Some surfactants based on the vegetable oils as CO₂ corrosion inhibitors for mild steel in oilfield formation water

V. M. Abbasov,¹ L. I. Aliyeva,¹* Hany M. Abd El-Lateef² and I. T. Ismayilov¹

¹Mamedaliev Institute of Petrochemical Processes, National Academy of Sciences of Azerbaijan, AZ1025 Baku, Azerbaijan ²Chemistry Department, Faculty of Science, Sohag University, Sohag, Egypt *E-mail: <u>leylufer-ipcp@rambler.ru</u>

Abstract

The new complex surfactants were synthesized based on sunflower oil and its inhibiting action on the corrosion of carbon steel in CO_2 -saturated oilfield formation water was investigated by means of weight loss and linear polarization resistance corrosion rate. The results revealed that the studied surfactants were excellent inhibitors. The percentage inhibition efficiency (*IE* %) increases by increasing the inhibitor concentration until the critical micelle concentration (CMC) is reached. The strong adsorption ability of the surfactant molecules leads to formation of a mono-layer, which isolates the surface from the environment and thereby reduces the corrosion attack on the surface. The adsorption process was found to obey the Langmuir adsorption isotherm.

Keywords: carbon steel pipelines, corrosion, complex surfactants, adsorption isotherm, weight loss measurements, oilfield formation water.

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1. Introduction

Corrosion in the oil industry represents one of the major problems. Carbon steels are extensively used in oil industries, *i.e.*, C1018 carbon steel is one of the most common material used as tubing in oil well. However, carbon steels are susceptible to corrosion in environment containing oil production. It has been acknowledged that CO_2 corrosion is one of major corrosion types resulting in oil well failure [1]. Due to the complexity of environment in oil well, CO_2 corrosion is dependent on various environmental conditions like temperature, CO_2 partial pressure, flow conditions and protective corrosion scales [2–7]. Corrosion inhibitors are the most effective and flexible mean of corrosion control in oil and gas production systems, but the selection and application of inhibitors are actually complicated because of the variable corrosive environments in these systems [8].

Many organic compounds containing oxygen, nitrogen and sulphur atoms have been used as corrosion inhibitors for carbon steel in various aggressive environments [9-14]. The addition of high molecular weight organic compounds such as surfactants to combat

corrosion of carbon steel has found wide application in many fields. It has been reported that these compounds possess high inhibition efficiencies for steel corrosion [15-17].

Surfactant inhibitors have many advantages, for example, high inhibition efficiency, low price, low toxicity, and easy production [18, 19]. Nowadays, surfactants are widely used and find a very large number of applications in the petroleum industry to protect iron and steel equipment used in drilling, production, transport, and refining of hydrocarbons [20]. This is attributed to their significant capability to influence the properties of surfaces and interfaces. The efficiency of the inhibition film depends on the inhibitor concentration and contact time with the metal surface. It was reported that the inhibition process by surfactants is attributed primarily to the adsorption of the surfactant molecules, via their functional group, onto the metal surface [21].

In the present work, we synthesized some environmentally complex surfactants based on sunflower oil and studied their inhibitive effect as corrosion inhibitors for carbon steel pipelines C1018 in CO₂-saturated oilfield formation water, and adsorption isotherm of the complex surfactants on the metal surface in the corrosive media is also described. The surface activity of the synthesized complex surfactants solutions was determined using surface and interfacial tension measurements at 25°C.

2. Material and methods

2.1. Chemical composition of carbon steel alloy

The rotating disk working electrodes for tests were made of carbon steel grade 080A15 and have an area of 4.55 cm² with a chemical composition (wt%) C 0.18%, Si 0.17%, Mn 0.70%, P 0.011%, S 0.03%, Ni 0.0%, Cr 0.01% and Fe balance. The data was provided by European Corrosion Supplies Ltd.

2.2. Synthesis of Complex Surfactants

The complex surfactants used as inhibitors are: sulfated fatty acid monoethanolamine complex (SFAMC) and sulfated fatty acid diethanolamine complex (SFADC), their molecular formulas are listed in Table 1. They were synthesized in our laboratory based on sunflower oil. The sunflower oil is a mixture of different fatty acids as shown in Table 2. Sunflower oil was hydrolyzed with solution of 25% NaOH for 7 hours at 90°C. This process yield in fatty acid sodium salt which is reacted with 37% HCl acid solution for extracting the fatty acids. Based on prepared fatty acid the sulfating syntheses were performed. The product is Sulfated fatty acid. The product was characterized by FT-IR spectroscopy (using a model FT-IR, Spectrum BX spectrometer using KBr disks). Sulfated fatty acid was taken at molar ratio 1:2 with Monoethanolamine and diethanolamine. The components of reactions were mixed well for a period 30 min at 25°C. The obtained surfactants are generally viscous liquids. The chemical structure of the synthesized complex surfactants was characterized by using FT-IR, Spectrum BX spectrometer using KBr disks.

Code number of the inhibitor	Name and abbreviation	Structure	Molecular weight (g /mol)
I	Sulfated fatty acids, Monoethanolamine complex (SFAMC)	$\begin{array}{c} \text{R-(CH}_2)_8\text{-}\text{CH-(CH}_2)_7\text{-}\text{COONH}_3\text{-}\text{CH}_2\text{-}\text{CH}_2\text{-}\text{OH}\\ \text{O} & \text{SO}_3^{+}\text{NH}_3\text{-}\text{CH}_2\text{-}\text{CH}_2\text{-}\text{OH} \end{array}$	462
П	Sulfated fatty acids, Diethanolamine complex (SFADC)	$\begin{array}{c} \text{R-(CH}_{2})_{8}\text{-} \text{CH-(CH}_{2})_{7}\text{-}\text{COONH}_{2}\text{-}(\text{CH}_{2}\text{-}\text{CH}_{2}\text{-}\text{OH})_{2} \\ \text{I} \\ \text{O} \\ \text{O} \\ \text{SO}_{3} \\ \text{NH}_{2}\text{-}(\text{CH}_{2}\text{-}\text{CH}_{2}\text{-}\text{OH})_{2} \end{array}$	551

Table 1. List of the synthesized surfactants including code number, name and structure.

Table 2: The	composition	of sunflower	oil (Percer	nt by weight o	f total fatty acids).
	1			2 0	2 /

	Composition, %	
Caturated	Palmitic Acid	7
Saturated	Stearic Acid	5
Mono unsaturated	Oleic Acid	19
Dolyungaturatad	Linoleic Acid	68
rory unsaturated	Alpha Linolenic Acid	1

2.3. Corrosion inhibition test

The oilfield formation water used in this investigation was obtained from Ramany oilfield in the Absheron peninsula (Azerbaijan). The most important characteristics of this water are listed in Table 3. The concentration range of the prepared surfactants was from 100 to 600 ppm used for corrosion measurements. All solutions were made from analytical grade reagents and deionized water.

Ions	Cations			Anions			Total ion	
	Na ⁺	\mathbf{K}^{+}	Mg ²⁺	Ca ²⁺	Cl⁻	\mathbf{SO}_4^{2-}	HCO ₃	concentration,
Concentration, ppm	40.599	5.632	987	8.134	65.156	198	360	121.066

Table 3: Chemical composition of oilfield formation water.

2.4. Corrosion measurements

2.4.1. Weight loss measurements

Weight loss experiments were done according to the method described previously [22]. The solution volume was 100 ml. The steel specimens used have a rectangular form (length = 2 cm, width = 1 cm, thickness = 0.07 cm). The immersion tests were carried out

at 50°C in CO₂-saturated oilfield formation water with different concentrations of inhibitors I and II for 24 h. Prior to tests, the solution was deoxygenated by purging CO₂ (99.81%) for 5 h. At the end of the tests, the specimens were taken out, washed carefully in ethanol under ultrasound until the corrosion products on the surface of carbon steel specimens were removed thoroughly, and then dried, weighed accurately. Duplicate experiments were performed in each case and the mean value of the weight loss is reported. Weight loss allowed calculation of the mean corrosion rate in mg cm⁻² h⁻¹.

2.4.2. Linear polarization resistance corrosion rate (LPR corrosion rate)

The linear polarization resistance (LPR) corrosion rate bubble-test method involves evaluating the corrosion of steel in simulated brine saturated with CO_2 at a temperature equivalent to that in the field. The LPR method is ideal for plant monitoring offering an almost instantaneous indication of corrosion rate, allowing for quick evaluation of remedial action and minimizing unscheduled downtime. The potential of the working electrode was varied by a CoreRunning programme (Version 5.1.3.) through an ACM instrument Gill AC. The apparatus consists of a personal computer HP Pavilion (monitor, CPU) and a Potentiostat/Galvanostat model ACM GILL AC (UK). The CoreRunning programme converts a corrosion current in mA/cm² to a corrosion rate in mm/year. A cylindrical carbon steel rod of the composition 080A15 GRADE STEEL was used as a working electrode. Gill AC technology allows measure DC and AC signals using standard Sequencer software. A small sweep from typically –10 mV to +10 mV at 10 mV/min around the rest potential is performed.

2.5. Surface tension measurements

The surface tensions were determined by DuNouy Tensiometer, Kruss Type 8451 and the temperature was maintained precisely at 25°C. Critical micelle concentration (CMC) values of surfactants were determined, according to the break points in plots of the surface tension versus log molar concentration of investigated surfactants.

3. Experimental results and discussion

3.1. Chemical structure of the synthesized surfactants

The structural characteristics of purified products of synthesized acids based on cottonseed oil (fatty acid and sulfated fatty acid) were confirmed by FT-IR spectroscopy in the range of 4000–500 cm⁻¹. The peaks of fatty acid are in accordance with the characteristic peaks of fatty acid reported in the literature [23]. The peak at about 1709 cm⁻¹ is due to the C=O carbonyl group of the carboxylic group, whereas the peak at 1550 cm⁻¹ arises due to C=C bond. This bond was broken after sulfating process. The peak at 1377 cm⁻¹ is due to S–O stretching absorption bands. It indicates the almost complete removal of C=C bond by sulfating process.

The chemical structure for surfactant complex was recorded in FT-IR. The FT-IR absorption spectra showed an absorption band in the 2341 cm^{-1} region, indicating that the

amine band was disappearing by emerging ammonium ion (RN^+H_3). In addition, there was a strong band at 871 cm⁻¹, indicating the presence of multiple (CH₂) groups. The very strong bands at the 2857 cm⁻¹ region was due mainly to the methyl asymmetric stretching vibration. The sharp band at 2926 cm⁻¹ due to the stretching vibration of the symmetric methylene group. The strong band at 1733 cm⁻¹ indicating to the increasing CO group frequency due to occurring a link between the acid proton with the amino nitrogen that leads to increase the attraction of electrons from carbonyl group. The FT-IR absorption spectra confirmed that the disappearance of OH band of acid (broad band), this confirmed the transfer of proton of acid to nitrogen atom of amine to form a $-N^+H_3$ group. The results are generally in agreement with the expected correlations.

3.2. LPR corrosion rate and surface tension measurements

The effects of the addition of various concentrations of SFAMC and SFADC surfactants on the corrosion of carbon steel C1018 were examined in CO₂-saturated oilfield formation water. Addition of either of these surfactants does not change significantly the pH of the formation water (pH 6.55). Figures 1 and 2 show the change in corrosion rate (CR) with time for carbon steel in CO₂-saturated oilfield formation water containing different concentrations of inhibitors SFAMC and SFADC at 50°C. The inhibitors were added after 1 hour of exposure because at this time the corrosion potential got stable, allowing the measurement of the corrosion rate prior to the injection of the inhibitor. The initial corrosion rate, without inhibitor, was measured to be between 1.7628 and 3.1754 mm y⁻¹. It can be observed from Figures 1 and 2 that the corrosion rate, in the absence of inhibitor, tends to increase with time. The increase in CR has been attributed to the galvanic effect between the ferrite phase and cementite (Fe₃C) which is part of the carbon steel [24].

The corrosion parameters were calculated on the basis of LPR corrosion rate test. The inhibition efficiency (*IE*, %) and surface coverage (θ) were calculated according to the following equations:

$$IE,\% = \frac{CR_0 - CR_i}{CR_0} \times 100$$
 (1)

Surface coverage (
$$\theta$$
): $\theta = 1 - \frac{CR_i}{CR_0}$ (2)

where CR_0 is the corrosion rate without inhibitor and CR_i the corrosion rate when inhibitor is present. It can be seen that the presence of inhibitors results in a high decrease in the corrosion rate. In the case of these inhibitors, the inhibition efficiency increases with increasing surfactant concentration reaching a maximum value around its critical concentration. The critical micelle concentration of the SFAMC and SFADC surfactants were determined at 25°C giving values of 468 and 457 ppm for SFAMC and SFADC, respectively (Figure 3).



Figure 1. Variation of the corrosion rate with time for carbon steel in CO₂-saturated oilfield formation water containing different concentrations of SFAMC at 50°C.



Figure 2. Variation of the corrosion rate with time for carbon steel in CO₂-saturated oilfield formation water containing different concentrations of SFADC at 50°C.



Figure 3. Plot of surface tension (γ) versus Ln concentration of complex surfactants SFAMC and SFADC at 25±0.1°C.

A general trend is observed in presence of the studied inhibitors; the corrosion rate of carbon steel in presence of these surfactants was decreased compared to the blank (CO₂-saturated formation water without inhibitor). By increasing the concentration of the surfactants, a further decrease in corrosion rate of carbon steel was observed. The maximum inhibition efficiency (*IE* %) was obtained at 600 ppm of inhibitors. This trend may result from the fact that adsorption of these complex surfactants forms thin inhibitor films on the metal surface which in order relatively isolate the metal surface from the corrosive environment causing much reduced corrosion rates. Inhibition efficiency of these films depends on various factors including but not limited to corrosivity of the environment, concentration of the surfactant, any synergetic effects with other molecules present in the environment and/or flow/shear effects [25].

The data in Table 4 shows that, the inhibition efficiency in the case of SFAMC and SFADC increases with increasing concentration of the surfactant reaching maximum values of 93.82 and 96.22%, respectively, around their critical micelle concentrations (CMC) (468 and 457 ppm for SFAMC and SFADC, respectively). The inhibition efficiency of metal slightly changes when the surfactant concentration exceeds the critical

micelle concentration. This behavior may be due to saturation of the surfactant adsorbed layer at the critical micelle concentration [26].

Inhibitors	Concentration, ppm	Corrosion rate (mm/year)	Surface coverage, θ	Inhibition efficiency, <i>IE</i> %
CO ₂ -saturated oilfield formation water without inhibitor	0.0	3.1754	_	_
	100	1.059	0.6664	66.64
	200	0.643	0.7975	79.75
SEAMC	300	0.429	0.8648	86.48
SFAMC	400	0.314	0.9011	90.11
	500	0.196	0.9382	93.82
	600	0.180	0.9433	94.33
	100	1.001	0.6847	68.47
	200	0.563	0.8226	82.26
SEA DO	300	0.373	0.8825	88.25
SFADC	400	0.221	0.9304	93.04
	500	0.120	0.9622	96.22
	600	0.076	0.9760	97.60

Table 4. The corrosion parameters for carbon steel electrode in CO_2 -saturated oilfield formation water in the absence and presence of various concentrations of complex surfactants at 50°C.

The effectiveness of studied complex surfactants as corrosion inhibitors depends on their structures. Corrosion inhibition of carbon steel in CO₂-saturated oilfield formation water by SFAMC and SFADC can be explained on the basis of molecular adsorption. It is apparent from the molecular structure that the surfactants are able to adsorb on the metal surface through the lone pair of electron of -N and -O atoms, besides the π -electron of C=O group [26].

It has to be noted that for a surfactant to be an excellent corrosion inhibitor it should exhibit a low CMC value, the inhibition effectiveness decreases as the CMC value increases [27]. On the basis of this view, surfactant **II** (SFADC) shows the lowest CMC value and hence it considers the most effective corrosion inhibitor for carbon steel in CO_2 saturated oilfield formation water. This, in fact, agrees LPR corrosion rate results. Surfactant **II** appears to create a good hydrophobic physical barrier to the aggressive ions and these accounts for its high inhibition efficiency. The data indicate that the inhibition efficiency of carbon steel in CO_2 -saturated oilfield formation in the presence of SFADC more than that obtained of SFAMC. This due to the fact SFADC contains the greatest number of carbon atoms, exhibits the best inhibition for the carbon steel corrosion in oilfield formation water saturated with CO_2 . This could be attributed to the presence of a long hydrocarbon chain that provides large surface coverage with the strongly adsorbed surfactant molecules forming a good barrier between the metal surface and the corrosive medium.

3.3. Weight loss measurements and adsorption isotherm

The previous section presented corrosion inhibition results obtained by LPR corrosion rate. It would be interesting to compare this LPR corrosion rate obtained value to corrosion rate values measured by simple weight-loss measurements. The inhibition efficiency obtained from weight loss measurements for different concentrations of inhibitors I and II in CO₂-saturated oilfield formation water were given in Figure 4 and Table 5. It could be seen that, the complex surfactants are a very good inhibitors against steel corrosion in the investigated media. And, *IE* (%) increased with inhibitors concentrations, which suggested that the corrosion inhibition of SFAMC and SFADC for carbon steel might be caused by the adsorption of surfactant onto the metal surface from aggressive media.



Figure 4. Variation of the corrosion rate and inhibition efficiency with the concentration of inhibitors **I** and **II** in ppm for carbon steel in CO₂-saturated oilfield formation water at 50°C.

Inhibitors	Concentration, ppm	Corrosion rate (mg cm ⁻² per hour)	Surface coverage (θ)	IE %
Blank	0	0.825	_	_
	100	0.288	0.6510	65.1
	200	0.181	0.7806	78.06
SEAMO	300	0.115	0.8600	86.00
SFAMC	400	0.078	0.9050	90.50
	500	0.054	0.9345	93.45
	600	0.045	0.9460	94.60
	100	0.274	0.6678	66.78
SFADC	200	0.167	0.7975	79.75
	300	0.101	0.8780	87.80
	400	0.064	0.9224	92.24
	500	0.04	0.9515	95.15
	600	0.031	0.9625	96.25

Table 5. Corrosion rate, inhibition efficiency and surface coverage (θ) data obtained from weight loss measurements for carbon steel in oilfield formation water in the absence and presence of various concentrations of SFAMC and SFADC at 50 °C

The degree of surface coverage (θ), the corrosion rate of carbon steel (k) and the percentage inhibition efficiency (*IE* %) were calculated from the following equation [28]:

$$\theta = \frac{W_0 - W}{W_0} \tag{3}$$

$$IE, \% = \left(\frac{W_0 - W}{W_0}\right) \times 100 \tag{4}$$

$$k = \frac{\text{weight loss (mg cm^{-2})}}{\text{time (h)}}$$
(5)

where W_0 and W are the values of weight losses in absence and presence of the inhibitor, respectively. All calculations concerning the adsorption characteristics of the inhibitor were made under the assumption that it has a purely blocking mechanism of action. The obtained data are summarized and listed in Table 5. These results showed that, by increasing the inhibitor concentration, both, surface coverage (θ) and (*IE* %) were increased while (k) was decreased. So, the dissolution of carbon steel in oilfield formation water in the presence of the investigated surfactants can be interpreted on the basis of interface inhibition mode, *i.e.* the inhibitor acts effectively at the metal solution interface

[29]. The results also indicate that, the percentage inhibition efficiency (*IE* %) of the inhibitor (**II**) is greater than that of the inhibitor (**I**); thereby, these results are in a good agreed with aforementioned results of LPR corrosion rate.

Basic information on the interaction between the inhibitor molecules and metal surfaces could be provided from the adsorption isotherms. The values of surface coverage (θ) which were defined as in Eq. (3) and obtained from weight loss measurements at 323 K.

A correlation between θ and inhibitor concentration in the corrosive medium can be represented by the Langmuir adsorption isotherm [30].

$$\frac{C_{\rm inh}}{\theta} = C_{\rm inh} + \frac{1}{K_{\rm ads}},\tag{6}$$

where K_{ads} is the equilibrium constant of the inhibitor adsorption process and C_{inh} is the inhibitor concentration.

Plots of C_{inh}/θ versus C_{inh} yielded a straight line as shown in Figure 5, which suggested that at 323 K the adsorption of investigated inhibitors on metal surface obeyed Langmuir adsorption isotherm model. This isotherm assumed that the adsorbed molecules occupied only one site and there was no interaction with other molecules adsorbed. The linear regression coefficients (r) and the slopes parameter were shown in Table 6. All correlation coefficients (r > 0.998) indicated that the inhibition of carbon steel by studied complex surfactants was attributed to the adsorption of inhibitors on the metal surface. However, the slopes of the C_{inh}/θ versus C_{inh} plots were close to 1 and showed a little deviation from unity which meant non-ideal simulating [31] and unexpected from Langmuir adsorption isotherm. They might be the results of the interactions between the adsorbed species on the metal surface [32, 33].

 K_{ads} values could be calculated from the intercepts of the straight lines on the C_{inh}/θ axis, the K_{ads} was related to the standard free energy of adsorption, ΔG_{ads}^0 , by the following equation [34]:

$$K_{ads} = \frac{1}{55.5} \exp\left(-\frac{\Delta G_{ads}^0}{RT}\right)$$
(7)

The value 55.5 in the above equation was the molar concentration of water in solution in mol/L [34]. The relatively high value of the adsorption equilibrium constant (K_{ads} ; Table 6) reflects the high adsorption ability of these complex surfactants on the metal surface [35]. It is also noted that, the high value of K_{ads} for inhibitor II indicate stronger adsorption on the carbon steel surface than the inhibitor I. Large values of K_{ads} imply more efficient adsorption hence better inhibition efficiency [36].



Figure 5. Langmuir plots for inhibitors **I** and **II** in CO₂-saturated oilfield formation water obtained from weight loss measurements at 50°C.

Table 6. Thermodynamic parameters for the adsorption of SFAMC and SFADC in CO₂-saturated oilfield formation water on the carbon steel surface at 323 K.

Inhibitors	Slope	Regression coefficients (r)	K _{ads} (M ⁻¹)	$\Delta G_{ m ads}^0$ (kJ mol ⁻¹)
SFAMC	1.03	0.998	7487.27	-39.36
SFADC	1.02	0.999	9345.79	-42.67

The high and negative values of free energy of adsorption (ΔG_{ads}^0) indicate spontaneous adsorption and strong interaction of the inhibitor molecule with the carbon steel surface [37]. Generally, values of ΔG_{ads}^0 up to -20 kJ mol⁻¹ are consistent with physisorption, while those around -40 kJ mol⁻¹ or higher are associated with chemisorption as a result of the sharing or transfer of electrons from organic molecules to the metal surface to form a coordinate bond [38]. In the present study, the ΔG_{ads}^0 values obtained for the SFAMC and SFADC surfactants on carbon steel in CO₂-saturated oilfield formation water are -39.36 and -42.67 kJ mol⁻¹, respectively. This indicates that the adsorption of studied inhibitors is typical chemisorption.

4. Conclusion

- 1. The synthesized complex surfactants act as a very good corrosion inhibitor for carbon steel in oilfield formation water.
- 2. The percentage inhibition efficiency (*IE* %) increases with increasing the inhibitor concentration and reaches a maximum value at near critical micelle concentration (CMC) and the inhibition efficiencies obtained from weight loss measurements and LPR corrosion rate were in good agreement.
- 3. The adsorption model obeyed Langmuir adsorption isotherm. The adsorption process was spontaneous. The relatively high value of K_{ads} reflects the high adsorption ability of the inhibitor molecules on carbon steel surface. The values of the standard free energies of adsorption indicated that investigated surfactants adsorbed on carbon steel in CO₂-saturated oilfield formation water were typical chemisorption mechanism.

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