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Development of novel anionic surfactants based on different vegetable oils for protection of carbon steel from CO_2 corrosion

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ABSTRACT

Inhibiting properties and adsorption of some inhibitors synthesized based on vegetable oils (sunflower and cottonseed oils) on *carbon* steel in CO_2-saturated solution was investigated by using linear polarization corrosion rate and surface tension measurements. The results showed that all synthesized inhibitors were good inhibitors and their inhibition efficiencies were significantly increased with increasing the concentration of surfactant. The inhibition efficiencies in the case of surfactants obtained on the basis of sunflower oil are higher compared with those of surfactants obtained on the basis of cottonseed oil at the same conditions. Adsorption of the all inhibitors on the carbon steel surface was found to obey Longmuir's adsorption isotherm. The change in free energy (ΔG^{o}_{ads}) values indicates that the adsorption is chemical.

KEYWORDS

Inhibiting properties;
Surface activity;
Adsorption;
Sunflower oil;
Cottonseed oil;
Carbon steel.

INTRODUCTION

Carbon dioxide corrosion of carbon steel is a major problem in the oil and gas industry, and it occurs at all stages of production from downhole to surface equipment and processing facilities[1]. Notably, the corrosion of carbon steel in wet gas and multiphase pipelines is responsible for lost production and costly repairs, and it is therefore imperative to monitor the carbon dioxide corrosion process by using appropriate electrochemical techniques. Consequently, in situ monitoring of corrosion is an integral part of any corrosion management program, enabling the early detection of unexpected changes in the corrosion activity of a system.

The half-reactions and overall reactions involved in the carbon dioxide corrosion of steel are[2]:

$Fe \rightarrow Fe^{2+} + 2e^-$

$2H_2CO_3 + 2e^- \rightarrow 2HCO_3^- + H_2$

$Fe + 2H_2CO_3 \rightarrow Fe(HCO)_3 + H_2$

And/ or

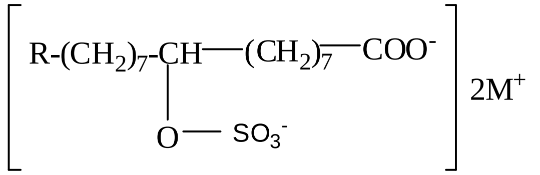
$Fe + H_2CO_3 \rightarrow FeCO_3 + H_2$

Importantly, the $Fe(HCO_3)_2$ and/or $FeCO_3$ corrosion products build up over time, partially passivating the corroding steel interface.

The corrosion scale may have important ramifications for the carbon dioxide corrosion of steel, as the corrosion scale may provide partial protection against corrosion, and it may influence the efficacy of corrosion control practices (e.g., the use of corrosion inhibitors) by altering the chemical nature of the steel surface.

Corrosion inhibitors are the chemicals that minimize or prevent corrosion if they are added at low concentrations in an aggressive environment. Effectiveness of the organic inhibitors depends on their adsorption rates and covering capabilities on metal surface[3]. It has been reported by many authorities that the adsorption of organic inhibitors is related mainly to the molecular structure, surface charge of a metal and type of electrolytes[4–6]. It is well known that the presence of hydrophilic and hydrophobic groups in the inhibitor favours the adsorption process at the electriferous surface[7]. Thus, the application of conventional surfactants made up of one hydrophilic head group and one hydrophobic chainas corrosion inhibitors have been widely studied. It was found that these amphiphilic compounds could adsorb on metal surface to form a protective layer and have a marked inhibiting efficiency near their critical micellar concentrations[8–17].

In this work, we have synthesized salts of sulfated fatty acids isolated from sunflower and cottonseed oils and studied their anti-corrosive properties in carbon dioxide environments at 50 °C. The salts were prepared in different compositions (Scheme1). The structures of synthesized surfactants were confirmed by physical-chemical spectroscopic methods.



$$\left[R\text{-}(CH_2)_7\text{-}\underset{\underset{O-SO_3^-}{|}}{CH}\text{—}(CH_2)_7\text{—}COO^- \right] 2M^+$$

Scheme 1: Molecular structure of the synthesized anionic surfactants, where M=Na^+ (Inhibitor I), K^+ (Inhibitor II), NH_4^+ (Inhibitor III), -HN-CH_2-CH_2-OH (Inhibitor IV), -N-(CH_2-CH_2-$OH)_2$ (Inhibitor V).

MATERIALS AND METHODS

Chemical composition of carbon steel alloy

Electrodes are made of carbon steel grade 080A15 and have an area of 7.9 cm^2. The mechanical properties of the carbon steel measured at room temperature were provided by supplier shown as follows: tensile strength equal to 490 MPa and elongation to failure equal to 16%. The Chemical composition of low carbon steel used in this study was given in TABLE 1. The data was provided by *European Corrosion Supplies Ltd*

TABLE 1 : Chemical composition of low carbon steel

Element	Si	Ni	Cr	C	S	P	Mn	Fe
Content, (wt. %)	0.24	0.01	0.10	0.18	0.05	0.05	0.50	Balance

Synthesis of the inhibitors

The inhibitors were synthesized in the laboratory on the basis of sunflower and cottonseed oil. Saponification of oils is producing a mixture from fatty acid salt and glycerol, then mix in the interaction of hydrochloric acid. The resulting acid was separated and -washed with hot water. The physical and chemical properties of acid were studied and given in TABLE 2.

Fatty acids which produced from cottonseed oil were used as base materials for sulfating process. The product is Sulfated fatty acid (pH=5.1). The chemical structure of the synthesized compound was confirmed by FTIR spectroscopy and physical- chemical methods (TABLE 2).

Five types from Salts of Sulfated fatty acids were synthesized in high purity by the following compositions: [R-CH-(OSO_3M)-COOM] (where M= Na, K, NH_4, –NH-CH_2-CH_2-OH and –N-(CH_2-CH_2-$OH)_2$). The alcoholic solutions of salts were prepared, and characterized by physical-chemical spectroscopic methods.

Corrosion inhibition test

The aggressive solution, 1% NaCl, was prepared by dissolving of analytical grade NaCl in distilled water. The concentration range of the prepared surfactants was from 25 to 100 ppm used for corrosion measurements. All solutions were prepared using a mixture from distilled water and isopropyl alcohol in a ratio 70:30.

Corrosion measurements

To study the corrosion protection ability of the synthesized surfactants was used by one of the newest devices in recent years ACM GILL AC. The apparatus consists of a monitor, CPU, potentiometer

TABLE 2 : Physical and chemical properties of fatty acid and sulfated fatty acid synthesized based on sunflower and cottonseed oil

Oil	Obtaining acid	Acid number mg KOH/g	Molecular weight g\mol	Iodine number 100 g iodine/g of sample	Density d_4^{20} kg/m^3	Refraction, η_{20}^{D}
Oil (I) sunflower	Fatty acid	145.6	279	112	907.5	1.4680
	Sulfated fatty acid	280	377	----	901.1	1.4610
Oil (II) cottonseed	Fatty acid	142.5	275	115	911	1.4670
	Sulfated fatty acid	269	373	----	917	1.4590

ACM GILL AC, four pieces of glasses with a capacity of 4000 ml, electrodes, CO_2 tank and installation, regulating the quantity fed CO_2. The prepared 1% - of the solution sodium chloride was stirred by a magnetic stirrer for 30 min in 4000 ml. The prepared solution poured into the 4 glass beakers (1000 ml for each one). Then these beakers were placed on a heater at 50 º C for 1 hour under a pressure of 0.9 bars. The solution was saturated with carbon dioxide. After that, the electrodes were placed in the medium and are connected through a potentiometer ACM GILL AC. The surface of working electrode is cleaned by acetone before using, these electrodes are using for one time. After 1 hour, except for 1 beaker, the remaining 3 is fed with the suitable amount of inhibitor and continued supply of CO_2 under pressure of 0.9 bar until the end of the experiment.

The potential of the working electrode was varied by a Core Running program (Version 5.1.3.) 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 +10mV at 10 mV/min around the rest potential is performed.

The Core Running program converts a corrosion current in mA/cm^2 to different relationships (building on the density of electricity mA/cm^2, the corrosion rate of the time (mm/year or hour)).

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 inhibitors.

RESULTS AND DISCUSSION

LPR corrosion rate

LPR corrosion rate (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, in which a small direct-current voltage is applied to a pair of identical electrodes and the resultant current is measured. A limitation of the bubble-test method is that it does not provide information on the effect of shear stress on the performance of a given corrosion inhibitor.

Figures 1 and 2 show that, the change in corrosion rate (CR) with time for low carbon steel in 1%NaCl solution saturated with CO_2 containing different concentrations form inhibitors I and V at 50°C synthesized based on sunflower and cottonseed oils. The inhibitor was added after 1 hour of exposure because at this time the corrosion potential got stable, allowing the measurement of the corrosion rate (CR) prior the injection of the inhibitor. The initial CR, without inhibitor, was measured to be between 2.59 and 3.78 mm y^{-1}.

Variation of the CR for inhibitors I and V (synthesized based on sunflower and cottonseed oils) at different concentrations are presented in Figures 1 and 2. Corrosion parameters were calculated on the basis of LPR test (TABLE 3). The inhibition efficiency (*IE*) and degree of surface coverage were calculated according to the following equations[18]:

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

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

Where CR_o is the corrosion rate without inhibitor and CR_i the corrosion rate when inhibitor is present.

The results showed that all synthesized inhibitors were good inhibitors and their inhibition efficiencies were significantly increased with increasing the concentration of surfactant. The increase of inhibitor efficiency with increasing the concentration can be interpreted on the basis the adsorption amount and the coverage of surfactants molecules, increases with increasing concentration. The inhibition efficiency of the investigated inhibitors which synthesized based on sunflower oil was increased in the following order: IV> V> III > I >II, but for the inhibitors which synthesized based on cottonseed oil was increased in the following order: V> IV> III > I >II (after 20 hours). This could be attributed to the increase of the number of actives sites, the electron densities and the molecular size[19].

The surface coverage (θ) is found to depend on the concentrations of the inhibitors. The surface coverage rates are increased with the increase of the surfactant concentrations. This indicates that the inhibitory action of the investigated inhibitors against carbon steel corrosion can be attributed to the adsorption of these molecules on the metal surface, limits the dissolution of carbon steel, and the adsorption amounts of surfactants on carbon steel increase with concentrations in the corrosive solutions. Conclusively, the surfactant inhibitor, having near unity θ (see TABLE 3), was considered as a good physical barrier shielding the corroding surface from corrosive medium and dumping the corrosion rate of carbon steel significantly.

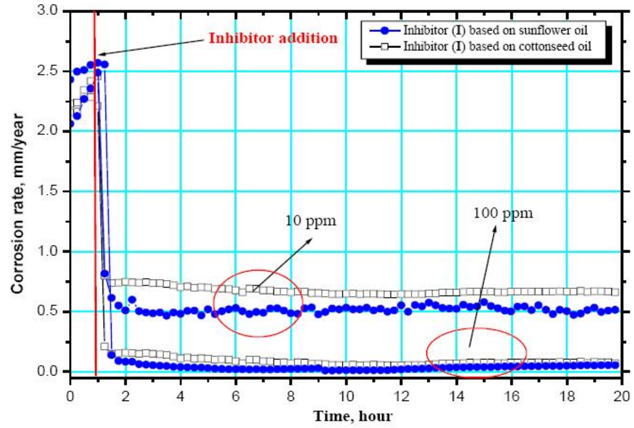


Figure 1 : Variation of the Corrosion rate with time for carbon steel in 1 % NaCl solution saturated with CO_2 containing different concentrations of inhibitor (I) at 50 °C.

—●— Inhibitor (I) based on sunflower oil,

—□— Inhibitor (I) based on cottonseed oil.

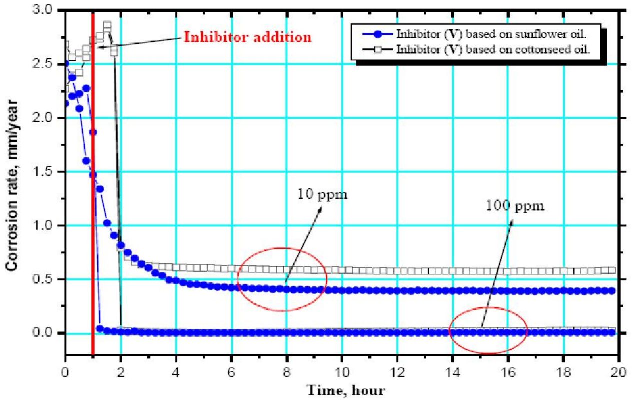


Figure 2 : Variation of the Corrosion rate with time for carbon steel in 1 % NaCl solution saturated with CO_2 containing different concentrations of inhibitor (V) at 50 °C.

—●— Inhibitor (V) based on sunflower oil,

—□— Inhibitor (V) based on cottonseed oil

The results also show that the inhibition efficiencies in the case of salts obtained on the basis of sunflower oil are higher compared with those of salts obtained on the basis of cottonseed oil at the same conditions. This behavior may be due to the difference in fatty acids compositions of two oils. The fatty acid composition of oils from vegetable sources varies depending on plant origin and sort[20].

Surface activity measurements

The values of surface tension (γ) were measured at 298 K for various concentrations of the inhibitors. The measured values of (γ) were plotted against of log inhibitor concentration, as shown in Figure 3. This figure showed two characteristic regions. The first at low concentrations and characterized by continuous decrease of the surface tension values due to the adsorption of surfactant molecules at the interface. The second region locates at higher surfactant concentrations where the surface tension values are almost stable. The intercept of the two straight lines designates the critical micelle concentration (CMC), where saturation in the surface adsorbed layer takes place[21,22].

The surface active properties of the surfactants; effectiveness (π_{cmc}), maximum surface excess (Γ_{max}) and

TABLE 3 : The corrosion parameters for carbon steel electrode in 1% solution of NaCl saturated with CO_2 in the absence and presence of various concentrations of inhibitor I (based on sunflower and cottonseed oils) at 50 ° C

10	Concentration, ppm	Corrosion rate (mm/year)	surface coverage θ	The Inhibition Efficiency, *IE%*
1% NaCl without Inhibitor	0.0	3.784		---------
Inhibtor I Based on sunflower oil	25	0.44915	0.88	88.11
	50	0.22591	0.94	94.02
	75	0.1353	0.96	96.42
	100	0.05279	0.98	98.60
Inhibtor I Based on cottonseed oil	25	0.51503	0.86	86.39
	50	0.29581	0.92	92.18
	75	0.20679	0.94	94.53
	100	0.12563	0.96	96.67

minimum area per molecule (A_{min}). Were calculated using the following equations[22]:

$$\pi_{cmc} = \gamma_0 - \gamma_{cmc} \qquad (3)$$

$$\Gamma_{max} = \frac{-1}{RT[\partial\gamma / \partial \ln C]_{\Gamma}} \qquad (4)$$

$$A_{min} = \frac{1}{\Gamma_{max} \times N_A} \qquad (5)$$

Where □^{3}/lnC is maximum slope, γ_0 is the surface tension of pure water, γ_{cmc} the surface tension at critical micelle concentration and N_A is the Avogadro's number.

The standard free energies of micellization ΔG^o_{mic} and adsorption ΔG^0_{ads} understanding the process of micellization and adsorption are important for explaining the effects of structural and environmental factors on the value of the CMC and for predicting the effects on it of new structural and environmental variations. Standard free energy of micellization and adsorption () play an important role in facilitating such understanding. The standard free energy of micellization and adsorption are given by[23]:

$$\Delta G^o_{mic} = RT \ln CMC \qquad (6)$$

$$\Delta G^o_{ads} = \Delta G^o_{mic} - 0.6023\, \pi_{CMC}\, A_{min} \qquad (7)$$

The data obtained from surface tension measurements were calculated. The values of π_{cmc} at 298 K indicate that the prepared compound gives large reduction of surface tension at CMC, so that, the compound acts as effective corrosion inhibitor for carbon steel in 1% NaCl solution saturated with CO_2. The order of decreasing inhibition efficiency of these anionic surfactants is IV> V> III > I >II (Sunflower oil) and/or V> IV> III > I >II (Cottonseed oil)**.** Higher molecular size and high electron density on the adsorption centers may be responsible for high corrosion efficiency.

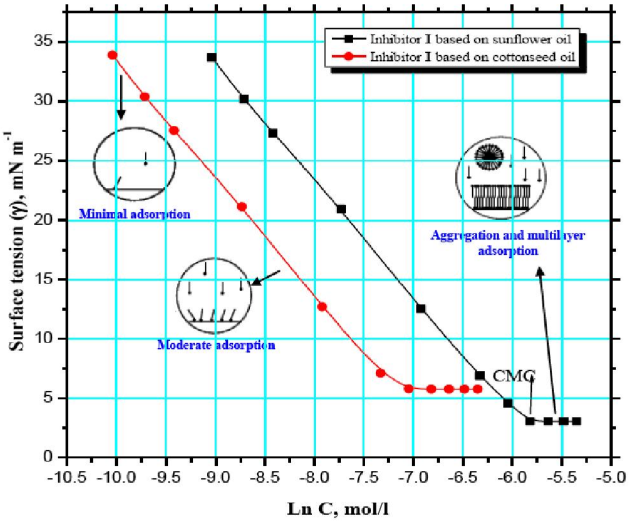


Figure 3 : Change of surface tension (*f*×) with the concentration of the inhibitor I at 25 ºC.

—■— Inhibitor I based on sunflower oil

—●— Inhibitor I based on cottonseed oil

The results also show that the values of ΔG^o_{mic} and are ΔG^0_{ads} always negative, indicating the spontaneous of these two processes, but there is more increase in negativity of than of , indicating the tendency of the molecules to be adsorbed at the interface[23]. Figure 4(a) shows the mode of adsorption of the surfactant as individual molecules at very low concentration of the inhibitor. Figure 4(b) shows the mode of adsorption of the surfactant as hemi-micelles at moderate concentration. The hemi-micelle phase in the surfactant

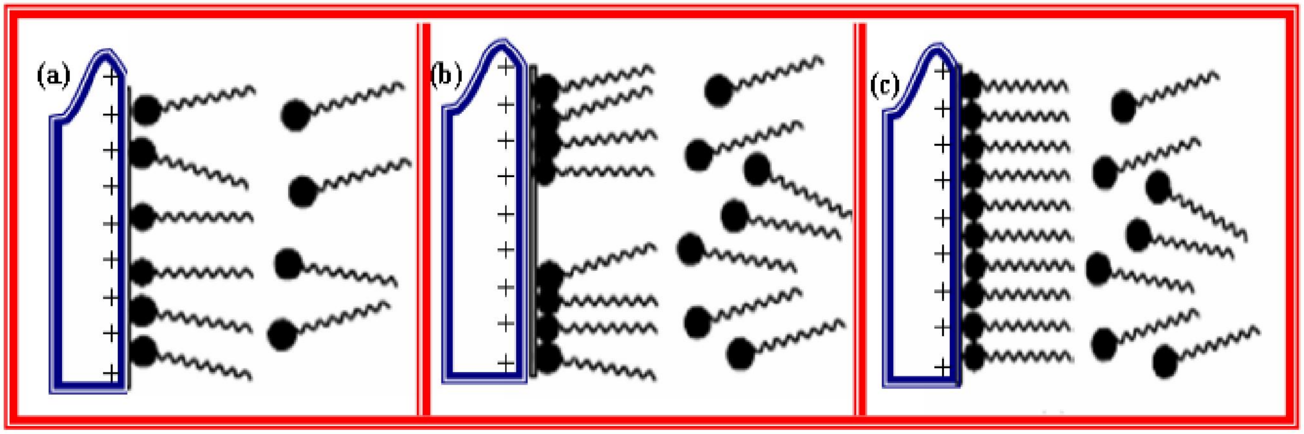


Figure 4 : Schematic representation of the inhibitor adsorption on carbon steel surface (a) Adsorption as single molecule at low concentration (b) Hemimicelle formation at higher concentration (c) Formation of multi-layers at very high concentration.

solution means the start point to collect the surfactant in duplet, triplet, or quadrate before forming the complete micelles.

Adsorption isotherm

In order to obtain the isotherm, the linear relation between θ values and C_{inh} must be found. Attempts were made to fit the θ values to various isotherms including Langmuir, Temkin, Frumkin and Flory–Huggins. By far the best fit is obtained with the Langmuir isotherm. This model has also been used for other inhibitor systems[24,25]. According to this isotherm, θ is related to C_{inh} by[26]:

$$\frac{C_{inh}}{\theta} = C_{inh} + \frac{1}{K_{ads}} \qquad (8)$$

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 the adsorption of 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^2) and the slopes parameter variations were calculated. All correlation coefficient ($R^2 > 0.9597$) indicated that the inhibition of carbon steel by these inhibitors was attributed to the adsorption of surfactant molecule 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[27] and unexpected from Langmuir adsorption isotherm. They might be the results of the interactions between the adsorbed species on the metal surface[28,29].

The values of K obtained from the Langmuir adsorption isotherm are calculated, together with the values of the Gibbs free energy of adsorption () calculated from

$$K_{ads} = \frac{1}{55.5}\exp\left(-\frac{\Delta G^{\circ}_{ads}}{RT}\right) \qquad (9)$$

Where R is the universal gas constant, T the thermodynamic temperature and the value of 55.5 is the concentration of water in the solution.

The high value of K_{ads} indicates that the inhibitors molecules possess strong adsorption ability onto the metal surface. It is also noted that, the high values of K for inhibitors V and IV (which synthesized based on two oils) indicate stronger adsorption on the carbon steel surface than the inhibitors I, II and III. This can be simply explained by the presence of additional donor atoms, such as nitrogen, in the appended functional groups.

The negative ΔG^{0}_{ads} values, calculated from Eq. (9), are consistent with the spontaneity of the adsorption process and the stability of the adsorbed layer on the carbon steel surface. Generally, values of ΔG^{0}_{ads} up to -20 kJ mol^{-1}, the types of adsorption was regarded as physisorption, the inhibition acted due to the electrostatic interactions between the charged molecules and the charged metal, while the values around -40 kJ mol^{-1} or smaller were associated with chemisorption as a result of sharing or transfer of electrons from organic

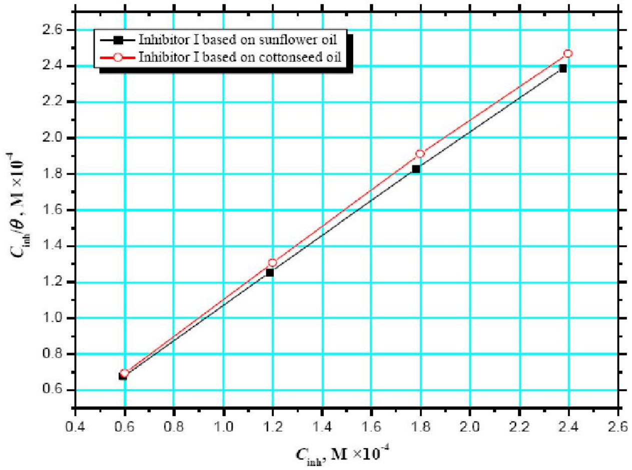


Figure 5 : Langmuir adsorption isotherms for carbon steel in 1% NaCl saturated with CO_2 in the presence of different inhibitors at 50 °C.

—■— Inhibitor I based on sunflower oil

—○— Inhibitor I based on cottonseed oil

molecules to the metal surface to form a coordinate type of bond (chemisorption)[30]. The values of ΔG^0_{ads} in our measurements for all inhibitors synthesized based on two oils were found around 40 kJ mol^{-1}, thus the adsorption mechanism of the surfactants on carbon steel in 1% NaCl solution saturated with CO_2 was typical chemisorption.

Corrosion inhibition mechanism by surfactant molecule

Corrosion inhibition has complex mechanism and depends on the formation of mono- or multidimensional protective layers on the metal surface. The protective nature of the surface layer depends on many factors: interaction between inhibitors and substrate, incorporation of the inhibitor in the surface layer, chemical reactions, electrode potentials, concentration of the inhibitor, temperature and properties of the corresponding surface, etc. The first stage in the action mechanism of the surfactants as corrosion inhibitors in aggressive media is adsorption of the surfactant molecules onto the metal surface. The adsorption process is influenced by the nature and the surface charge of the metal, the chemical structure of the surfactant, and the nature of the aggressive electrolyte. Adsorption of the surfactant molecules on the metal surface can be expressed according to the following equation[31]:

$$\mathbf{Surfactant_{(sol.)} + nH_2O_{(ads.)} = Surfactant_{(ads.)} + nH_2O_{(sol.)}} \quad \mathbf{(10)}$$

Where n is the number of water molecules removed from the metal surface for each molecule of surfactant adsorbed. It is clear that the value of n depends on the cross sectional area of the surfactant molecule with respect to that of the water molecule. Adsorption of the surfactant molecules occurs because the interaction energy between the surfactant molecules and the metal surface is higher than that between water molecules and the metal surface. So the inhibition effect by surfactants is attributed to the adsorption of the surfactant molecules via their functional groups onto the metal surface. The adsorption rate is usually rapid and hence the reactive metal is shielded from the aggressive environment. Corrosion inhibition depends on the adsorption ability of the surfactant molecules on the corroding surface, which is directly related to the capacity of the surfactant to aggregate to form clusters (micelles)[32]. Also, the adsorbed molecules can be incorporated into the oxide layer and react chemically to form a more protective surface network[33].

CONCLUSIONS

Linear polarization corrosion rate and surface tension measurements were employed to study the corrosion inhibition of carbon steel in CO_2-saturated solutions using some novel anionic surfactants as corrosion inhibitors synthesized based on sunflower and cottonseed oils. The principal conclusions are:

1. The structures of synthesized inhibitors were confirmed by physical-chemical spectroscopic methods.
2. All inhibitors were found to be effective inhibitors for carbon steel corrosion in CO_2-saturated solutions.
3. The corrosion process is inhibited by the adsorption of these surfactants on carbon steel surface. Inhibition efficiency increased with increase in concentration of the surfactants.
4. The inhibition efficiencies in the case of inhibitors obtained on the basis of sunflower oil are higher compared with those of inhibitors obtained on the basis of cottonseed oil at the same conditions. This behavior may be due to the difference in fatty acids compositions of two oils.
5. The adsorption of synthesized surfactants on car-

bon steel surface obeyed Langmuir adsorption isotherm. The high value of adsorption equilibrium constant and negative value of standard free energy of adsorption suggested that surfactants are strongly adsorbed on carbon steel surface, and are chemically adsorbed on the metal surface.

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