FULL LENGTH ARTICLE

Demulsification of W/O emulsion at petroleum field and reservoir conditions using some demulsifiers based on polyethylene and propylene oxides

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Abstract In this work, polymer molecules of alkene oxides diesters with varying HLB values and molecular weights (ED_1, ED_2, ED_3, PD_1, PD_2 and PD_3) were synthesized, elsewhere (Alsabagh et al., 2016). The demulsification efficiency was evaluated at field and reservoir conditions. At field conditions (60 °C and 1 atm) the data revealed that the maximum demulsification efficiency was obtained by ED_3 and PD_3 at 60 °C, 600 ppm after 55 and 40 min, respectively. At reservoir conditions (85 °C and 5000 psi), the PD_3 and ED_3 showed also the maximum demulsification efficiency was 76% and 70%, respectively, in spite of the 2% from the blank emulsion (12% BS&W) separated after 7 days. The interfacial tension (IFT) at the crude oil/water interface was measured for PD_3 and ED_3. From the results, it was found that the values of IFT were 0.7 and 0.8 mN m^{-1} respectively. The rheological behavior of the same demulsifiers was investigated. The results showed that the demulsifiers PD_3 and ED_3 enhance the dynamic viscosities (3.9 and 3.8 mPa s, respectively) and the (τ_B) yield values were 0.77 and 1.23 Pa s, respectively at temperature 85 °C, whereas, they were 3.95 mPa s and 1.5 Pa s for the blank emulsion sample.

1. Introduction

The emulsification of crude oil and brine/water is a common problem in the oilfield industry that is most frequently resolved through the use of chemical demulsifier additives.

Water-in-crude-oil emulsions are formed during the production of crude oil, which is often accompanied with water. The emulsions have stability ranging from a few minutes to years, depending upon the nature of crude oil and the extent of water. Natural surfactants such as asphaltenes, resins, carboxylic acids, and solids such as clay and waxes stabilize these emulsions. It is essential to break these emulsions before transportation through pipelines and prior to refining [1–6].

According to Bancroft (1913), the stability of any emulsion is largely due to the nature of the interfacial film that is formed. The stability of this film is strongly dependent upon the surfactant adsorption–desorption kinetics, solubility, and interfacial rheological properties such as elasticity, interfacial tension gradient and interfacial viscosity [7]. Crude oil free from water is required for pipeline flow and refinery operation. Chemical demulsification forms the most important step in demulsification of water-in-crude oil emulsions.

Demulsification is a process, which involves flocculation, coalescence and separation of two immiscible liquids. Chemical demulsification is a process in which the film-thinning rate is enhanced and stability of the film is reduced by a chemical demulsifier. It has been established that the role of the demulsifier is to change the interfacial rheological properties and to destabilize the surfactant-stabilized emulsion films [8–13]. Demulsifiers are all polymeric surfactant capable of adsorbing at the oil–water interface and displacing the interfacial film formed. Asphaltene in the oil possess a sufficient number of functional groups to form an interfacial layer of great mechanical strength. Demulsification can take place only when an agent breaks the interfacial layer whose surface activity is superior to that of the asphaltenes. Several factors affect the demulsification process, some of them being dependent on the characteristic of the continuous phase, such as viscosity, and asphaltenes content, others depend on the structural modification of the demulsifier molecules.

Commercial demulsifiers are polymeric surfactants such as polyoxyethylene and polypropylene of different surface-active substances. These demulsifiers are surface-active agents and develop high surface pressures at crude oil/water interfaces [14,15]. This results in replacement of rigid films of natural crude oil surfactants by a film which is conducive to coalescence of water droplets. The efficiency with which a surfactant acts as a demulsifier depends on many factors related to the structure of the surfactant. Such factors include the distribution of the demulsifier throughout the bulk volume of the emulsion, the partitioning of the demulsifier between the phases, and the temperature, pH, and salt content of the aqueous phase. Other factors of importance are the mode of injection of the demulsifier, the concentration of the demulsifier, the type of solvent carrier, the amount of water in the emulsion, and the age of the emulsion [16–20].

In the present work, the first object of this work is to investigate the demulsification efficiency of the prepared polymers from alkene oxides diester at field and reservoir conditions. The second object is to study the interfacial tension and the rheological properties for the treated emulsions.

2. Experimental

2.1. Materials

The preparation of the used demulsifiers in this paper was investigated elsewhere [1], the abbreviations, structures, HLB, molecular weights and the ratio of ethylene and propylene oxides of the used demulsifiers are shown in Table 2.

2.2. Crude oil used

The waxy crude oil (BS&W = 12% naturally occurrence) was conducted from Qarun Petroleum Company, Egypt. The specifications of crude oil are shown in Table 1.

2.3. Interfacial tension (IFT)

The interfacial tension as a function of the concentration of the active component of the demulsifier in the model system was measured at (60 °C) using De-NoüyTensiometer (Kruss-K6 type) and applying a platinum ring technique. Before each measurement the scratched platinum was well washed and fired over a Bunsen burner flame to ensure proper wettability [21–28].

2.4. Demulsification performance

2.4.1. At petroleum field temperature

The graduated bottle test (Sany-glass) was used to estimate the efficiency of demulsifiers toward resolving water-in-oil emulsions. Each demulsifier, was dissolved in xylene (10% active material), and then added into 100 ml of the emulsion at different concentrations (ppm). The mixture was added into a 100 ml Sany-glass and then was shacked vigorously for 1 min (60 times). The bottle was placed in a thermostated water bath at 40° and 60 °C. Water separation (in ml) is observed and

Table 1 It shows the specification Qarun north-7 crude oil.

Experiment	Method	Result
Density@15.56 °C		0.8201
Specific gravity	ASTM D-4052	0.8209
API gravity@60 °F		40.87
Kinematic viscosity, cSt, @ 40 °C	ASTM D-445	5.05
Total sulfur, wt.%	ASTM D-4294	0.24
Asphaltene content, wt.%	IP-143	2.56
Wax content, wt.%	UOP-64	1.05
Pour point, °C	ASTM D-97	24
Water content, vol.%	ASTM D-95	12
BS&W, vol.%	ASTM D-96	0.2
n-Paraffins, wt.%	ASTM D2887 (GLC)	27.5
Iso-paraffins, wt.%	ASTM D2887 (GLC)	22.4
Total paraffin content, wt.%	ASTM D2887 (GLC)	49.9
Average carbon number (n)	IP 372/85 (GLC)	18.34

Table 2 Name and abbreviation of prepared polymers.

Name and abbreviations	Structure	HLB	M.W.	Alkylene oxide	
				e.o. unit	p.o. unit
Polyethylene oxide of oleate Methacrylate diester (ED$_1$)	[C$_{17}$H$_{32}$CO(OCH$_2$CH$_2$)$_9$OCOCH$_2$CH$_2$]m	1.7	4943	9.39	0.0
Polyethylene oxide of oleate Methacrylate diester (ED$_2$)	[C$_{17}$H$_{32}$CO(OCH$_2$CH$_2$)$_{14}$OCOCH$_2$CH$_2$]m	1.8	7231	14.38	0.0
Polyethylene oxide of oleate Methacrylate diester (ED$_3$)	[C$_{17}$H$_{32}$CO(OCH$_2$CH$_2$)$_{24}$OCOCH$_2$CH$_2$]m	1.9	12088	24.61	0.0
Polypropylene oxide of oleate Methacrylate diester (PD$_1$)	[C$_{17}$H$_{32}$CO(OCHCH$_3$CH$_2$)$_7$OCOCH$_2$CH$_2$]m	0.02	5964	0.0	7.98
Polypropylene oxide of oleate Methacrylate diester(PD$_2$)	[C$_{17}$H$_{32}$CO(OCHCH$_3$CH$_2$)$_{13}$OCOCH$_2$CH$_2$]m	0.02	7362	0.0	13.86
Polypropylene oxide of oleate Methacrylate diester (PD$_3$)	[C$_{17}$H$_{32}$CO(OCHCH$_3$CH$_2$)$_{21}$OCOCH$_2$CH$_2$]m	0.02	14450	0.0	21.81

Where e.o represents ethylene oxide and p.o represents propylene oxide.

recorded at different time intervals depending on the efficiency of the demulsifier under investigation [29–33]. A blank sample was considered in each set of experiments

$$\text{Water separation} = (v/v_o) \times 100\% \qquad (1)$$

where v is the volume of the separated water and v_o is the original volume of water content in the crude oil.

2.4.2. At reservoir conditions

A sample of 250 CC of crude oil emulsion is placed in a sample bottle, where the crude oil emulsion (S$_1$) is obtained from Qarun Petroleum Company (12% BS&W) from a depth of 4200 ft. The temperature was adjusted to 85 °C and pressure up to 1700 psi (reservoir conditions). Demulsifier is injected into the crude oil emulsion at a concentration of 500 ppm. The fluid inside the cylinder is agitated for 3 h using an agitator. The container was kept under reservoir conditions for 48 h. The separated water was then determined and the efficiency is calculated.

2.5. Hydrophile–lipophile balance HLB

The demulsifier ingredients are chemicals that have surface-active properties characterized by hydrophilic–lipophilic balance (HLB) values. The HLB scale varies from 0 to 20. The HLB values of the prepared demulsifiers were calculated according to the following equation [34];

$$\text{HLB} = 20[M_H/(M_H + M_L)]$$

where M_H is formula weight of the hydrophilic portion of the molecules and M_L is the formula weight of the lipophilic (hydrophobic) portion of the molecule.

2.6. Rheological properties

Oil viscosity was measured using rheometer programmable UV-III with a heating/cooling water bath. The crude oil used in this study was considered as non-Newtonian fluid because the viscosity of the crude oil changes while being measured with a rheometer at different speed rotation. The viscosity measurement started from low shear rates. The shear rate was then increased at constant temperature (40, 60 and 85 °C). The rheological properties were measured over a shear range of 1–1200 s. The flow curve can be analyzed using the Herschel–Bulkley equation [35,36]:

$$\tau = \tau_B + kD$$

where τ is the shear stress; τ_B is the dynamic yield stress "Bingham yield value"; k is the consistency index; m is the shear thinning index and D is the shear rate. The Bingham yield value (τ_B) can be obtained from the intercept of the graph relation between shear rate and shear stress. On the other hand, the linear line from the relation between shear rate and viscosity gives us the dynamic "apparent" viscosity.

The rheology parameters were obtained from the following equations:

$$\text{Shear stress, } \tau = A \cdot S(\text{Pa})$$

$$\text{Viscosity, } \eta = G \cdot S/n(\text{mPa s})$$

$$\text{Shear rate, } D = M \cdot n(\text{s})$$

wherein n = number of revolution, rpm; S = Torque; $M = 2.34$ (min/s); $A = 3.22$ (Pa/S K t) and $G = 1347$ (mPa s/S K t min)

3. Results and discussion

3.1. Demulsification efficiency

The demulsification efficiency data for the prepared demulsifiers individually are shown in Table 3 while Fig. 1 illustrates the demulsification efficiency for the diesters of oleate methacrylate polyethylene oxide and polypropylene oxide polymers. By inspection of the obtained data, it was found that the separation of the blank crude emulsion was very slow 30% demulsification efficiency was exhibited after 38 days while the maximum demulsification efficiency was obtained by PD$_3$ > PD$_2$ > ED$_3$ after 40, 45 and 55 min, respectively at 60 °C.

Some investigators (16–19) have studied the relationship between the molecular weight of the demulsifiers and their efficiency and they found a direct relation between the molecular

Table 3 The maximum demulsification efficiency (100%) at different concentrations and temperatures for the investigated demulsifiers individually.

Demulsifier	Temperature (°C)	100 ppm (min)	200 ppm (min)	300 ppm (min)	400 ppm (min)	500 ppm (min)	600 ppm (min)
ED_1	60	300	280	250	240	225	210
	85	210	165	100	95	90	75
ED_2	60	200	195	170	155	130	120
	85	140	110	95	90	85	70
ED_3	60	170	165	130	100	90	80
	85	130	105	85	75	70	55
PD_1	60	150	140	100	90	80	65
	85	100	90	85	80	75	60
PD_2	60	135	120	80	80	70	60
	85	90	80	75	65	60	45
PD_3	60	110	100	90	70	60	50
	85	85	80	75	60	50	40

weight and the efficiency of demulsifier, where the molecular weight is varying as; $PD_3 > PD_2 > PD_1$ and $ED_3 > ED_2 > ED_1$. The PD_3 exhibited complete water separation at times; 80, 60 and 40 min against concentrations 200, 400 and 600 ppm at 60 °C. The ED_1, ED_2, ED_3, PD_1 and PD_2 exhibited complete water separation also, but at high concentration and time taken was greater than that which was consumed with the PD_3 (40 min at conc 600 ppm) as shown in Table 3. The increase in chain length of the demulsifier molecule (ethylene and propylene oxide chain) decreases the HLB and makes the molecule more oil soluble specially with propylene oxide and increases the interfacial activity that suppresses the interfacial tension gradient, which accelerates the rate of film drainage hence promoting coalescence. The resulting demulsifiers were prepared from macromonomers of ethylene oxides with molecular weights of 430, 650 and 1100. This finding was revealed that, the increase of molecular weight increases the demulsification efficiency. The same was seen for those prepared from propylene oxide with molecular weights 425, 725 and 1130 as in Table 4.

The demulsification efficiencies of PD_1, PD_2, PD_3, ED_1, ED_2 and ED_3 at different concentrations and different contact times have been investigated as shown in Table 3. The contact time of the demulsifier at which they exhibited 100% water separation was situated between 40 and 300 min. These efficiencies were obtained against concentrations from 100 to 600 ppm. The increase of concentration may lead to increase in the adsorption of the demulsifier molecules on the water/oil interface. This may effect the W/O interface properties and leads to destabilize the interface layer and break the emulsion into water and crude oil. The data in Table 3 reveals that, the demulsification efficiency increases with increasing the contact time and concentration.

In this respect; the demulsifier can be ranked according to their ability to break the O/W emulsion as: $PD_3 > PD_2 > ED_3 > PD_1 > ED_2 > ED_1$. This is may be due to the increasing of molecular weight (M.W.) as a function of polyethylene or polypropylene oxides. The propylene oxide in the skeleton structure of the polymer played an important role to place the demulsifier PD_3 in the former citation based on the demulsification efficiency comparing with the other used demulsifiers.

The increase of temperature is not sufficient to produce a desirable effect on emulsion stability without demulsifier. W/O emulsions are much more viscose than the crude oil itself. The increase in viscosity will hinder the diffusion rate to coalescence the water droplets. The increase of temperature from 60 to 85 °C resulted in a substantial decrease in the viscosity which leads to ease the adsorption of the demulsifier molecules on the interface [37] this concept is clear in Table 3 regarding to PD_3, which exhibited 100% separation at 85 °C after 40 min, while the time was 50 min at temperature 60 °C. Meanwhile the ED_3 exhibited the maximum efficiency of 100% after 80 min at 60 °C and after 55 min at 85 °C.

The blends of demulsifiers were based basically on mixing two demulsifiers in the three different ratios; 3:1, 1:1, and 1:3. It was found that, in general, the blends (mixtures) exhibited greater efficiency than that obtained by the individual demulsifiers of the mixture. Table 5 and Fig. 2 shows that, the blends have a positive synergetic property toward water separation from the emulsion. The increase of polypropylene oxide (PPO) ratio in the mixture enhanced the demulsification efficiency. The best demulsification efficiency was achieved by using blend of ED_3 and PD_3 (M3) in ratio 1:3 with concentration 400 ppm meanwhile, the best results achieved by ED_3 and PD_3 separately were 75 and 60 min with the same concentration (400 ppm). These results agreed with other investigators [38–40].

The HLB concept, is normally used as an important parameter to predict the action of demulsifiers on a certain water- in-oil emulsion. In the present study, the HLB value of blends were varied by altering the degree of propoxylation and ethoxylation

$$HLB_m = HLB \cdot X_1 + HLB \cdot X_2$$

where X_1, and X_2 are the weight fraction of the surfactant (demulsifier) present in the mixture. The calculated values of HLB for the mixtures of the undertaken demulsifiers are presented in Table 5. It is observed that, the HLB of the surfactants increases with increasing the ratio of ethylene oxide relative to propylene oxide. Accordingly, it is obvious that both HLB depend primarily on the degree of propoxylation. The data revealed that the amount of water separated after a certain time, expressed as percentage coalescence, is in
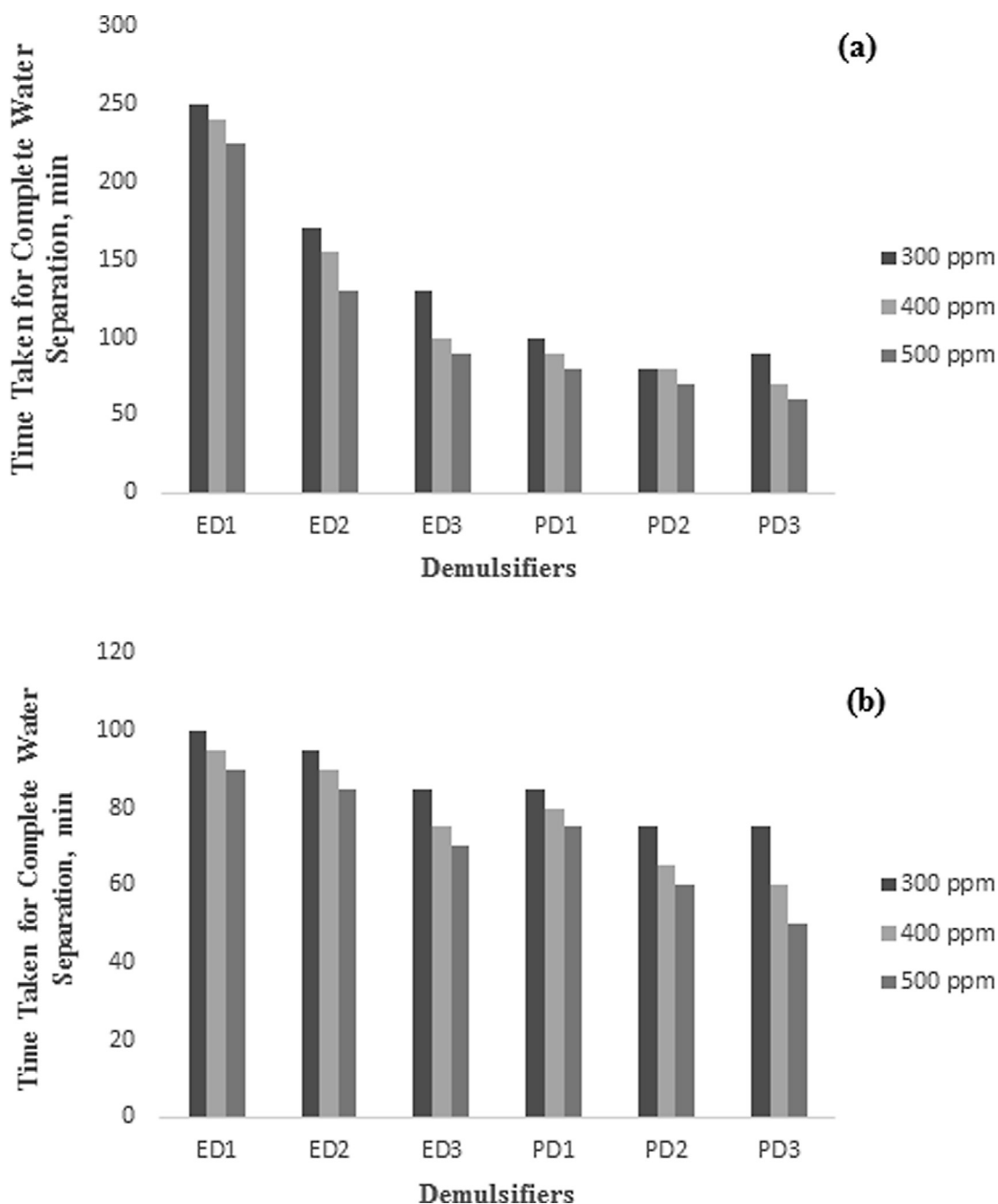
Figure 1 Demulsification efficiency for macropolymer of oleate methacrylate polyethylene oxide and polypropylene oxide with three different concentrations at different temperatures (a) 60 °C and (b) 85 °C.

accordance with the increase of HLB. This finding may be explained as the increase of HLB value increases the solubility of the surfactant in the aqueous phase (dispersed phase) [39]. When the demulsifier is initially introduced to the water-in-oil emulsion, it will be more thermodynamically stable at the interface of the water droplets. Accordingly, the concentration of the surfactants in the interface increases by increasing their HLB value. As the concentration of the surfactant increases at the interface, a continuous hydrophilic pathway is formed between the dispersed water droplets. This leads to rupture of the interfacial oil film surrounding the water droplets [39].

The formation of crude oil emulsion is mainly caused by high shear rates and zones of turbulence encountered at different points of production facilities, especially at the wellhead in the choke valve. These emulsions can be very stable due to the presence of polar compounds such as asphaltenes and resins that play an important role in the stability of W/O emulsion as the results of their mechanical stability on the interface. This work is tackling of the main problem that arose in EPRI-PVT Center during carrying out the study of reservoir phase behavior. The crude oil sample is delivered from the petroleum fields in the pressurized sample vessels under reservoir conditions on

Table 4 Rheological parameters for the freshly prepared crude oil emulsions treated with 500 ppm of ED_3 and PD_3 individually at 40 °C, 60 °C and 85 °C.

Water/oil emulsion	Temperature (°C)	Plastic viscosity, η (mPa s)	Yield stress, τ (Pa s)	IFT (mN m^{-1})
Crude oil emulsion	40	9.12	2.1	–
	60	6.39	2.0	
	85	3.95	1.5	
Dry crude oil	40	7.73	1.71	2.0
	60	5.47	1.51	
	85	4.14	0.62	
Treated emulsion with ED_3	40	7.74	0.06	5×10^{-2}
	60	5.15	0.65	
	85	3.87	1.23	
Treated emulsion with PD_3	40	5.65	1.46	7×10^{-3}
	60	5.06	0.65	
	85	3.9	0.77	

Table 5 The maximum demulsification efficiency (100%) at different concentrations for the mixture of demulsifiers at 60 °C.

Demulsifier mixture	HLB	Effective conc.			Alkylene oxide ratio	
		200 ppm (min)	300 ppm (min)	400 ppm (min)	e.o. %	p.o. %
$ED_1 + PD_1$	1.19	120	117	90	70	30
(M1)	0.52	105	75	60	50	50
	0.86	90	70	55	30	70
$ED_2 + PD_2$	1.27	90	75	70	70	30
(M2)	0.91	85	60	45	50	50
	0.55	60	45	30	30	70
$ED_3 + PD_3$	1.34	60	45	30	70	30
(M3)	0.96	45	30	20	50	50
	0.58	30	25	15	30	70
$ED_1 + PD_2$	1.19	110	80	75	70	30
(M4)	0.52	90	65	55	50	50
	0.86	80	50	45	30	70
$ED_1 + PD_3$	1.19	90	85	60	70	30
(M5)	0.52	75	60	45	50	50
	0.86	65	45	40	30	70
$ED_2 + PD_1$	1.26	115	110	85	70	30
(M6)	0.91	100	75	60	50	50
	0.55	85	65	50	30	70
$ED_2 + PD_3$	1.26	75	60	45	70	30
(M7)	0.91	60	45	30	50	50
	0.55	45	30	25	30	70
$ED_3 + PD_1$	1.34	110	100	80	70	30
(M8)	0.96	95	75	60	50	50
	0.58	80	60	50	30	70
$ED_3 + PD_2$	1.34	100	75	70	70	30
(M9)	0.96	85	60	50	50	50
	0.58	75	55	40	30	70

phase of W/O emulsion. So that, before starting studies of phase behavior, water must be resolved to liberate oil. All Egyptian petroleum companies demulsifiers failed to resolve this problem. The undertaken demulsifiers are a trial to solve this problem Fig. 3 shows the application of pressure, psi; atm; 1000, 2000, 3000, 4000 and 5000 psi on the PD_3 and ED_3. In the blank sample only 2% water was separated. But

the demulsifier PD_3 exhibited 93.1% water separation, while ED_3 recorded 91.5% at 1000 psi, meanwhile at pressure 5000 psi they gave separation efficiency of 76% and 70%, respectively.

This means that, increase of pressure on the blank sample increases the mechanical stability of asphaltene and resins on the interface to cause the water droplets coalescence to make
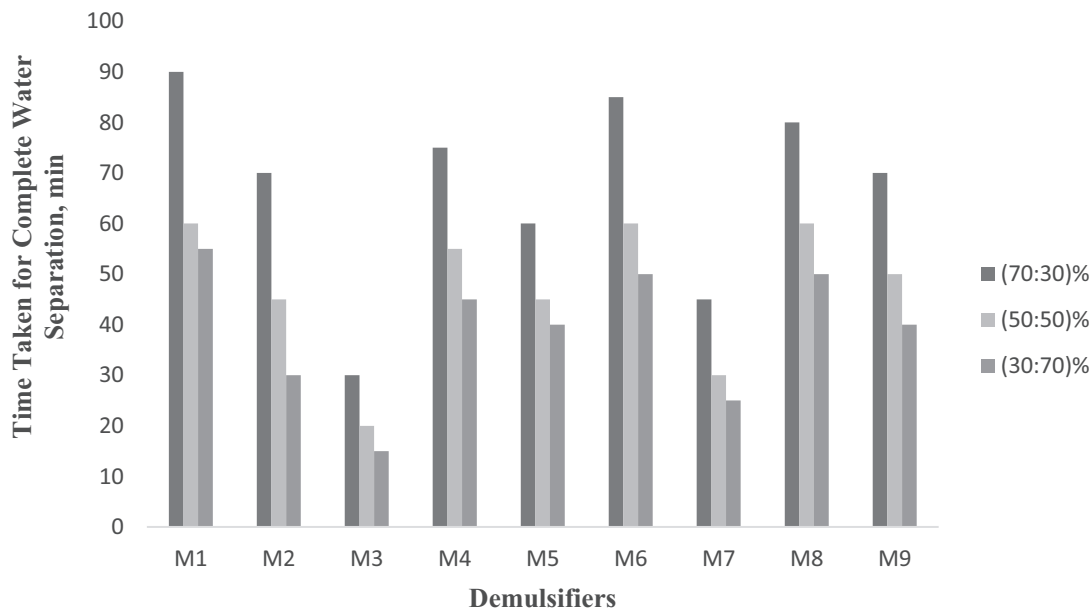
Figure 2 Demulsification efficiency for demulsifiers blends with three different concentrations at 60 °C.
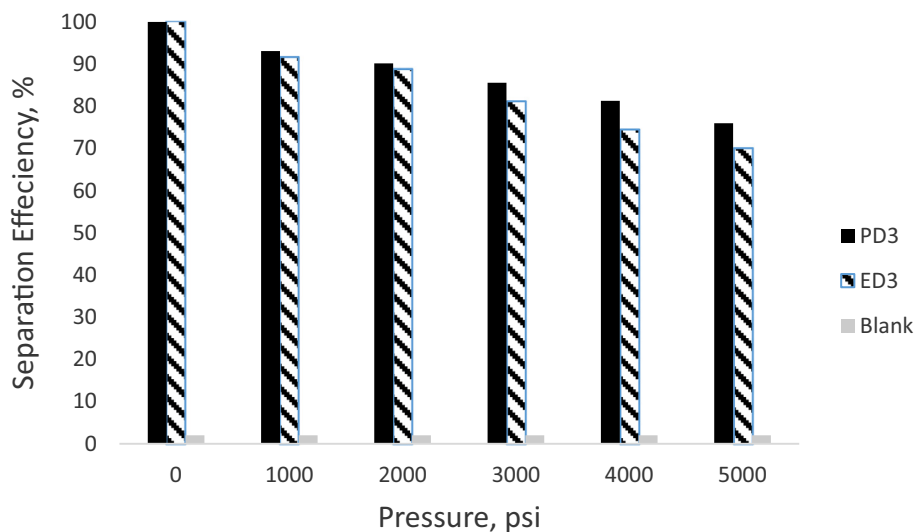

Figure 3 It shows the relation between the pressure in psi and the separation efficiency percent for demulsifiers PD$_3$ and ED$_3$ at 85 °C.

separation. In the case of the applied pressure in the presence of demulsifiers an increase in the demulsifiers' molecules mobilization in the oil phase is seen to get the interface and make adsorption to decrease the mechanical stability of asphaltene and resin on the water in oil interface, further the drainage starts, the coalescence and the separation followed to make demulsification reach up to 76.1% at 5000 psi.

3.2. Rheological behavior of the studied emulsions

The relationship between the elastic deformation and the applied stress is linear. However, above the yield stress point, the applied stress leads to unlimited deformation which causes the sample to start with flow (26). From Figs. 4–6, it can be observed that the yield stress which is required to start the flow decreases with decreasing water content in the emulsion. The yield stress of the dry crude oil was 1.71, 1.51, 0.62 Pa s at 40, 60, 85 °C and the yield value for the water- in- oil emulsion (BS&W = 12%) was 2.1, 2.0 and 1.5 Pa s at the same temperature. The increase of (τ) means that the internal pressure of water droplets in the oil phase during applying the shear rate as yield stress expressed as Bingham yield value (τ_B). On the other hand, the crude oil emulsion (12% BS&W) was treated by the demulsifier PD$_3$ which exhibited the maximum demulsification efficiency at 60 °C and 600 ppm (Table 3).

The first observation was decrease of the (τ_B) at 40, 60, 85 °C up to 0.06, 0.65, 1.23 and 1.46, 0.65, 0.77 Pa s. The second observation was increase of temperature, decreases (τ_B) the demulsifier PD$_3$, exhibited good results to decrease the rheological viscosity of crude oil emulsion (from 9.12 to 5.65);
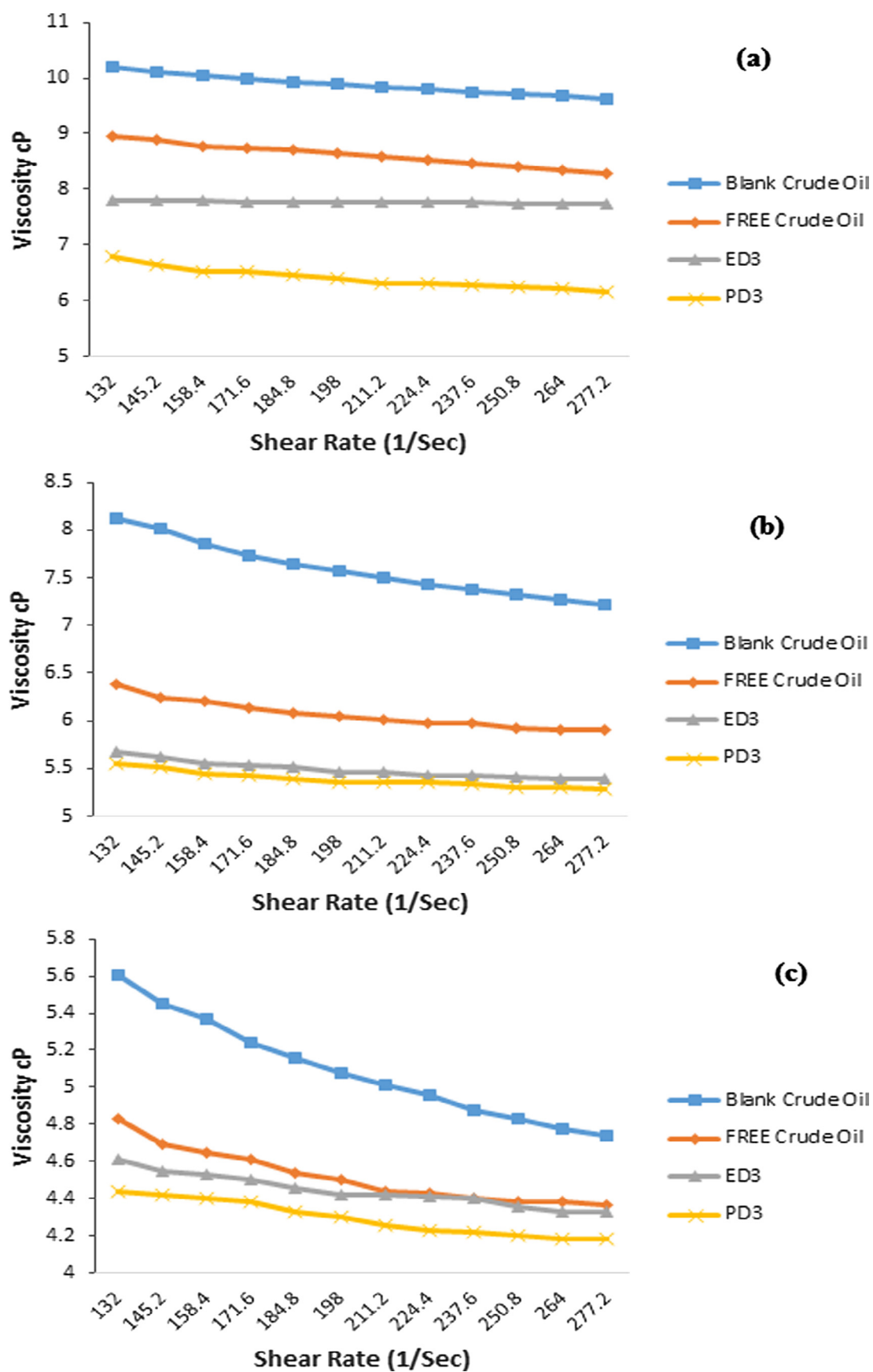
Figure 4 Relation between shear rate and viscosity for the untreated crude oil emulsions, free crude oil, treated crude oil with 500 ppm of ED_3 and treated crude oil with 500 ppm of PD_3 individually at different temperatures (a. 40 °C, b. 60 °C and c. 85 °C).
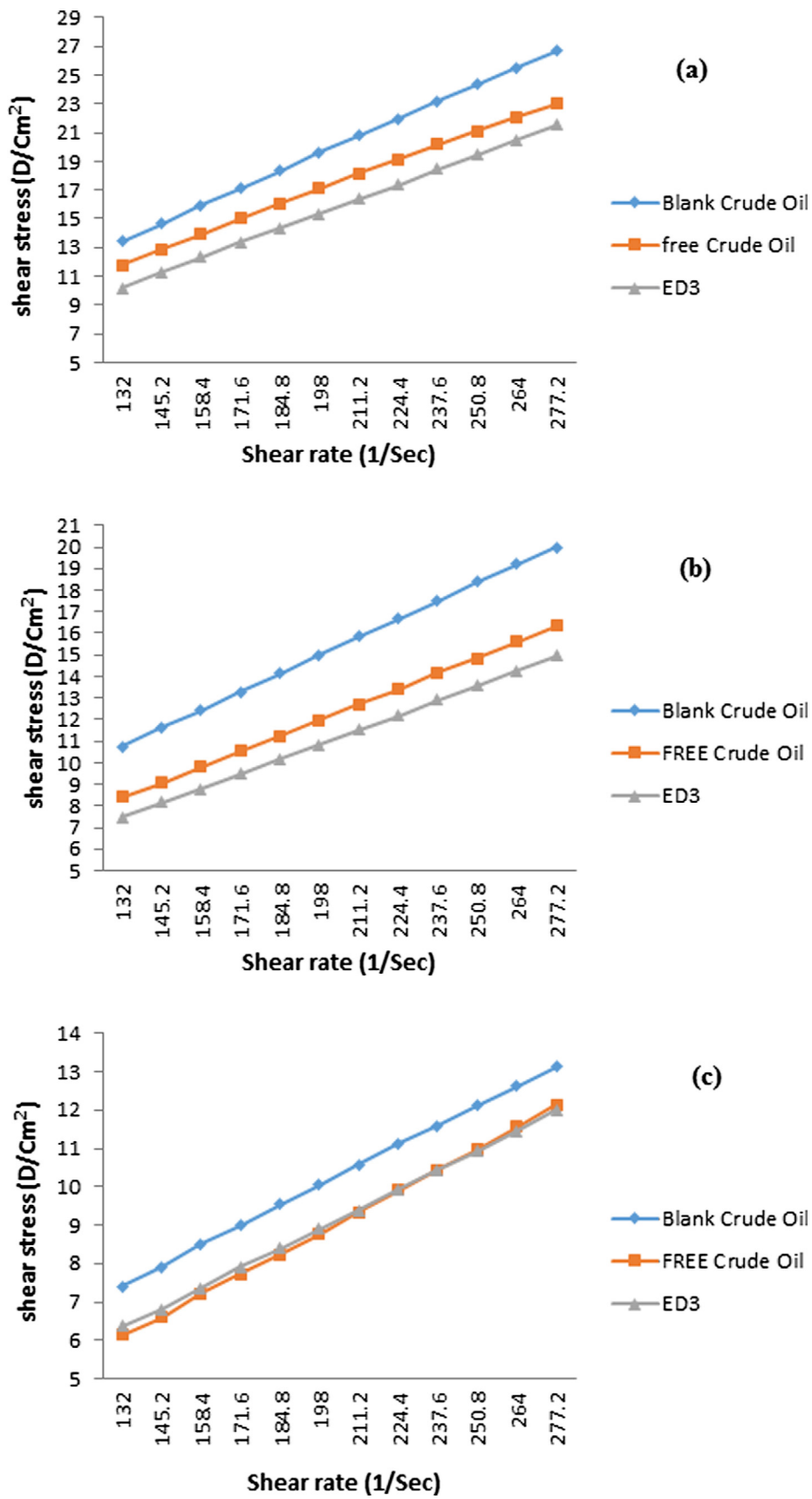
Figure 5 Relation between shear rate and shear stress for the untreated crude oil emulsions, free crude oil and treated crude oil with 500 ppm of ED$_3$ at different temperatures (a. 40 °C, b. 60 °C and C. 85 °C).
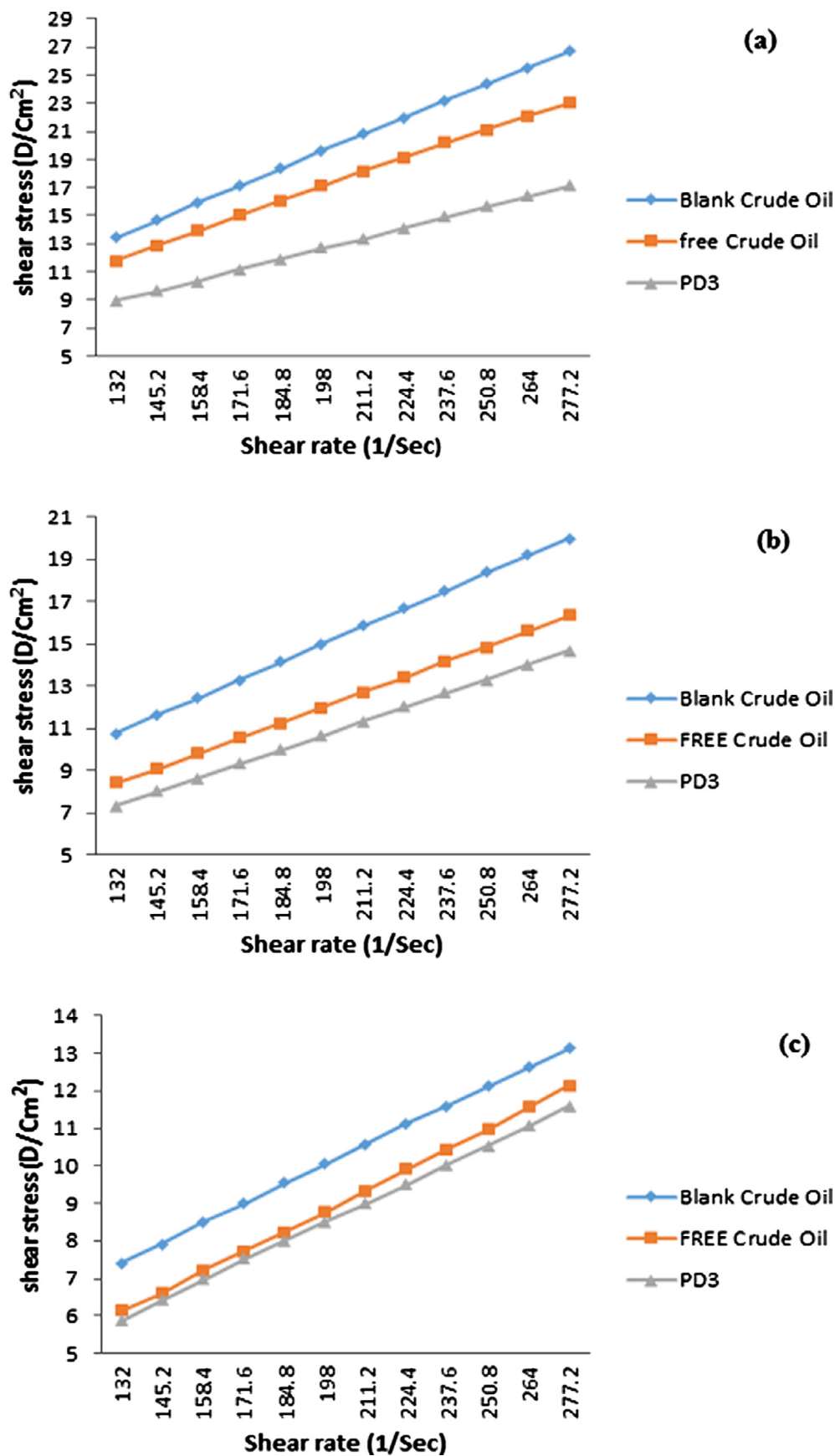
Figure 6 Relation between shear rate and shear stress for the untreated crude oil emulsions, free crude oil and treated crude oil with 500 ppm of PD$_3$ individually at different temperatures (a. 40 °C, b. 60 °C and c. 85 °C).

further decrease for (τ_B) (from 2.0 to 7×10^{-3}). This modification in the rheology of emulsion was better than the rheology of the dry crude oil or the emulsion without treatment.

This finding means that the demulsifier molecules play a central role to decrease the viscosity of emulsion during its mobilization in the phase and the decrease of interfacial tension leads to enhance the water separation.

Table 4 shows that, the plastic viscosities (η) were 9.12, 6.39, 3.95 mPa s for untreated 12% W/O emulsion at 40, 60, 85 °C, while it was 7.73, 5.47 and 4.14 mPa s for the dry crude oil. At the same time the dynamic viscosity of the treated emulsion was 5.65, 5.06, 3.9 mPa s at 40, 60, 85 °C. The viscosity obtained reflected directly on the (τ) yield values which obtained for the three; dry crude oil, crude oil emulsion and the treated emulsion as discussed above.

4. Conclusion

The conclusion can be summarised in the following points:

- The chemical structure of the demulsifiers which contain propylene oxide, may be play a central role to facilitate and maximize the demulsification efficiency.
- The increasing of the demulsifier concentrations (100–600 ppm) decrease the time taken to complete water separation.
- Increasing the temperature from 40 to 85 °C results in decreasing of the viscosity which leads to ease the adsorption of the demulsifier molecules on the interface, then the rate of water separation increases.
- There is a direct relation between the HLB values and the demulsification efficiency.
- The investigated demulsifiers can be used in reservoir conditions (85 °C and 5000 psi).
- The IFT decreases by increasing the concentration of surfactants.

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