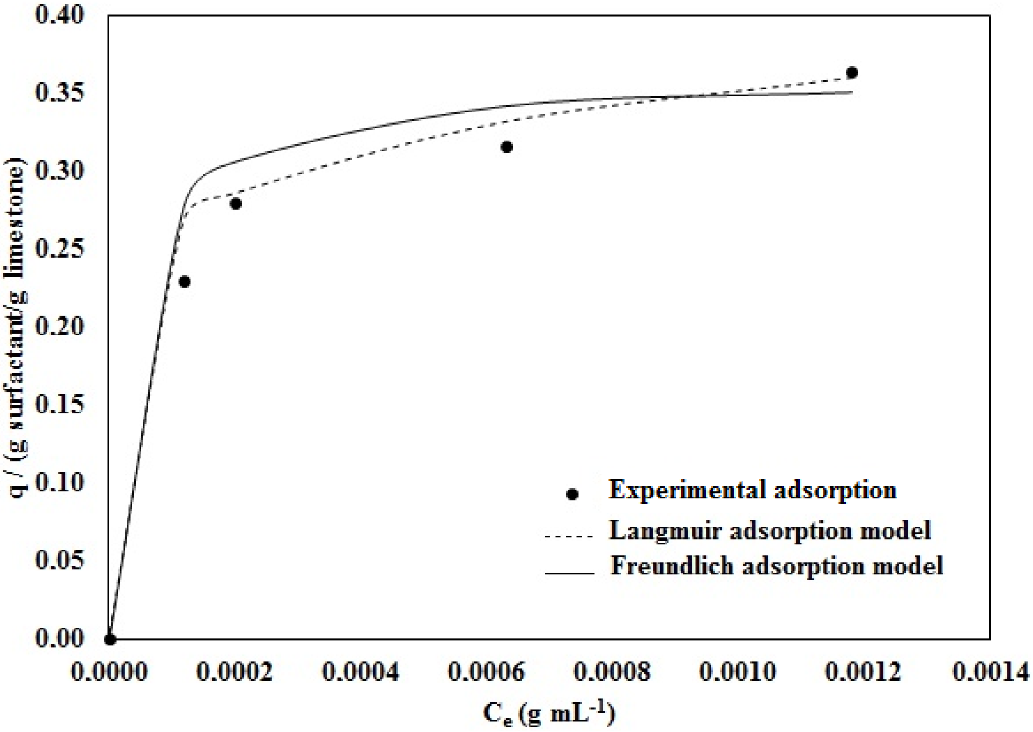

Figure 3. Langmuir and Freundlich adsorption isotherms for the SCO solution (3 g L^{-1}) at 50 °C.

Table III. Isotherm models for the SCO surfactant adsorption on limestone.

Temperature	Model	Correlation coefficient
30 °C	Langmuir, q = (27656*0.3616*C_e)/(1+27656*C_e) Freundlich, q = 0.8617*C_e^(1/7.7160)	0.9995 0.7375
50 °C	Langmuir, q = (12991.5*0.3849*C_e)/(1+12991.5*C_e) Freundlich, q = 1.2113*C_e^(1/5.5866)	0.9957 0.9472

Wettability assays

In order to determine the wettability of the rock and how it can be changed, contact angle measurements were made with tablets prepared with *in-natura* limestone and tablets made from the limestone samples collected in the filters after surfactant treatment in the finite-bath assays. Table IV shows the corresponding results.

It can be observed by the results that the original (*in-natura*) limestone samples were highly oil-wet, which normally occurs in most carbonaceous reservoirs and impairs oil production. The contact angles were actually low, varying between 11.9° and 12.7°. After treatment with the surfactant solution at 30 °C, the wettability was reduced towards oil, with an increase in the contact angle. At 50 °C, a slightly similar behaviour was observed, although the increase in the contact angle was lower than at 30 °C. However, it could be demonstrated that the treatment of the rock surface with surfactant solution effectively reduces its oil-wet character, which is a requirement for enhanced oil production, but the surface was not more hydrophilic because of such treatment. This was possibly due to the fact that only monolayer adsorption takes place with the amount of surfactant used. The direct electrostatic interactions between the surfactant anionic headgroups with the positively-charged calcium ions of the limestone samples drive the surfactant hydrophobic tails away from the surface, thereby impairing the contact of water molecules with the surface.

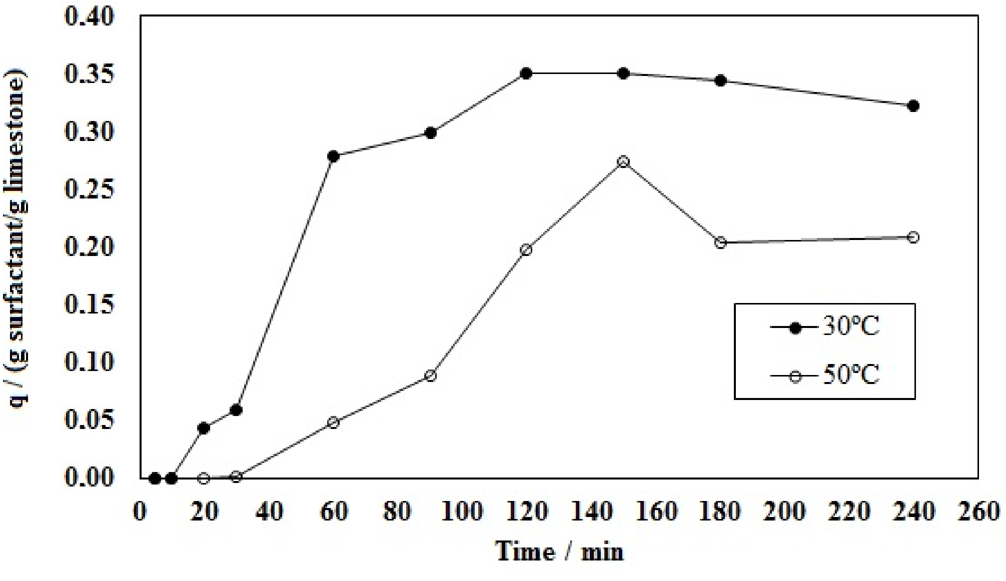

Figure 4. Adsorption capacity as a function of contact time for the SCO solution at 30 ºC and 50 °C.

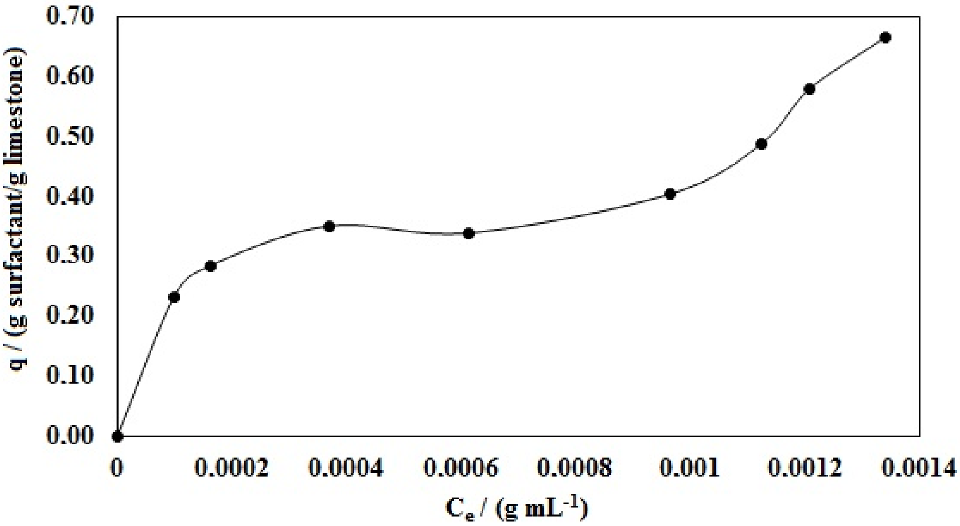

Figure 5. Adsorption isotherm obtained by varying SCO concentration at 30 °C.

Analysis of zeta potential

Changes in electrical charges on the solid surface could be detected by means of zeta potential measurements. Finite-bath experiments were conducted with the SCO solutions (3 g L^{-1} in 2 % KCl) and brine for 2 h, with 0.8 g of limestone in suspension at 30 °C. The results are shown in Table V. It can be confirmed that electrostatic interactions play an important role in the surfactant adsorption. The more-negative zeta potential of the SCO solution suggests enhanced adsorption. This is in agreement with reports by Wang et al. 2015, who changed the concentration of NaCl in the solution and observed that the adsorption capacity increased linearly with increasing zeta potential. Similarly, Andersen et al. (1991) shows the adsorption of two different anionic surfactants on limestone and concluded that both electrostatic (as in this work) and chemical interactions are involved in the adsorption mechanism. These authors obtained good surfactant adsorption capacity by varying the zeta potential even under positive values.

CONCLUSION

The main objective of this work was to investigate the adsorption capacity of SCO, an anionic surfactant obtained from coconut oil, on limestone. Some parameters that affect the adsorption mechanism have been examined, particularly with respect to changes in the rock wettability. Currently, literature references adsorption, wettability, and zeta potential parameters separately. This study innovates by examining these parameters together, comparing the results in a unique and interesting way. Therefore, it could be concluded that:

- Increasing temperature impairs the adsorption capacity;
- The SCO surfactant could be considerably adsorbed on the rock;
- The Langmuir adsorption isotherm was a better adjustment to the experimental data at 30 °C and 50 °C, indicating that monolayer adsorption takes place;
- By varying the contact time, it was possible to establish the equilibrium state of the adsorption phenomena within the time interval assessed;

Table IV. **Contact angle data for tablets made with *in natura* and SCO-treated limestone at 30 ºC and 50 ºC.**

Limestone sample	Tablet	Fluid	Contact angle (Θ)
In natura	1	Oil	12.7°
	2	Oil	11.9°
Treated with SCO (3 g L^{-1}) at 30 ºC	3	Oil	35.5°
	4	Oil	38.3°
Treated with SCO (3 g L^{-1}) at 50 ºC	5	Oil	34.4°
	6	Oil	29.2°

- By increasing the concentration of SCO, indicates that the adsorption may occur in multilayer;
- After treatment with SCO, the contact angle increased with respect to oil, which confirmed the reduction in the rock's oil-wet character;
- The zeta potential of the SCO solution confirmed that electrostatic interactions play an important role in the surfactant adsorption on the rock;
- The SCO surfactant adsorbed on the rock, reduced the rock's oil-wet character, and this adsorption was confirmed by zeta potential measurements. These results are important requirements for enhanced oil production because increase oil flow and recovery.

Acknowledgments

The authors would like to thank the CNPq – Conselho Nacional de Desenvolvimento Cientifico e Tecnológico – for the financial support.

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Table V. **Zeta potential data for the solutions on limestone.**

Solution	pH	Zeta Potencial / mV
KCl 2 %	9.86	– 4.61
SCO (3 g L^{-1})	10.55	– 38.68

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How to cite

NEVES AM, SANTANNA VC, BARILLAS JLM, CASTRO DANTAS TN, OLIVEIRA KC & GÓIS AGB. 2021. Investigations on the adsorption, wettability and zeta potential of anionic surfactant in limestone. An Acad Bras Cienc 93: e20190534. DOI 10.1590/0001-3765202120190534.

Manuscript received on June 26, 2019;
accepted for publication on May 17, 2021

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Author contributions

Vanessa C. Santanna conceived the original idea and supervised the project. Allan M. Neves and Alisson G. B. Góis carried out the experiments. Vanessa C. Santanna wrote the manuscript with support from Allan M. Neves, Jennys L. M. Barillas and Tereza N. Castro Dantas. Katherine C. Oliveira helped supervise the experiments of adsorption.