

Evaluation of New Complex Surfactants Based on Vegetable Oils as Corrosion Inhibitors for Mild Steel in CO₂-Saturated 1.0% NaCl Solutions

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Abstract Novel complex surfactants were prepared based on some vegetable oils (Monoethanolamine sulfated fatty acid; SM). The chemical structures of the prepared compounds were confirmed by physical-chemical spectroscopic methods. The studied surfactants were evaluated as corrosion inhibitors for mild steel in CO₂-saturated 1.0 % NaCl solution. The data showed that, the corrosion rate decreased and inhibition increased with the inhibitor concentration from 10 to 100 ppm. The corrosion inhibition of mild steel in CO₂-saturated brine containing SM has been attributed to adsorption of inhibitors over the metal surface. It was found that the adsorption of SM inhibitor is in accordance with the Langmuir adsorption isotherm. Scanning Electron Microscopy (SEM) is used to examine the surface morphology of the mild steel samples both in absence and presence of investigated inhibitor at optimum conditions.

Keywords: corrosion inhibition, mild steel, complex surfactants, vegetable oils, SM, SEM

1. Introduction

Corrosion of steel by CO₂ has been one of the major problems in the oil industry since 1940. Recently, it has again come to the fore because of the technique of CO₂ injection for enhanced oil recovery and exploitation of deep natural gas reservoirs containing carbon dioxide [1]. The major concern with CO₂ corrosion in oil and gas industry is that CO₂ corrosion can cause failure on the equipment especially the main downhole tubing and transmission pipelines and thus can disrupt the oil/gas production.

The addition of corrosion inhibitors is a standard practice in oil and gas production systems to control the internal corrosion of mild steel structures [2,3]. The use of surfactants as inhibitors is one of the best-known methods of corrosion protection. Surfactants, when used as inhibitors, act through a process of surface adsorption. Adsorption of surfactant on solid surfaces can modify the surface charge, and also the hydrophobic and other key properties of the solid surface that influence interfacial processes [4]. The ability of a surfactant molecule to adsorb is generally related to its ability to aggregate to form micelles. Most acid inhibitors are organic compounds containing nitrogen, sulphur and/or oxygen atoms [5,6]. The concern of these materials arises from their significance in recent civilization. Inhibition of metal corrosion by organic compounds is a result of adsorption of organic molecules or ions at the metal surface forming a protective layer. This layer reduces or prevents corrosion of the metal. The degree of adsorption depends on the nature of the metal, the metal surface condition, the mode

of adsorption, the chemical structure of the inhibitor, and the type of corrosion media [7].

Many organic compounds were tested and industrially applied as corrosion inhibitors for steel in CO₂ environments. These compounds, are hydroxyethyl, aminoethyl, and amidoethyl imidazolines [8] and *N,N*-di(poly oxy ethylene) [9]. The data show that, the most organic inhibitors act by adsorption on the metal surface. This phenomenon is influenced by the nature and the surface charge of metal, by the type of aggressive electrolyte and by the chemical structure of inhibitors [10]. Monoethanolamine sulfated fatty acid complex (SM; Figure 1) are relatively cheap and easy to produce in high purity. In order to develop “green” or environmentally “friendly” inhibitors, the recent investigations [2,3] on nontoxic SM seem really worthwhile. Our present research was to prepare novel complex surfactants based on vegetable oils and study their inhibition effect against the corrosion of mild steel in CO₂ environments. The inhibition performance of the synthesized inhibitors was evaluated by LPR corrosion rate method complemented with some SEM observations. The mechanism of inhibition is discussed on the basis of an adsorption isotherm, as well as the calculated thermodynamic parameters.

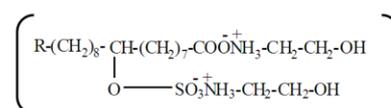


Figure 1. Molecular structure of Monoethanolamine sulfated fatty acid complex (SM).

2. Material and Methods

2.1. Chemical Composition of Mild Steel Alloy C1018

The rotating disk working electrodes for tests were made of mild steel grade 080A 15 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 and by Energy dispersive X-ray fluorescence (EDRF, Figure 2) (HORIBA XGT-7000).

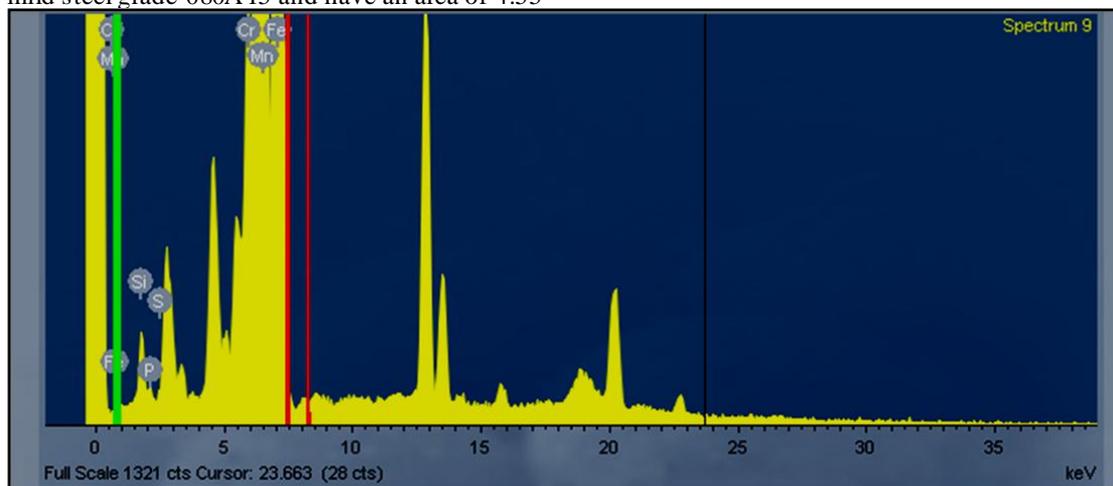


Figure 2. EDRF analysis of mild steel electrode surface

2.2. Synthesis of Complex Surfactant

The complex surfactant used as inhibitor is *Monoethanolamine* sulfated fatty acid complex (SM). The inhibitor was synthesized in our laboratory based on different four types of vegetable oils (SM_I based on sunflower oil, SM_{II} based on cottonseed oil, SM_{III} based on corn oil and SM_{IV} based on palm oil). The 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*. The components of reactions were mixed well for a period 30 min at 25 °C. The chemical structure of the synthesized complex surfactant was characterized by using FT-IR, Spectrum BX spectrometer using KBr disks.

2.3. Corrosion Inhibition Test

The aggressive solution, 1.0% NaCl, was prepared by dissolving of analytical grade NaCl in distilled water. The concentration range of the prepared inhibitors was from 10 to 100 ppm used for corrosion measurements.

2.4. Corrosion Measurements

To study the corrosion protection ability of the synthesized surfactants, one of the newest devices in recent years ACM, GILL AC was used. The apparatus consists of a personal computer HP Pavilion (monitor, CPU), a Potentiostat/Galvanostat model ACM GILL AC (UK), four pieces of glasses with a capacity of 4000ml, electrodes, CO₂ tank and installations-regulating the quantity fed CO₂. A magnetic stirrer stirred the prepared

1.0% of the sodium chloride solution for 30 minutes in 4L beaker. The prepared solution poured into the four glass beakers (1000ml for each one). These beakers were, then placed on a heater at 50 °C for 1 hour under a pressure of 0.9 bars carbon dioxide saturated the solution. The electrodes were immersed in the medium and are connected through a potentiometer ACM GILL AC. The surfaces of working electrode were cleaned by acetone before using. Electrodes are used for one time only. After 1 hour, except for one beaker, the remaining three were fed with the suitable amount of inhibitor and continued supply of CO₂ under 0.9 bar pressure till the end of the experiment.

A Core Running program (Version 5.1.3.) varied the potential of the working electrode through a potentiometer ACM instruments Gill AC. Gill AC technology allows measuring DC and AC signals using standard Sequencer software. A small sweep from typically, -10mV to +10 mV, at 10mV min⁻¹ around the rest potential is performed. The Core Running program converts a corrosion current in mA cm⁻² to different relationships (building on the corrosion rate per time (mm year⁻¹), the metal loss per time (mg h⁻¹).

Each experiment was performed with freshly prepared solution and clean set of electrodes. Measurements were conducted at temperature 50 °C for the investigated NaCl solution. For this purpose, magnetic stirrer with heater (115V, 50/60 Hz) was used.

2.5. Surface Characterization

In order to observe any changes in surface morphologies of the mild steel samples after testing, the specimens were first immersed in the aggressive media with and without an inhibitor SM_{III} for 5 days, then cleaned with bi-distilled water and acetone, and dried with cool air. Then the morphology of the tested sample was observed by using scanning electron microscope (JEOL, model 5300).

3. Results and Discussion

3.1. Chemical Structure of the Synthesized Surfactants

The structural characteristics of purified products of synthesized acids (fatty acid and sulfated fatty acid) were confirmed by FT-IR spectroscopy in the range 4000–500 cm^{-1} as shown in Figure 3. The spectrum showed that, peak at about 1709 cm^{-1} is due to the C=O carbonyl group of the carboxylic group, whereas the peak at 1490 cm^{-1} arises due to C=C bond. This bond was broken after sulfating process. The peak at 1366 cm^{-1} is due to S-O stretching absorption bands. It indicates the almost complete removal of C=C bond by addition process. The FT-IR absorption spectra for the synthesized complex surfactants showed, an absorption band at the 2330-2350

cm^{-1} region for all prepared complexes, indicating that the amine band was disappearing by emerging ammonium ion (RN^+H_3). In addition, there was a strong band at 855-908 cm^{-1} , indicating the presence of multiple (CH_2) groups. The very strong bands at the 2846-2855 cm^{-1} region for the prepared compound were due mainly to the methyl asymmetric stretching vibration. The sharp band at 2925-2966 cm^{-1} was observed for all prepared surfactants due to the stretching vibration of the symmetric methylene group. The strong band at 1725-1740 cm^{-1} for all compounds indicating to the increasing C=O group frequency due to occurring a link between the acid proton with the amino nitrogen that leads to increasing the attraction of electrons from carbonyl group. The peak at 1377 cm^{-1} is due to S-O stretching absorption bands. The FT-IR absorption spectra confirmed that the disappearance of -OH band of acid in sulfated fatty acid.

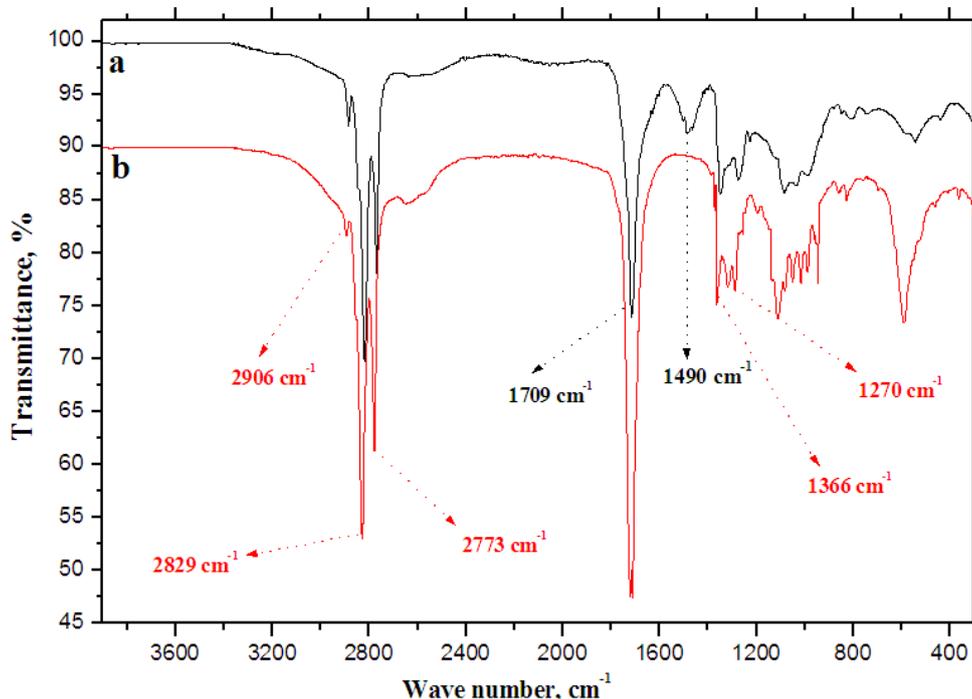


Figure 3. FT-IR spectrum of (a) Fatty acids derived from palm oil, (b) Fatty acids after sulfating process.

3.2. Inhibition of Mild Steel in CO_2 -Saturated Solution by Synthesized Surfactants

The synthesized inhibitors were tested with linear polarization- resistance corrosion rate (LPR) and rotating-cylinder-electrode (RCE) techniques. In all cases, tests were performed on mild steel under CO_2 -saturated 1.0% NaCl solution. The bubble-test method involves evaluating the corrosion of a given metal in simulated brine saturated with CO_2 at a temperature equivalent to that in the field. During the test, CO_2 gas is sparged continuously into the test solution. The rate of corrosion is determined instantaneously with the LPR technique,

Figure 4 shows that, the change in corrosion rate (CR) with time for mild steel in CO_2 -saturated 1.0% NaCl solution containing different concentrations form inhibitor SM_1 at 50 °C. The inhibitor was added after 1 hour of exposure because at this time the corrosion potential got stable, allowing the measurement of the CR prior the injection of the inhibitor. The initial corrosion rate, without inhibitor, was measured to be between 2.59 and

3.784 mm y^{-1} . It was found from Figure 4 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 (Fe_3C) which is a part of the original steel in the non-oxidized state and accumulates on the surface after the preferential dissolution of ferrite ($\alpha\text{-Fe}$) into Fe^{2+} [11]. Fe_3C is known to be less active than the ferrite phase. Therefore, there is a preferential dissolution of ferrite over cementite, working the former as the anode and latter as the cathode, favoring the hydrogen evolved reaction (HER) during the corrosion process [12,13].

The corrosion parameters were calculated on the basis of LPR corrosion rate test. The inhibition efficiency ($\eta\%$) was calculated according to the following equation:

$$\eta\% = \frac{CR_0 - CR_1}{CR_0} \times 100 \quad (1)$$

where CR_0 is the corrosion rate without inhibitor and CR_1 is the corrosion rate when inhibitor is present. The obtained experimental results show that, the addition of

the under testing surfactants greatly decreases the corrosion rate of mild steel in the given aggressive media, the corrosion rate decreases as the inhibitor concentration increases, getting maximum inhibition efficiency 99.92% at 100 ppm after 20 hour of exposure for SM_{III} (Table 1).

This trend may results from the fact that adsorption and surface coverage increase with the increase in concentration; thus the surface is efficiently separated from the medium [14].

Table 1. The corrosion parameters obtained from LPR corrosion rate measurements for mild steel electrode in CO₂-saturated 1.0% solution of NaCl in the absence and presence of various concentrations of SM obtained based on vegetable oils at 50 °C

Inhibitor	Concentration of inhibitors, ppm	Corrosion rate (mm/year)	Surface coverage θ	The inhibition efficiency, $\eta\%$
CO ₂ -saturated 1.0 % NaCl without inhibitor	0.0	3.784	----	----
SM _I	10	0.441	0.883	88.33
	25	0.279	0.926	92.60
	50	0.179	0.952	95.24
	75	0.068	0.981	98.18
	100	0.003	0.990	99.90
SM _{II}	10	0.397	0.894	89.49
	25	0.329	0.913	91.30
	50	0.147	0.961	96.11
	75	0.082	0.978	97.82
	100	0.010	0.997	99.70
SM _{III}	10	0.374	0.900	90.09
	25	0.109	0.971	97.10
	50	0.059	0.984	98.42
	75	0.037	0.990	99.00
	100	0.002	0.999	99.92
SM _{IV}	10	0.562	0.851	85.13
	25	0.446	0.881	88.19
	50	0.249	0.933	93.39
	75	0.134	0.964	96.46
	100	0.051	0.986	98.63

Table 1 shows the calculated values of corrosion rates, the inhibition efficiencies and the surface coverage in the absence and presence of different concentrations of different inhibitors at 50 °C. The data exhibited that, the corrosion rates, the inhibition efficiencies and the surface coverage are found to depend on the concentrations of the inhibitors. The corrosion rates (CR) decreased, and the inhibition efficiencies ($\eta\%$) and the surface coverage (θ) increased with the increase of the surfactant concentrations. This indicates that the inhibitory action of the inhibitors against mild steel corrosion can be attributed to the adsorption of these molecules on the metal surface, limits the dissolution of mild steel, and the adsorption amounts of surfactants on mild steel increased with the concentrations in the corrosive solutions. This ability of formation of protective surface layers increased with the increase of surfactant concentration.

The results also show that the inhibition efficiencies in the case of surfactant complex obtained on the basis of corn oil are higher compared with those of inhibitors obtained based on other oils at the same conditions (Figure 5), and the results showed the following trend at 100 ppm of different inhibitors:

$$SM_{III} > SM_I > SM_{II} > SM_{IV}$$

This behavior may be due to the difference in fatty acids compositions of the investigated oils. The fatty acid

composition of oils from vegetable sources varies depending on plant origin and sort [15]. The inhibition efficiency values of inhibitors depend on the molecular weight. The $\eta\%$ values of inhibitor SM_{III} are higher than those of other inhibitors, due to the larger molecular weight of the former compared with that of the latter. The molecular weight of investigated compounds increased in the following order:

$$SM_{III} (463) > SM_I (462) > SM_{II} (459) > SM_{IV} (453)$$

The high θ value (Table 1) near unity indicates almost a full coverage of the metal surface with adsorbed surfactant molecules. Conclusively, the surfactant inhibitor having θ near unity is considered as a good physical barrier shielding the corroding surface from corrosive medium and dumping the corrosion rate of mild steel significantly.

Figure 5 shows the variation of the corrosion rate with time for mild steel in CO₂-saturated 1.0% NaCl solution containing 100 ppm from SM based on all oils at 50 °C. This plot indicates that, the presence of different inhibitors decreases the rate of corrosion. However, the maximum decreased in corrosion rate was observed for the inhibitor SM_{III}. The highest inhibition efficiency of the investigated inhibitors may be attributed to the following reasons:

I. The higher electron density on the functional groups (N, O and S) leads to easier bond formation, greater adsorption, and consequently, higher inhibition [15].

II. The long length of the hydrocarbon chain in surfactant molecule means a more bulk molecule, which screen the surface from attack [16].

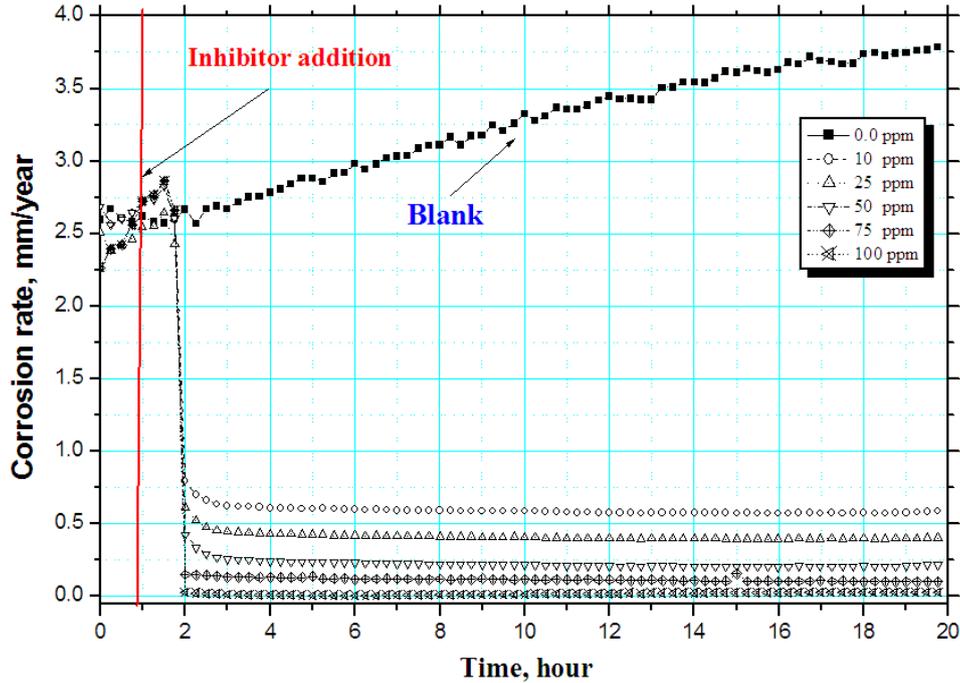


Figure 4. Variation of the Corrosion rate with time for mild steel in CO_2 -saturated 1.0% NaCl solution containing different concentrations of SM at 50°C

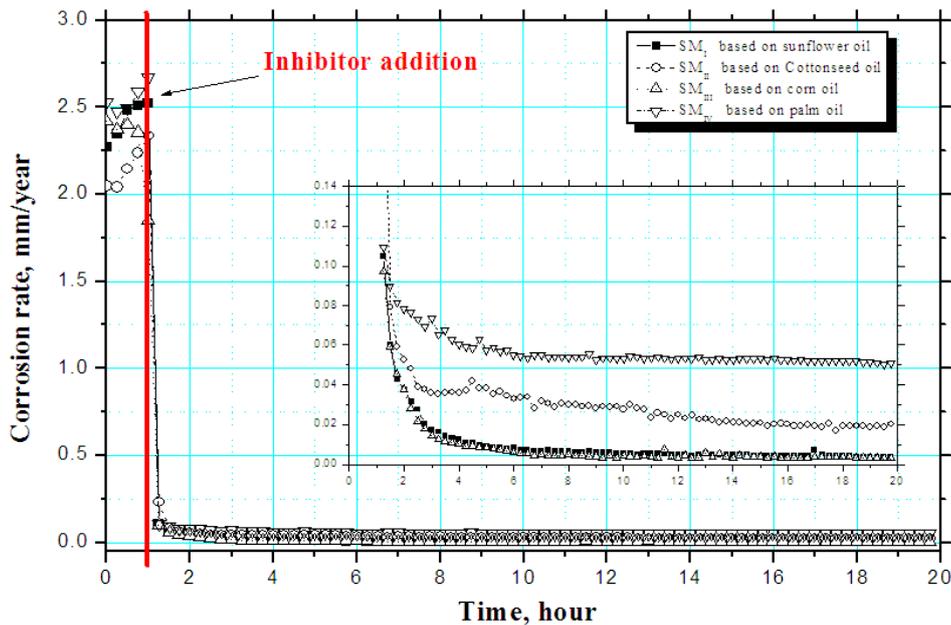


Figure 5. Variation of the Corrosion rate with time for mild steel in CO_2 -saturated 1.0% NaCl solution containing 100 ppm of SM at 50°C

3.3. Adsorption Isotherm and Thermodynamic Parameters for the Corrosion Process

The extent of corrosion inhibition depends on the surface conditions and mode of adsorption of inhibitors [14]. Assuming that the corrosion on the covered parts of the surface is equal to zero and that the corrosion takes place only on the uncovered parts of the surface, the degree of coverage (θ) was calculated from:

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

where CR_0 and CR_i are the uninhibited and inhibited corrosion rates. In order to get more information about the mode of adsorption of the complex surfactants on the surface of the investigated metal at 50°C , the data obtained from LPR corrosion rate have been tested with several adsorption isotherms. Langmuir adsorption isotherms [17] were found to fit well with the experimental data. The adsorption isotherm relationship of Langmuir is represented by the following equation:

$$\frac{C_{inh}}{\theta} = C_{inh} + \frac{1}{K_{ads}} \quad (3)$$

C_{inh} is the inhibitor concentration, θ is the surface covered and K_{ads} is the adsorption coefficient which is evaluated from the plots and is related to the standard free energy of adsorption, ΔG_{ads}^o by [18]:

$$K_{ads} = \frac{1}{55.5} \exp\left(-\frac{\Delta G_{ads}^o}{RT}\right) \quad (4)$$

The plot between C_{inh}/θ versus C_{inh} gave a straight line (Figure 6), which suggests that the adsorption of the inhibitor molecules on the metal surface obeyed the Langmuir adsorption model [19]. The values of the linear regression coefficient (R), slope and adsorption coefficients of the different SM inhibitors are listed in Table 2. The correlation coefficient values ($R > 0.996$) indicate that the inhibition of the mild steel by investigated surfactants was attributed to the adsorption of the inhibitor molecules on the metal surface. The calculated values of K_{ads} and ΔG_{ads}^o are given in Table 2.

The high values of K_{ads} for studied inhibitors more than 8.45×10^4 indicate strongest adsorption on the mild steel surface in CO_2 -saturated 1.0% NaCl solution. It is also noted that, the high values of K_{ads} for inhibitor SM_{III} indicate stronger adsorption on the mild steel surface than the other inhibitors. Large values of K_{ads} imply more efficient adsorption hence better inhibition efficiency [14]. The large value of K_{ads} obtained for the investigated

surfactants agree with the high inhibition efficiency obtained.

The value and sign of ΔG_{ads}^o provide information about the mechanism of the inhibitor molecules adsorption at the metal surface. The negative values of ΔG_{ads}^o indicate that the inhibitor molecules adsorbed spontaneously onto the metal surface [18]. On the other hand, the ΔG_{ads}^o values describe the type of inhibitor molecule adsorption on the metal surface. It is generally accepted that the values of ΔG_{ads}^o around 20 kJ mol^{-1} or less is assumed for electrostatic interactions exist between inhibitor and the charged metal surface (physisorption) [20]. Values of 40 kJ mol^{-1} or more indicate charge transferring from organic specie to the metal surface, to form a coordinate type of metal bond (Chemisorption) [18]. It is clear from Table 2 that, the values of ΔG_{ads}^o in our measurements are ranged between -40.98 and $-44.23 \text{ kJ mol}^{-1}$, which suggests the adsorption of inhibitor molecules onto the metal surface as chemisorption. Chemisorption of the surfactant molecules could occur due to the formation of links between the d orbital of iron atoms, involving the displacement of water molecules from the metal surface, and the lone sp^2 electron pairs present on the N, S and/or O atoms of the inhibitor.

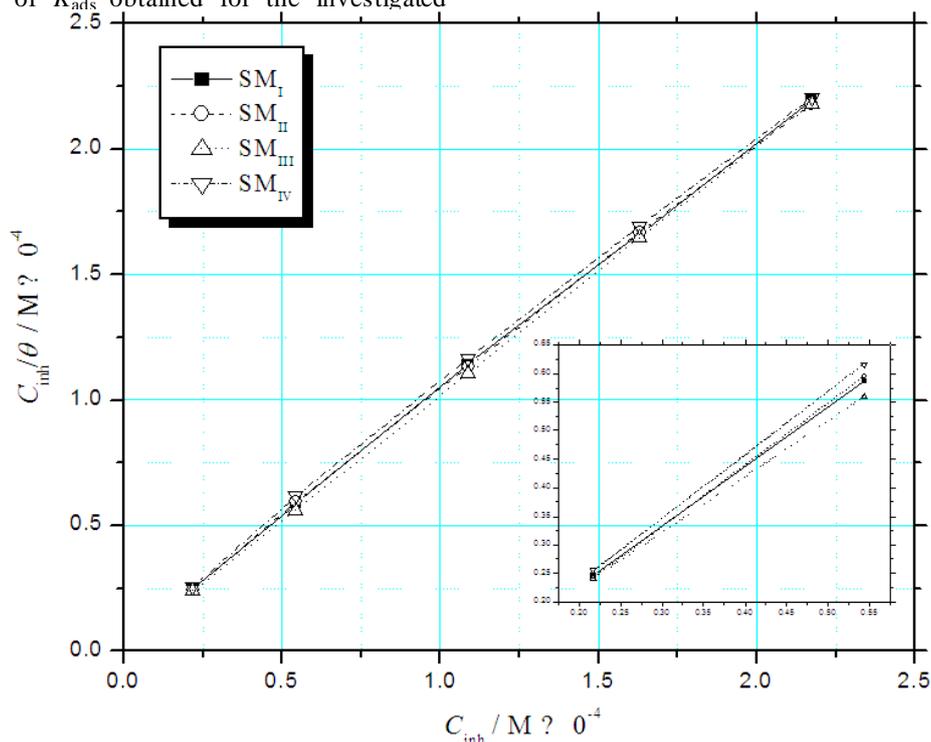


Figure 6. Langmuir adsorption isotherm (C_{inh}/θ vs. C_{inh}) fitting of the obtained from LPR corrosion rate data for mild steel in CO_2 saturated 1.0% NaCl solution containing various concentrations of SM inhibitors at $50^\circ C$

Table 2. Thermodynamic parameters for the adsorption of the studied surfactants on mild steel electrode in CO_2 -saturated 1.0% NaCl solution

Inhibitors	Slope	Regression coefficients/ R	K_{ads} $M^{-1} \times 10^4$	ΔG_{ads}^o kJ mol^{-1}
SM _I	1.04	0.987	9.81	-42.34
SM _{II}	1.07	0.988	9.02	-41.12
SM _{III}	1.01	0.999	10.09	-44.23
SM _{IV}	1.08	0.996	8.45	-40.98

3.4. Surface Characterization by Scanning Electron Microscopy

Figure 7 a, b shows SEM image of the surface of the mild steel specimen after immersion in CO_2 -saturated 1.0% NaCl solution for 5 days in the absence and presence of 100 ppm SM_{III} at $50^\circ C$. The micrograph reveals that (Figure 7a), the surface is strongly damaged in the absence of the inhibitor (Thick porous layer of corrosion product

was formed). Figure 7b and SEM image of the surface of mild steel specimen after immersion for the same time interval in the investigated aggressive media containing 100 ppm of the inhibitor (SM_{III}). The micrograph reveals that, there is a decrease in the corrosion sites and pits over the surface of the mild steel. In Figure 7b it appears that, the most of electrode surface is covered by protective

layer and surface became more smoothly. This suggests that the inhibitor is strongly adsorbed on metal surface, and this hinders the corrosion process. From these observations we can say that, SM_{III} inhibitor give a good inhibition effect for the mild steel and this confirms the results obtained from the corrosion measurements.

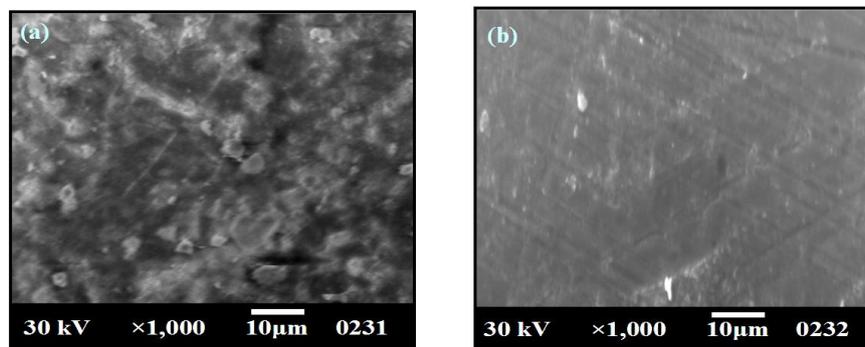


Figure 7. Scanning electron micrographs of steel samples (at $x = 1000$) (a) after immersion in 1.0% NaCl solution saturated with CO_2 for 5 days, (b) after immersion in 1.0% NaCl solution saturated with CO_2 for 5 days in presence of 100 ppm of the inhibitor SM_{III}

4. Conclusion

In the above results and discussion, the following conclusions are drawn:

1) Complex surfactants from the type of fatty acids were synthesized based on vegetable oils, and their structure was confirmed by physical-chemical spectroscopic methods.

2) The corrosion inhibition efficiencies of SM inhibitors were studied by LPR corrosion rate. Test results show that the surfactants studied are efficient inhibitors for mild steel corrosion in CO_2 -saturated solution even at low concentration. The inhibition efficiencies increased with the increase of SM concentrations.

3) The results also show that the inhibition efficiencies in the case of inhibitors obtained on the basis of corn oil are higher compared with those of inhibitors obtained based on other oils at the same conditions. This behavior may be due to the difference in fatty acids compositions of the investigated oils.

4) The adsorption of the investigated surfactants on the mild steel obeyed the Langmuir adsorption isotherm model.

5) SEM observations of the electrode surface showed that a surface film of inhibitor is formed on the electrode surface.

Acknowledgments

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