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Oil removal of oilfield-produced water by induced air flotation using nonionic surfactants

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ABSTRACT

Oil separation from produced water is commonly carried out at oilfields for resource recovery as well as environmental reasons. In this work, the separation of oil by induced air flotation was studied in a batch unit. A series of nonionic surfactants with different hydrophile–lipophile balance (HLB) values were used as a flotation agent. The effects of different HLB values and surfactant concentration on the oil removal of oilfield produced water were investigated. The removal rate constant (k) was determined and the flotation process followed first-order kinetics. The maximum oil removal obtained was approx. 80% after 4 min of flotation.

Keywords: Flotation; Oilfield, Produced water; Surfactant; HLB

1. Introduction

Produced water is an aqueous fraction generated by oilfields simultaneously with the production of oil and gas. Large amounts of this effluent are generated due to the water/oil ratio increasing over time and reaching values up to 95% of the volume produced [1]. The produced water originates from the following: the connate water, which represents the fraction of the water imprisoned in the rock pores during its formation—this water is without any contact with the atmosphere for a long geological time [2]; the formation water, which is the portion of water that is present in the rock immediately before the drilling process; the aquifers derived from the adjacent formations or directly linked to the rocks with hydrocarbons; and, finally, from the water and/or steam and aqueous solutions injected in the wells as recovery mechanisms.

This effluent presents dispersed oil-containing organic compounds, such as benzene, toluene, ethylbenzene and xylene (BTEX), phenols, naphthalene, phenanthrene, dibenzothiophene (NFD), polyaromatic hydrocarbons (HPA), metals, and a high concentration of inorganic salts [3]. Oil separation is one of the most important stages for treating these wastewaters, and the Total of Oil and Grease (TOG), as one of the main control parameters when this effluent is disposed of in the seabed. In the United States, the US Environmental Protection Agency (USEPA) [4] establishes as a

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monthly average 29 ppm of TOG and the daily limit of 42 ppm; the limit in Australia is 30 ppm as a daily average value, and it can reach a peak of 50 ppm [5]. In the Mediterranean countries, the monthly average is 40 ppm, and it can reach a peak of 100 ppm (Barcelona Convention and Protocols) [6]. In Brazil, the National Council on Environmental (CONAMA) [7] establishes 29 ppm as the maximum monthly value, and it can reach daily maximum values of 42 ppm.

Some methods have been employed for the treatment of oily effluents, such as biodegradation [8], flocculated magnetic separation [9], filtration [10], continuous electro-oxidation process [11], biosorption [12], flotation [13], and electrochemistry [14]. The advantage of employing flotation is in the transferring phase and the concentration of the dispersed and emulsified oil for a subsequent refining process. Motor oil removal from water by continuous froth flotation using extended surfactant has been investigated [15]. A commercial surfactant has been used as a collector to recover oil from synthetically prepared sludge containing oil through the induced air flotation (IAF) [16]. Sulfate sodium salt ($C_{14–15}(PO)_5SO_4Na$) has been used as an extended surfactant to form a colloidal gas aphron in order remove diesel oil from water by froth flotation [17]. The effect of the surfactant concentration on the treatment of an olive oil wastewater model solution by IAF has been investigated [18]. However, studies with the use of surfactants for the oil removal of oilfield-produced water are scarce.

The flotation process is started by a collision between the oil drop or dispersed particles in a continuous phase and gas bubbles. Then, the adherence of the oil drops or particles to the bubbles should occur and, through this, originate a bubble–drop aggregate that is transported up to the liquid surface. Finally, the enriched foam that is thereby formed is removed at the top of the flotation unit [19]. Therefore, the success of flotation depends on three stages: (1) collision between the gas bubbles and dispersed particles; (2) formation of the bubble–drop aggregate; (3) transport of the aggregate to the liquid surface (Eq. (1)) [20].

$$P_F = P_C \times P_A \times P_T \tag{1}$$

where P_C, P_A, and P_T are the probabilities of collision, adherence, and transport, respectively. In addition, it is known that the addition of metallic coagulants and polymers helps flotation by increasing the size of the dispersed particle and thus, favoring the probability of collision [21,22].

The aim of this work was to evaluate the TOG reduction kinetics of the produced water by IAF that was enhanced using a series of nonionic surfactants. In addition, the influence of the hydrophile–lipophile balance (HLB) of the different surfactants employed on separation kinetics was evaluated.

2. Materials and experimental methodology

2.1. Crude oil

In this study, crude oil from Potiguar Basin (Brazil) was used as a contaminant model. The oil was free of dissolved gas and water and its properties are summarized in Table 1.

2.2 Chemicals

The ethoxylated surfactants were supplied by Oxiteno®. Table 2 shows the HLB and molecular mass of each surfactant studied in this work. The other used reagents were of analytical grade, with a minimum purity of 99%. They were supplied by VETEC: sodium nitrate ($NaNO_3$), sodium chloride (NaCl), sodium sulfate (Na_2SO_4), potassium chloride (KCl), aluminum chloride ($AlCl_3$), magnesium chloride ($MgCl_2$), calcium chloride ($CaCl_2$), and chloroform ($CHCl_3$).

2.3. Apparatus and analytical procedure

Aeration by diffused air was applied using a compressed air stream through a porous plate with a pore size of between 16 and 40 mm in order to promote the formation of the desired bubbles. The column with a capacity of 1 L has the following dimensions: 0.80 m high, 0.040 m, and 0.042 m of the internal and external diameters, respectively (Fig. 1). In all of the experiments, the air flow was kept constant at 3,209 $cm^3 \ min^{-1}$ with the pressure of air injected at 5 psi.

Experiments were carried out employing the synthetic effluents prepared from the dispersion of

Table 1
Physical–chemical properties of oil studied

Physical–chemical property	Value
Density at 25˚C (g mL^{-1})	0.88
API gravity (˚API)[a]	27.00
Viscosity (cP)	65.00
Superficial tension (mN m^{-1})	29.95
Interfacial tension (water/oil) (mN m^{-1})	10.98

[a] Calculated according to the relation $^\circ API = \left(\frac{141.5}{d_{60F}}\right) - 131,5$, where d is the relative density.

Table 2
Physicochemical properties of the nonionic surfactants studied

Surfactant	Molecular formula	Molecular mass (g mol^{-1})	Hydrophile–lipophile balance (HLB)	CMC[1](mM)
EO3	$C_{12}H_{25}O(CH_2CH_2O)_3H$	318	8.3	–
EO7	$C_{12}H_{25}O(CH_2CH_2O)_7H$	494	12.5	$5.0 \times 0^{-2(a)}$
EO10	$C_{12}H_{25}O(CH_2CH_2O)_{10}H$	626	14.1	–
EO23	$C_{12}H_{25}O(CH_2CH_2O)_{23}H$	1,198	16.8	$9.0 \times 10^{-2(b)}$

[1] Critical micelle concentration. [a][23]. [b][24].

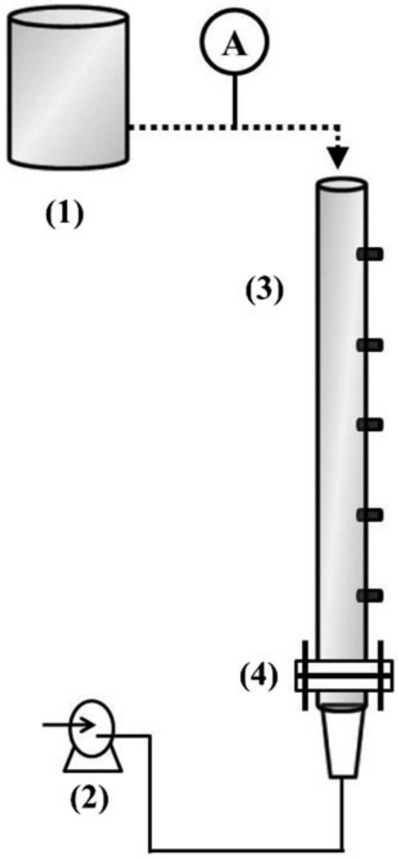

Fig. 1. Schematic diagram of the flotation apparatus used to reduce the TOG of produced water. (1) Tank containing synthetic wastewater, (2) compressor, (3) flotation column, (4) porous plate, (5) sampling point.

crude oil in a saline solution: 17 ppm ($NaNO_3$), 4,229 ppm (NaCl), 204 ppm (Na_2SO_4), 1,497 ppm (KCl), 2.35 ppm ($AlCl_3$), 1,506 ppm ($MgCl_2$), and 4,875 ppm ($CaCl_2$). The selection and concentration of the salts for the synthetic effluent used in this work were established from the average values found in the literature for produced waters in oilfields [4,25–29]. The mixture was mechanically agitated for 25 min at 33,000 rpm. Then, the effluent was kept at rest for 50 min in order to allow for the separation and removal of the free oil. Then, the surfactant was added to the effluent and transferred to the flotation unit. For all of the experiments, the initial TOG and pH values were measured after the addition of surfactant, and their values were kept constant at 300 ppm and approx. 7.0, respectively.

The flotation efficiency (η) was evaluated according to the reduction of TOG in the effluent (Eq. (2)), in which TOG_0 and TOG_t are the oil and greases concentration in initial time (t = 0) and in time t, respectively.

$$\eta(\%) = 100 - \left(\frac{\text{TOG}_t}{\text{TOG}_0} \times 100\right) \qquad (2)$$

The samples were collected in the central part of the column at predetermined times during the flotation process. The measurement of TOG in the aqueous phase was determined by the aid of liquid–liquid extraction, by employing chloroform as a solvent [30]. The concentration of oil in the extract was determined by the spectroscopy of molecular absorption based on a calibration curve gravimetrically prepared in the interval from 0 to 110 ppm. The interfacial tension oil–water (O/W) was determined by the drop method using a tensiometer (Kruss, DAS100 model).

3. Results and discussion

3.1. Flotation kinetics

The mathematical description of reducing the oil concentration according to time can be represented by Eq. (3). In all of the experiments, the air flow, temperature, pH, diameter of bubbles, and volume of the column were kept constant. The best adjustment of the experimental data was found by supposing the first-order kinetics (n = 1) (Eq. (4)).

$$\frac{dC}{dt} = -kC^n \tag{3}$$

$$\ln\left(\frac{C_0}{C}\right) = kt \tag{4}$$

where C_0 and C are the initial oil concentration and oil concentration at time t, respectively; k is the kinetic constant (min^{-1}), n is the order of separation process.

Fig. 2 shows a linearization of the oil removal results according to the concentration for each surfactant studied. The following concentrations were evaluated for each surfactant: 9.53×10^{-3}, 2.86×10^{-2}, 6.82×10^{-2}, 1.91×10^{-2}, and 1.21×10^{-1} mM. An experiment without surfactant was also carried out in order to compare the flotation performance with the presence of surfactants. The results showed a high separation rate during the initial 6 min, and then stabilization is likely. The first-order kinetic model assumes that the bubble–particle collision rate and bubble concentration remain constant over time [31]. First-order kinetics for oil/water separation has also been found by other researchers [32–34].

From Fig. 2(a) it is that only the concentration of 9.53×10^{-3} mM (k = 0.54 min^{-1}) and 2.86×10^{-2} mM (k = 0.30 min^{-1}) of EO3 surfactant has shown a high flotation performance when compared to the flotation in the absence of a surfactant (k = 0.24 min^{-1}). These results show that removing oil for the concentration of 9.53×10^{-3} mM of EO3 is twofold as high in relation to flotation without employing surfactants. For the conditions of 0 mM, 9.53×10^{-3} mM and 2.86×10^{-2} mM of surfactant, oil removals of 64, 80, and 69% were obtained after 4 min of flotation, respectively. However, the other surfactants (Fig. 2(b)–(d)) have shown separation efficiency equal or lower to the other obtained in the absence of surfactant. The higher flotation efficiency observed with the use of EO3 can be attributed to its low solubility in water (HLB = 8.3). The higher affinity of EO3 for the oily phase causes the migration of the surfactant molecules added in the aqueous phase, for the oil dispersed, helping in the

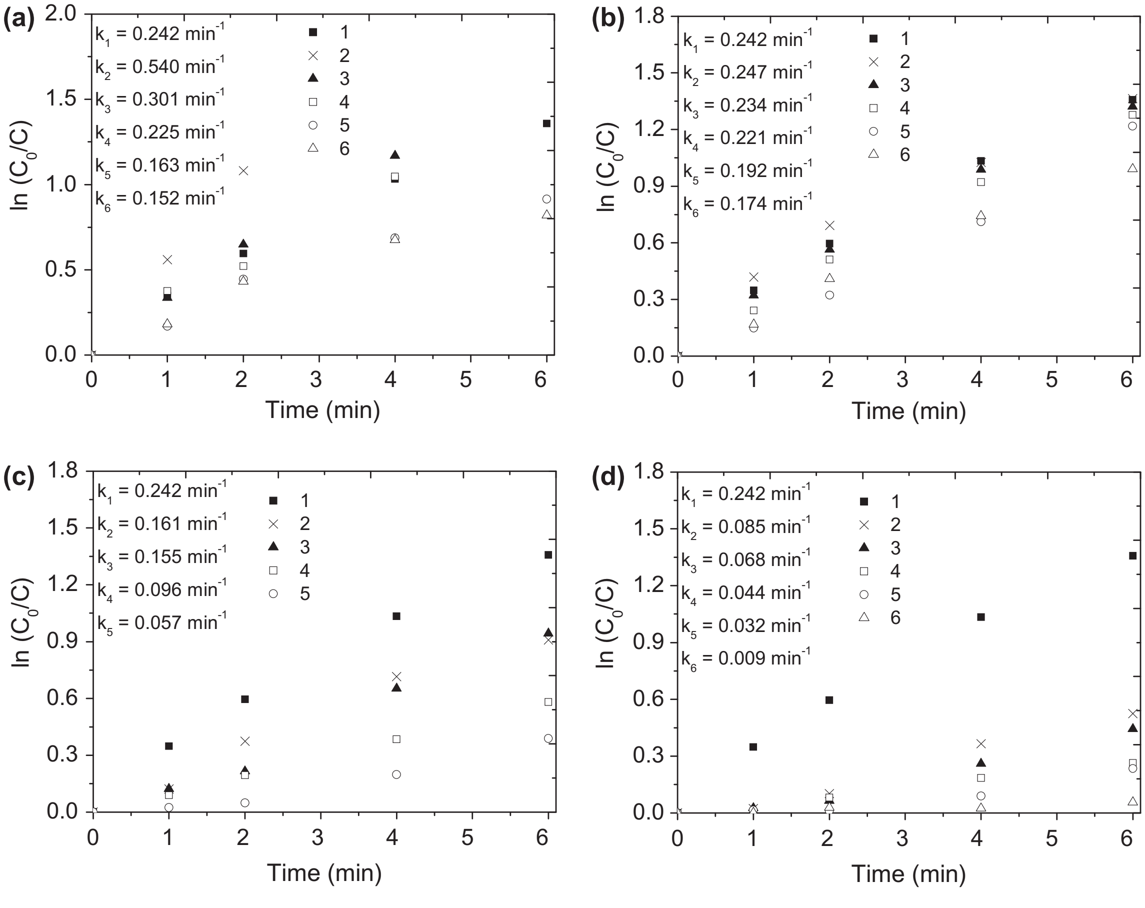

Fig. 2. Kinetic of oil removal by IAF for different nonionic surfactants as a function of concentration: (a) EO3, (b) EO7, (c) EO10, (d) EO2: Curve 1: absence of surfactant (0 mM); Curve 2: 9.53×10^{-3} mM; Curve 3: 1.91×10^{-2} mM; Curve 4: 2.86×10^{-2} mM; Curve 5: 6.82×10^{-2} mM; Curve 6: 1.21×10^{-1} mM.

rupture of the interfacial liquid film that is available among oil drops. The rupture of the interfacial film was not observed for the other surfactants. This behavior is attributed to the solubility of surfactants in the aqueous phase, where the HLB value is a variable of great importance in this behavior.

3.2. *Influence of the HLB value on O/W separation*

The hydrophilicity of the surfactants employed in this work comes from the ethoxy group present in their molecules. The higher the number of ethoxy groups, the more hydrophilic the surfactant is and the lower the number of ethoxy groups, the more lipophilic it is. The effect of HLB value and surfactant EO3 concentration on the separation kinetics is shown in Fig. 3. It is noteworthy that the lower the HLB is, the higher the separation efficiency will be. This result is attributed to the higher affinity of the surfactants with lower HLB, in the oil phase, corroborating the results presented by Roodbari et al. [35]. EO3 is a surfactant of lower HLB in the series studied (Table 2) and, therefore, it has higher solubility in the oil phase. Due to the low solubility of this surfactant in the aqueous phase, when it is added to the system, it moves to the interior of the oil bubbles until it reaches the Critical Micelle Concentration (CMC) in the oil, in which, from this point, to the extent that the surfactant concentration is increased, the monomers will take their position in the interface water–oil. Initially, this behavior is favorable because it helps in the film rupture. On the other hand, the excess of surfactant conducts to the saturation of the oil drop by the surfactant. The saturation of the interface represents the point in which the oil drop is covered with surfactants and with their heads oriented to the aqueous phase and, therefore, the oil drop becomes hydrophilic. Thus, the probability of adherence (Eq. (1)) is reduced and, consequently, the flotation efficiency is also reduced. The efficiency of the demulsification caused by the excess of surfactant was also observed by Goldszal and Bourrel [36], and it is attributed to the increase of rigidity of the interfacial film.

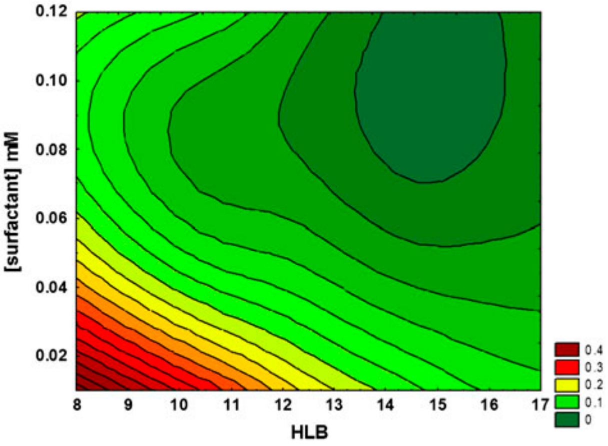

Fig. 3. The influence of HLB value and surfactant EO3 concentration on the kinetics constant (k) (expressed in min^{-1}) of oil removal.

Considering that the highest flotation efficiency goes to EO3, this surfactant was chosen to confirm the surfactant behavior in the emulsion previously mentioned through the curve of interfacial tension oil–water. Fig. 4 shows that, to the extent that the surfactant concentration is increased, the interfacial tension is reduced up to a constant value, while the kinetic constant shows a peak and, then, it decreases substantially. This fact occurs due to the fact that surfactants tend to take up a position in the interface thereby reducing the interfacial tension. When the surface of the oil drop is saturated with surfactant, there is a stabilization of the interfacial tension, which confirms the behavior previously explained. The probability of adherence includes the time, thinning, and rupture of the liquid film during the time of contact. Oliveira et al. [19] noted that the induction time that is necessary for the bubble–drop adherence and the oil spreading on the surface of the bubble is determined by the surfactant affinity regarding the phase in which it is dissolved. The results show that when a surfactant of low HLB value is present in the aqueous phase, the increase of the surfactant concentration reduces the induction time and increases the stabilization time. These authors have attributed this to the increase of surfactant molecules transferring through the interface, causing film rupture until the contact point. This favors the adherence and spreading of the oil drop on the bubble.

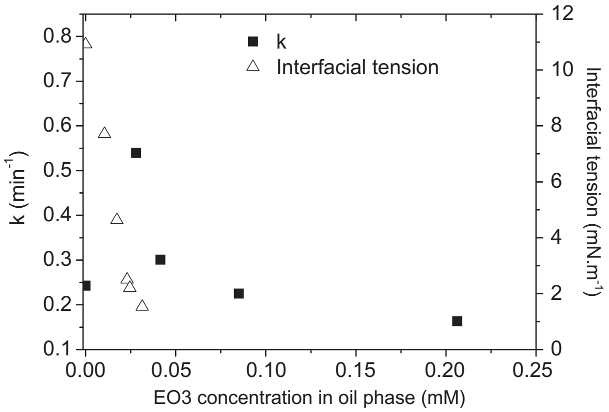

Fig. 4. Interfacial tension O/W and kinetic constant of oil removal as a function of EO3 concentration.

For the other surfactants, even for the lower concentrations used, the surfactant effect was inhibitory on the separation kinetics. This effect may be attributed to the higher affinity of EO7, EO10, and EO23 for the aqueous phase, which, when it is adsorbed in the oil drop, it impedes the coalescence of drops and, therefore, it stabilizes the emulsion [37]. The HLB of these surfactants is enough to emulsify the oil dispersed due to its preferential solubilization in the aqueous phase. Thus, it is possible to note that there is an optimum HLB value to favor the instability of the emulsion [38].

4. Conclusions

The results show that the efficiency of the separation oil–water (O/W) by flotation obeyed first-order kinetics and it depends on the concentration and HLB value of the surfactant. For all surfactants of the series studied, it was verified that the excess concentration of surfactant was detrimental to the kinetics of separation. The kinetics constant (k) decreases with the increase of the HLB value. The separation efficiency obeyed the following order: EO3 > EO7 > EO10 > EO23. The best result was obtained for the concentration of 9.53×10^{-3} mM (k = 0.54 min^{-1}) EO 3 (HLB = 8.3), removing 80% of the TOG after 4 min of flotation.

Acknowledgments

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