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Vacuum evaporation of surfactant solutions and oil-in-water emulsions

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ABSTRACT

Vacuum evaporation of surfactant solutions and oil-in-water (O/W) emulsions at laboratory-scale set-up was investigated. Experiments were performed with surfactant solutions and model emulsions formulated with a base oil (85–15% (w/w) mixture of a synthetic poly-α-olefin and a trimethylol propane trioleate ester, respectively) and the following surfactants: Brij-76 (polyethylene glycol octadecyl ether, non-ionic), CTAB (hexadecyltrimethyl ammonium bromide, cationic), or Oleth-10 (glycolic acid ethoxylate oleyl ether, anionic). Evaporation rates are strongly influenced by operating pressure and temperature. Surfactants enhance oil emulsification in water and increase the evaporation temperature and the water evaporation rate, especially at low pressures. Surfactant concentration effects depend on the type of surfactant. For surfactant solutions the evaporation rate is mainly controlled by the boundary layer which is formed at the solution surface, primarily by surfactants. For the O/W emulsions, the transfer of water to the liquid–vapour interface and the development of an oil boundary layer at the emulsion surface are also controlling steps. The chemical oxygen demand (COD) of the evaporation condensate was lower than 2% with respect to the original O/W emulsion/surfactant solution, which permits to recycle water in the closed-loop process.

1. Introduction

Metalworking fluids, in the form of oil-in-water (O/W) emulsions, are widely used in metalworking plants to provide lubrication, cooling, surface cleaning and corrosion protection. These fluids slowly deteriorate because of solids contamination, bacterial degradation and water evaporation. The disposal of these O/W emulsions and final wastewater treatment, for reuse or discharge, involve several processes.

Typical oily waste treatment methods are deep bed filtration, coagulation, flocculation, centrifugation, flotation and ultrafiltration [1–4]. The first step is to separate the free oil from the waste stream. However, dissolved and emulsified oil remain in the aqueous effluent and the oily phase may have high water content.

A common alternative to treat O/W emulsion effluents is to use a combination of a primary filter and an evaporator. Despite the wide use of evaporation in industrial processes such as water desalination [5,6], fruit juice concentration [7–10], sugar production [11,12] and landfill leachate treatment [13], few works have been performed on oily wastewaters treatment. Some studies have indicated the economic advantages of techniques such as membrane processes versus evaporation [14,15], because of the high energy costs associated with the latter technique.

Vacuum evaporation is a suitable alternative for the treatment of oily wastes, especially when it is attempted to recycle the water. This method is employed in industry, but very few studies have been reported [16–18]. The use of vacuum allows to reduce the evaporation energy consumption [18], especially when operating at low pressures, such as 8 kPa [6], 10–40 kPa [17,18], or 40–90 kPa [16]. Nevertheless, the quality of the resulting aqueous effluent is much higher for vacuum evaporation than for other treatments, such as ultrafiltration [18].

Evaporation of O/W emulsions is influenced by factors such as oil concentration, type of surfactant and operating conditions. As a general trend, surfactants decrease the evaporation rate, as it has been shown using either static equipments or heated surfaces [19–26]. Furthermore, the foaming ability of surfactants increases the contact area between water and air, thus enhancing the evaporation of organic compounds in the emulsion, as has been reported in studies employing an air stripping column [27]. The type of surfactant also affects the evaporation rate: water insoluble surfactants (*i.e.* with a long hydrocarbon chain) accumulate at the top of the liquid being evaporated, thus providing additional resistance to water evaporation [25–27]. It has also been reported that the evaporation rate of oil droplets in an O/W emulsion decreases with increasing droplet size [6,28,29].

Operating conditions are also important in O/W emulsion evaporation. High heat transfer rates increase the evaporation rate, with a subsequent decrease of the condensate quality [13,19,30,31]. The effect of operating pressure has also been investigated, at constant

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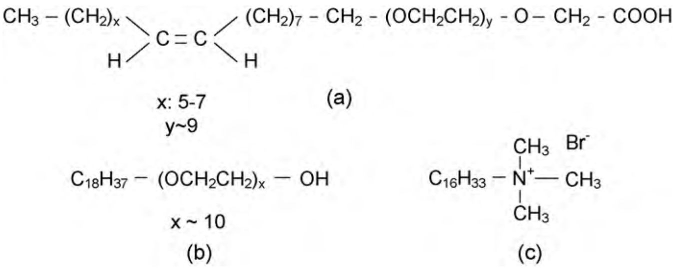

Fig. 1. Chemical structure of emulsifiers used: (a) anionic, (b) non-ionic and (c) cationic.

heat transfer rates [16,32]. No clear conclusion has been reached, but likely a decrease in the working pressure increases the condensate quality.

Laboratory-scale vacuum evaporation trials of several surfactant solutions and O/W model emulsions were carried out in this study. The effect of surfactants on evaporation parameters was studied, with the goal of obtaining high quality condensates. Experimental results are compared with those reported, under different operating conditions and surfactant concentrations, in a previous work [17]. Comparisons are made between the lower evaporation rates either for surfactant solutions or O/W emulsions containing non-ionic or ionic surfactants to establish the limiting evaporation steps.

2. Materials and methods

A mixture of a synthetic poly-α-olefin (PAO-6, Repsol-YPF, Spain) and a trimethylol propane trioleate ester (TMP, Fuchs Lubricantes S.A., Spain) in a ratio 85–15% (w/w), respectively, was used as the base oil for the formulation of the model O/W emulsions. Three surfactants (emulsifiers), supplied by Sigma–Aldrich Co. (Germany) with purities higher than 99%, were added to stabilise the emulsions: Brij-76 (polyethylene glycol octadecyl ether, non-ionic, CMC = 200 mg/L), CTAB (hexadecyltrimethyl ammonium bromide, cationic, CMC = 350 mg/L), or Oleth-10 (glycolic acid ethoxylate oleyl ether, anionic, CMC = 20 mg/L); their formulae are shown in Fig. 1. The critical micelle concentration (CMC) of the aqueous surfactant solutions was determined at 20 °C from surface tension measurements, using a Krüss K-8 tensiometer, following the Du Noüy's platinum ring method.

All the emulsions were prepared with 3% (w/w) base oil content and the emulsifier was added at concentrations of 0.5, 1.0, and 2.0 CMC (measured at 20 °C). The base oil was first blended with the emulsifier by mechanical stirring on a hot plate. This concentrate was subsequently dispersed in deionised water (Millipore Elix 5 deioniser) to reach the desired concentration and thoroughly mixed with a Heidolph DIAX 900 homogeniser at 10,000 rpm for 10 min.

Some tests were carried out using surfactant solutions with no oil and also with emulsions with no surfactant. Table 1 shows the COD values for the emulsions and solutions prepared in the laboratory.

Evaporation experiments were performed using a Büchi R205 evaporator described in previous works [17,18]. Evaporation rates of formulated O/W emulsions (E) and of pure water (E_W) were determined by measuring the mass of vapour condensed along time and collected in a receiving flask. The quality of the condensate was defined by its chemical oxygen demand (COD), determined by the reactor digestion method [33] using a Hach DR2010 UV spectrophotometer.

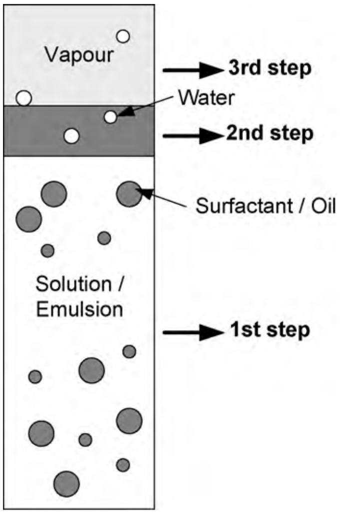

Fig. 2. Scheme of the three-step process in O/W emulsion evaporation.

3. Results and discussion

O/W emulsion evaporation can be described as a three-step process (Fig. 2):

- The first step consists of water transport to the top of the liquid sample, a process controlled by mass transfer. Repulsive forces between oil droplets (or surfactant micelles), droplet size and interfacial tension are important in this step.
- The second step is water transport through a thin layer of a secondary phase formed by oil or surfactant and is also controlled by mass transfer [21–23], a process influenced by stability and interfacial tension. This step does not happen with surfactants of high water solubility or for O/W emulsions with high stability during the entire evaporation process.
- The third step is vapour transfer from the top of the liquid phase or the thin layer of a secondary phase into the vapour phase. This depends on emulsion/solution properties such as surface tension, which may enhance or hinder the transfer rates.

The mass of water evaporated as a function of time follows a straight line whose slope is the evaporation rate of pure water (E_W). Surfactant solutions and O/W emulsions follow the same trend at least up to 70% (w/w) of the initial sample is evaporated. Further evaporation does not follow a linear trend, likely because water drops are trapped in the surfactant structure or by formation of reverse water-in-oil (W/O) emulsions. However, it was not possible to study the non-linear range for the concentrations used in this

Table 1
COD values (mg/L) of model O/W emulsions and surfactant solutions before being treated.

	Surfactant concentration									
	No surfactant	Non-ionic			Cationic			Anionic		
	0.0	0.5 CMC	1.0 CMC	2.0 CMC	0.5 CMC	1.0 CMC	2.0 CMC	0.5 CMC	1.0 CMC	2.0 CMC
Surfactant solution	~0	240	455	855	300	505	1200	25	45	85
O/W emulsion	1400	21,850	22,480	26,600	9130	13,250	17,750	1720	7680	8260

work. Initial concentrations of oil and surfactants were very low, and most of the emulsion was evaporated before the non-linear range was reached.

The relationship between E (emulsion evaporation rate) and E_w is given by:

$$c = \frac{E}{E_w} \tag{1}$$

where c ranges from 0 to 1, since the presence of surfactants and oil are expected to reduce the evaporation rate. This definition of c allows comparison of experimental evaporation rates obtained for surfactant solutions and O/W emulsions, even for experiments at different operating conditions, since all of them are referred to pure water evaporation at the same conditions.

3.1. Surfactant solutions

Below the CMC, all surfactants remain in solution and at the air–water interface, forming a layer which hinders water transport towards the air–liquid interface. Above the CMC the surfactants form micelles, and water transport decreases because of repulsive forces, particularly for ionic surfactants [34].

The evaporation temperature influences the surfactants CMC: as temperature increases the CMC decreases to a minimum value and then increases again. The minimum CMC value is normally reached at 25 °C for ionic surfactants and at 50 °C for non-ionic ones [34].

In this work the operating temperature was above 50 °C for all experiments and surfactant micelles are not likely to be found. Moreover, solubility of the non-ionic surfactant in water would decrease, especially when the cloud point is reached, leading to a thicker layer at the air–water interface. Despite few or no micelles are present at the operating temperature, CMC value at 20 °C will be used in this work for easier comparison of the surfactant concentrations on the formulated aqueous solutions and O/W emulsions.

The heating bath temperature was fixed at the lowest value of the solution boiling temperature at the operating pressure, to reduce energy consumption and to obtain a better quality condensate [13,19,30]. The selected operating pressures were 10 and 40 kPa, at a bath temperature of 100 and 140 °C, respectively. These correspond to a ΔT_{HE} value (temperature difference between the surfactant solution boiling temperature and the minimum heating bath temperature needed to achieve water evaporation) of 54 and 105 °C, respectively.

Fig. 3 shows c values for different surfactant solutions at 10 and 40 kPa. A decrease in c is observed when operating pressure increases (and thus, ΔT_{HE}) for non-ionic surfactants and the opposite effect is observed for ionic surfactants. As previously mentioned, the lack of non-ionic surfactant micelles, the tendency of that type of surfactants to remain at the air–water interface, and their water solubility decrease at high temperatures, might favour the formation of a thick surfactant layer at the top of the evaporating medium. This layer results in a clear separation between water and vapour phases, lowering water evaporation, especially at higher pressures and temperatures. However, the solubility of ionic surfactants is scarcely affected at high temperatures and may even increase. This indicates the top layer of ionic surfactant is thinner, or even might not exist, when the operating pressure increases.

Additionally, a decrease in c is observed for the non-ionic surfactant solutions when its concentration increases for both operating pressures. This agrees with the previous explanation, since the thickness of the top boundary layer will increase with the concentration of non-ionic surfactant. An increase in c is observed at increasing ionic surfactant concentrations, which confirms the high solubility of those surfactants at high temperatures. It may be concluded that for the evaporation of ionic surfactant solutions the top layer does not exist, and likewise the second aforementioned evaporation step. A drastic decrease in c is observed for experiments with 2.0 CMC of cationic surfactant concentration, at 10 kPa (Fig. 3a). This is likely because of the presence of some CTAB micelles, since the temperature is not as high as in the case of 40 kPa (Fig. 3b), and the repulsive forces between micelles could retard the evaporation.

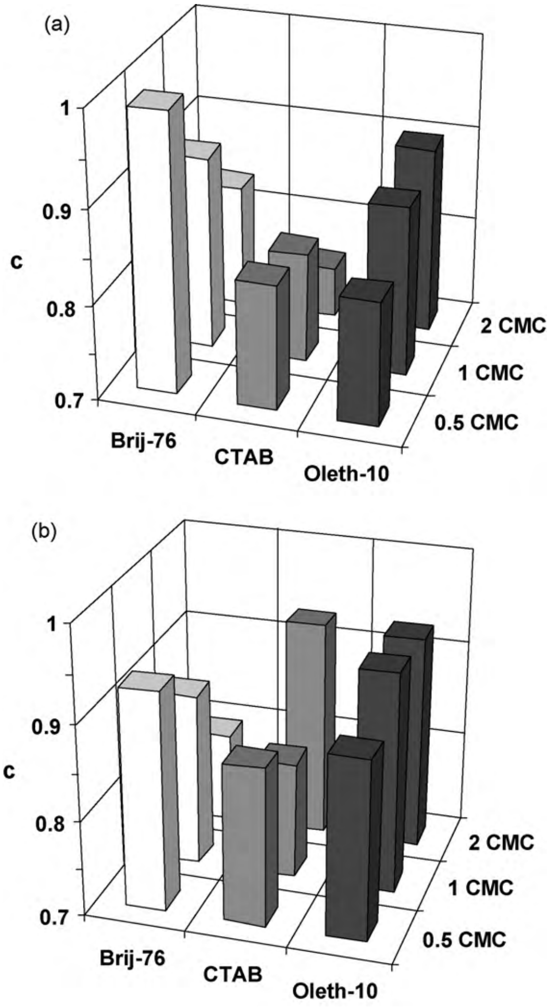

Fig. 3. Factor c values for surfactant solutions at different surfactant concentrations. Operating pressure: (a) 10 kPa and (b) 40 kPa.

Fig. 4 shows the COD values of the condensates obtained from evaporation of surfactant solutions at 10 and 40 kPa. The condensate quality from non-ionic surfactant solutions was better at 10 kPa than at 40 kPa, as increased thickness of the surfactant top layer at higher pressures increases the entrainment of surfactant to the vapour phase. However, for ionic surfactants, especially for the cationic surfactant, the opposite trend is observed: COD values of the condensates are lower when working at the higher pressure, due to the increase in solubility of the ionic surfactant in water, avoiding their entrainment to the vapour phase.

There is not a clear correlation between COD values and surfactant concentration, however CODs are very low, the range of surfactant concentrations in the condensate being: 4.5–11.0, 7.0–13.0, and 5.5–10.5 mg/L for non-ionic, cationic and anionic surfactant solutions, respectively. While the initial surfactant concentration differs greatly in each case, their concentration in the condensate is nearly the same, because of the different CMC values.

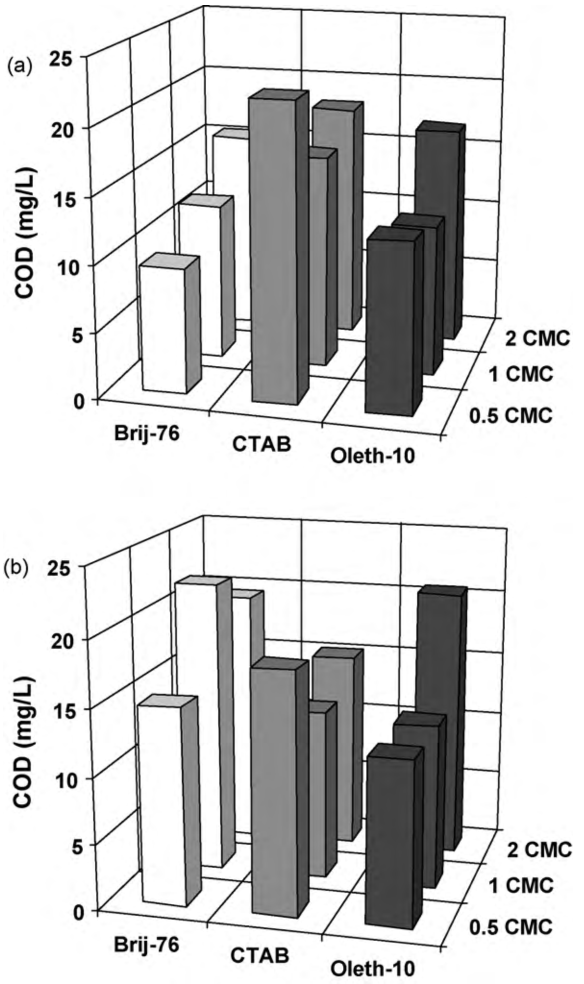

Fig. 4. COD content of the condensate (aqueous effluent) for the evaporation of surfactant solutions at different surfactant concentrations. Operating pressure: (a) 10 kPa and (b) 40 kPa.

3.2. Model O/W emulsions

The change of evaporation temperature (T_v) with time for pure water, surfactant solutions, emulsions without surfactants and surfactant-stabilised emulsions (the surfactant concentration was the CMC in all cases), at a pressure of 10 kPa, has been previously reported [17]. Similar experiments were carried out at a pressure of 40 kPa in this study and results for T_v and evaporation rates are shown in Figs. 5 and 6, respectively. Within the range of oil and surfactant concentrations studied, the following conclusions may be drawn: (i) the lower T_v values correspond to emulsions without surfactants; (ii) the higher the T_v, the faster the evaporation rate; (iii) evaporation rates for pure water are higher in all cases, as reported elsewhere [20,27]; and (iv) evaporation rates are lower for emulsions without surfactants than for surfactant-stabilised emulsions.

The type of emulsifier and its concentration also influence the evaporation process, since these factors affect emulsion properties, such as oil droplet size distribution, zeta potential and surface and interfacial tensions. Oil solubility in water greatly increases with increasing emulsifier concentration. Electrostatic repulsions or steric barriers between oil droplets also play a key role. The water film between droplets is closely related to these interactions, especially at high oil concentrations, *i.e.* at the end of the evaporation process [25,32]. Water films are thicker when ionic surfactants are employed [23,24], because of the high electrostatic repulsions between oil droplets. Furthermore, interfacial tension is also related to the ability of oil droplets to diffuse across the aqueous phase and vice versa and to coalesce forming larger oil droplets.

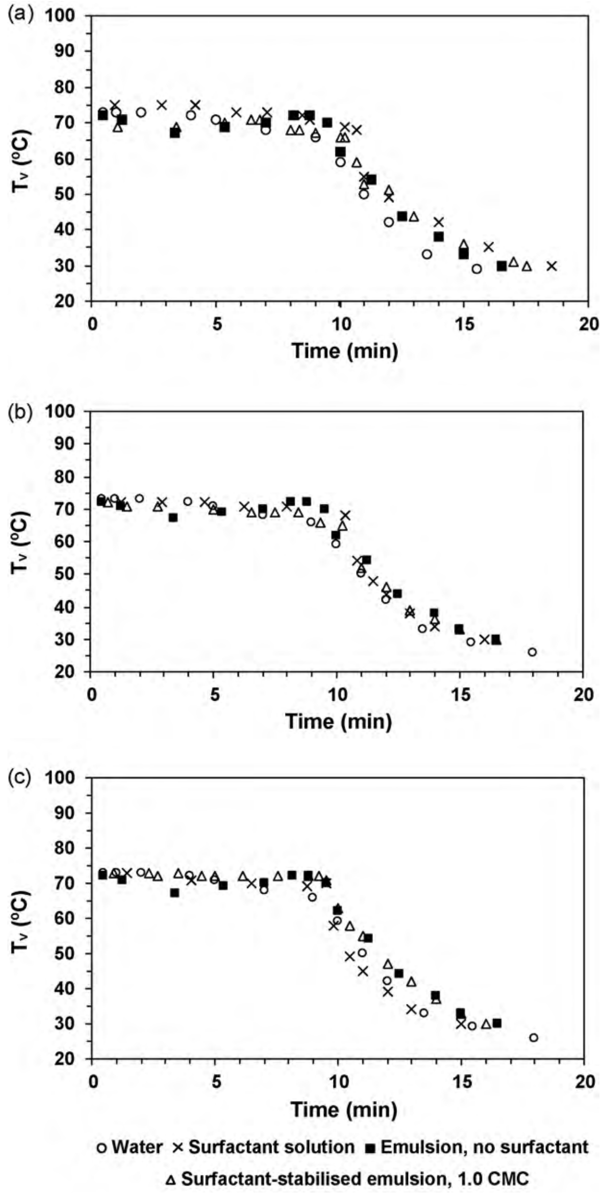

Fig. 5. Evolution of evaporation temperature (T_v) with time for pure water, surfactant solutions, emulsions without surfactants and emulsions stabilised with (a) non-ionic, (b) cationic, and (c) anionic surfactants. Operating pressure: 40 kPa; surfactant concentration: 1.0 CMC.

An increase in surfactant concentration may have the following effects on the evaporation rate of O/W emulsions: (i) higher surfactant concentrations may increase interactions between oil droplets that will hinder water transport through the water phase (first evaporation step); moreover, an increase in surfactant concentration leads to an increase of the thickness of the boundary layer (second evaporation step) [27], and hence, retarding water transport through the surfactant layer during evaporation; (ii) surface tension decreases with increasing surfactant concentration, which would enhance water evaporation (and also the third evaporation step) [20]; and (iii) changes in zeta potential with surfactant concentration may affect emulsion stability [35], and hence the appearance of the boundary oil top layer (second evaporation step).

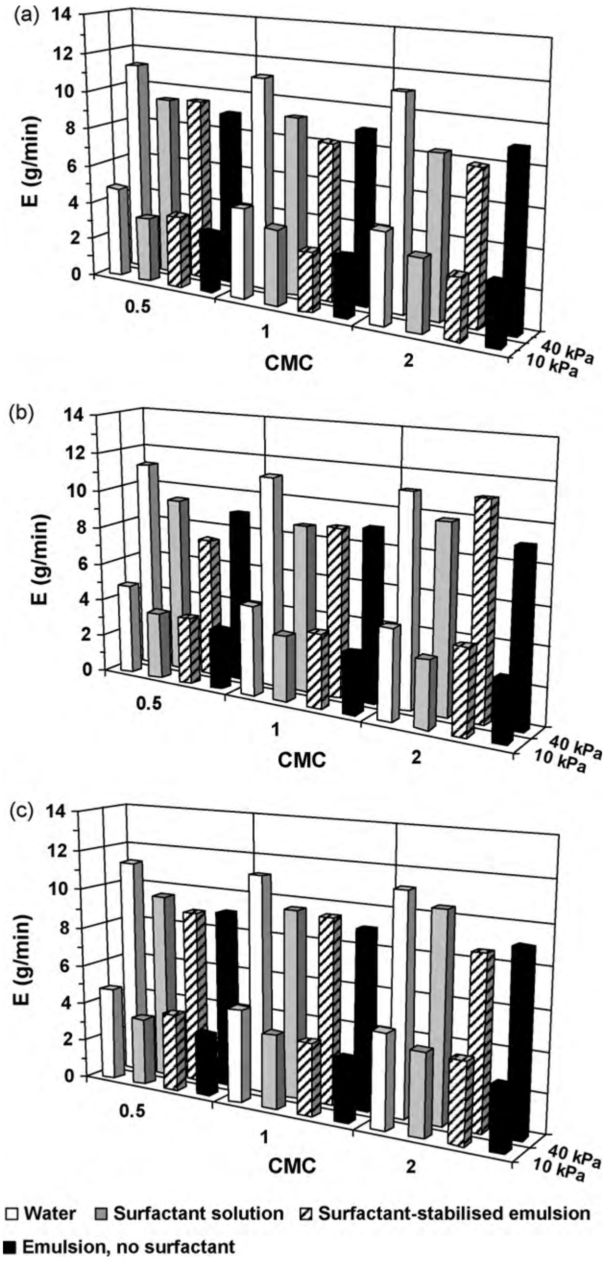

Fig. 6. Evaporation rate (E) at two different operating pressures for pure water, surfactant solutions, emulsions without surfactant and emulsions stabilised with (a) non-ionic, (b) cationic, and (c) anionic surfactants.

For emulsions without surfactants, an oil layer is formed at the top of the water phase and water has to cross this layer to reach the vapour phase. This results in lower evaporation rates which is more noticeable at 10 kPa. Fig. 6 also shows that a pressure change from 10 to 40 kPa leads to a 2.5-fold increase in the evaporation rate. Several factors might explain this behaviour. Firstly, heat transfer rates to the evaporation flask are higher at 40 kPa, since ΔT_{HE} increases from 54 to 105 °C. Furthermore, surface tension decreases as the temperature increases reducing the rate of bubble formation [13,19,20]. Moreover, the solubility of organic compounds increases at higher temperatures [27] and water encounters less resistance to diffuse and evaporate through the oil droplets or oil layers.

Factor c defined by Eq. (1) was also determined for model O/W emulsions evaporation. Emulsions without surfactants have lower c values than surfactant-stabilised emulsions and surfactant solutions, as shown in Fig. 7, and c values were also lower for evaporation at 10 kPa (Fig. 7a) than at 40 kPa (Fig. 7b). This might be explained because O/W emulsions are not stable at high temperature, and the oil top layer appears, being thicker at 40 kPa (corresponding to a $\Delta T_{\mathrm{HE}} = 105\,^{\circ}\mathrm{C}$). However, surface and interfacial tensions are lower at 40 kPa than at 10 kPa, and since both tensions decrease at increasing temperatures [35], and also the transport of water through the top oil layer, the water drops leave the surface at a higher rate at 40 kPa. This suggests that evaporation is controlled by water diffusion through the top oil layer and its transport through the liquid–vapour interface, for the model O/W emulsions studied.

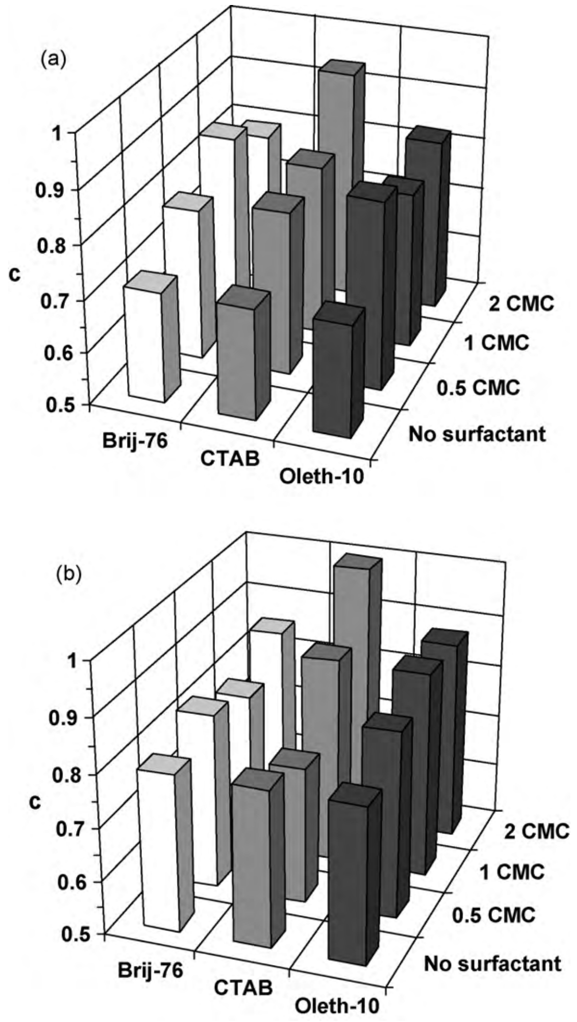

Fig. 7. Factor c values for emulsions with and without surfactants at different surfactant concentrations. Operating pressure: (a) 10 kPa and (b) 40 kPa.

No substantial differences in c values were observed for evaporation performed with several surfactant concentrations and different surfactants. The oil present in the emulsion seems to have a greater influence than surfactants. Only an increase in the c value was noticed for emulsions stabilised by the cationic surfactant (CTAB). The high foaming ability of this surfactant might explain this behaviour since the contact surface area increased because of the air bubbles (foam) formed, increasing the evaporation rate, as was previously reported [26]. Moreover, surface and interfacial tensions of cationic surfactant-stabilised emulsions are lower than for the other emulsions, facilitating water diffusion through the oil layer, and enhancing the detachment of water drops.

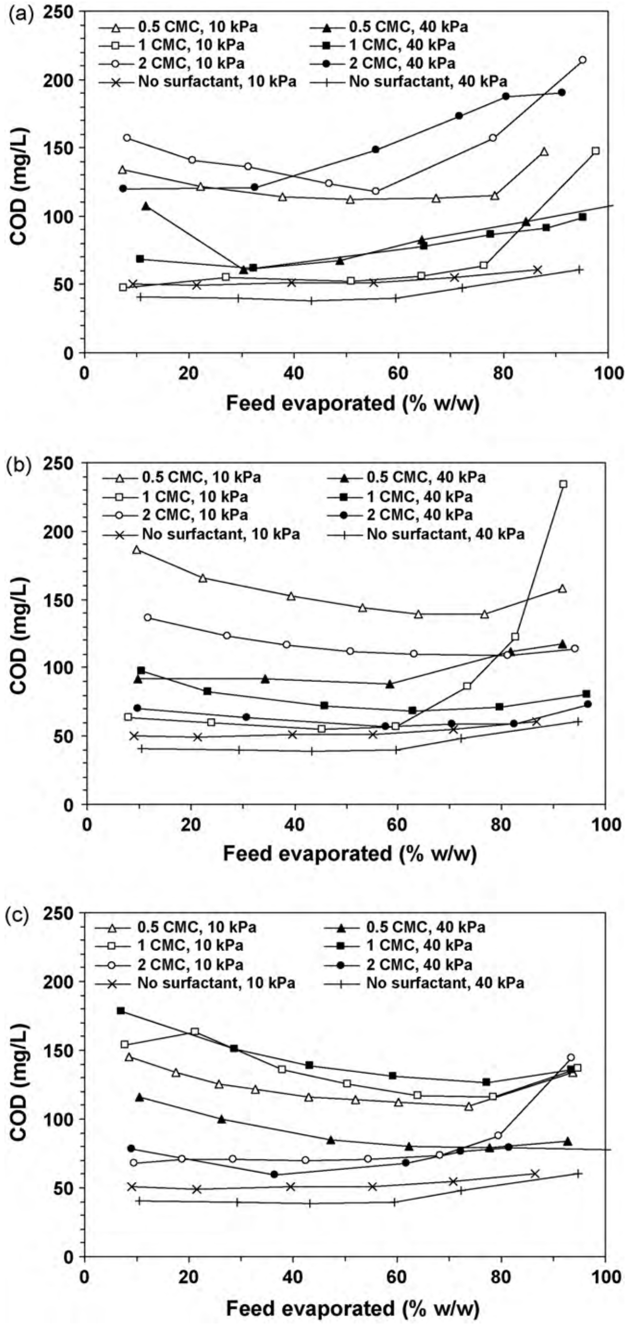

Fig. 8. COD content of the condensate (aqueous effluent) versus the feed evaporated for emulsions with and without surfactants at 10 and 40 kPa: (a) non-ionic surfactant, (b) cationic surfactant, and (c) anionic surfactant.

Surfactants influence the organic content of the evaporation condensate, as shown in Fig. 8. Condensates have a higher COD for emulsions stabilised with surfactants than in their absence. This is because the solubility of oil molecules in water increases through micelle formation. Visual inspection of the evaporation process reveals that the dispersed oil droplets cream to the surface, although they remain separated from the vapour phase by a thin water film [23]. The oil has to diffuse across this film to reach the vapour phase and micelles may enhance diffusion [22]. It is also apparent that surfactant solution CODs (Fig. 4) are very low compared with values for the O/W emulsions.

Operating conditions play a key role in O/W emulsions stabilised by a non-ionic surfactant (Brij-76) with a concentration of 0.5 CMC, as shown in Fig. 8a. Results differ for the two operating pressures. An increase in COD was observed when the surfactant concentration was increased from 1.0 to 2.0 CMC. The zeta potential tends to zero at those conditions [35] and the emulsion becomes unstable. Hence, the surfactant layer increases [26] and therefore oil and surfactants are easily entrained to the vapour phase increasing its COD value. It should be remembered that a non-ionic surfactant tends to be water insoluble at high temperature, and prone to go into the air–water interface, increasing the thickness of the oil layer.

Results for emulsions stabilised by the cationic surfactant (CTAB) are shown in Fig. 8b. At high surfactant concentrations COD values are lower. The decrease in surface tension is more pronounced in this case [35], and water can easily reach the vapour phase without entraining oil. This leads to a better condensate quality. However, Fig. 8b also reveals an increase in condensate COD at decreasing operating pressures. This finding is related to the aforementioned high foaming ability of this surfactant at all concentrations, especially at low pressures. Water molecules have to cross the foam layer, entraining surfactant molecules into the vapour phase.

Condensate CODs from emulsions stabilised by the anionic surfactant (Oleth-10) are shown in Fig. 8c. A more complex behaviour is observed, which is probably because of the combined effect of surface/interfacial tension, zeta potential and oil solubility changes. There is no clear correlation among these properties. A decrease in COD in the aqueous effluent is observed when the surfactant concentration increases from 1.0 to 2.0 CMC. This may be explained by a decrease in surface tension [35], while the zeta potential remains almost constant, which should result in a more selective evaporation process. It must be noted that the CMC value of the anionic surfactant – and therefore the concentrations used in this study – is much lower than the CMC of the other surfactants employed.

COD reductions in condensates higher than 98%, with respect to the original O/W emulsion/surfactant solution were achieved for all cases within the pressure, temperature and surfactant concentration ranges studied.

4. Conclusions

It is shown in this study that vacuum evaporation of surfactant solutions and oil-in-water (O/W) emulsions depends greatly on the pressure and the temperature. Surfactants decrease the water evaporation rate to an extent that depends on the type of surfactant.

For surfactant solutions, the non-ionic surfactant decreases the evaporation rate. This reduction enhances its entrainment to the vapour phase and lowers the quality of the condensate. Ionic surfactants also decrease the evaporation rate when their concentrations increase, but to a lesser extent than for non-ionic surfactant. A better quality (lower COD value) of the condensate is obtained.

Evaporation rates of surfactant solutions indicate a clear dependence on the second evaporation step, *i.e.* the water transport through the boundary top layer. Evaporation rate increases with increasing surfactant solubility in water.

The addition of surfactants to model O/W emulsions increases the evaporation rate in comparison with emulsions without stabilising agents. The condensate CODs are higher for emulsions stabilised with surfactants than when they are no present. However, the influence of their concentration on the evaporation rate is negligible.

The evaporation rate of O/W emulsions is lower at 10 kPa than at 40 kPa, indicating that evaporation is controlled by the second and third aforementioned steps.

There is not a clear correlation between the type of surfactant and the quality of the condensate, due to the several factors that affect evaporation. However, for the range of pressure, temperature and surfactants concentration studied, condensates COD reductions were higher than 98% with respect to the original O/W emulsion.

Experimental results also suggest that a proper selection of additives, mainly emulsifiers, is required for the correct formulation and subsequent evaporation treatment of metalworking O/W emulsions, to achieve high quality condensates. Vacuum evaporation of O/W emulsion reduces operation costs and enhances the evaporation rate, because of the intrinsic increase of emulsion stability at lower temperatures.

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