

# Novel Synthesized Surfactants Based on Palm Oil and Monoethanolamine as Corrosion Inhibitors for Mild Steel in CO<sub>2</sub> Environments

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**Abstract** Potentiodynamic polarization and linear polarization resistance corrosion rate measurements were carried out to study the corrosion inhibition of mild steel in 1% NaCl saturated with CO<sub>2</sub> by some novel surfactants type of fatty acids derivatives synthesized based on palm oil and monoethanolamine. The surface tension at 298 K was measured; the critical micelle concentration (CMC) and some surface active parameters were calculated. The inhibition efficiency was found to increase with increasing concentration. The results obtained show that the prepared surfactants act as mixed-type inhibitors. The inhibitive effect of these compounds was explained on the basis of adsorption on the metal surface. The adsorption process follows Langmuir adsorption isotherm. Some activated thermodynamic parameters were computed and discussed.

**Keywords** Surfactants, Mild steel, Palm oil, Corrosion inhibition, CO<sub>2</sub>-saturated brine

## 1. Introduction

Mild steel is widely applied as the constructional materials in many industries due to its excellent mechanical properties and low cost. Carbon dioxide (CO<sub>2</sub>) corrosion is one of the most studied of corrosion in oil and gas industry. This is generally due to the fact that the crude oil and natural gas from the oil reservoir / gas well usually contains some level of CO<sub>2</sub>. The major concern with CO<sub>2</sub> corrosion in oil and gas industry is that CO<sub>2</sub> corrosion can cause failure on the equipment especially the main down hole tubing and transmission pipelines and thus can disrupt the oil/gas production [1-3]. Because of the general aggression of solutions saturated with CO<sub>2</sub>, inhibitors are commonly used to reduce the corrosive attack on metallic materials [4]. The selection of inhibitor is controlled by its economic availability, its efficiency to inhibit the substrate material and its environmental side effects. So that, most of the excellent inhibitors for corrosion of steel in carbon dioxide environments are organic compound containing nitrogen, oxygen and/or sulphur atoms [5-15]. The inhibiting action of these compounds is attributed as a first stage, to the adsorption of the additives to the metal/solution interface.

The adsorption process depends upon the nature and surface charge of the metal, the type of aggressive media, the structure of the inhibitor and the nature of its interaction with the metal surface.

Surfactants can be easily synthesized from relatively cheap raw materials, nontoxic and have surface active property. Surfactants based on fatty acids were used as corrosion inhibitors for steel in CO<sub>2</sub>-saturated solutions [16]. They inhibit the corrosion by the adsorption on the steel surface. The aim of this investigation is to examine the inhibitory effect of some surfactants based on palm oil toward the corrosion of mild steel corrosion in CO<sub>2</sub>-saturated 1% NaCl solution. Linear polarization resistance corrosion rate and potentiodynamic polarization techniques were used in this work to evaluate the inhibition efficiency of the tested compounds.

## 2. Experimental methods

### 2.1. Synthesis of Surfactants

Palm oil was reacted with monoethanolamine for 14 hours at 423-433 K. These processes produce fatty acid monoethanolamine amide. Based on the last prepared compound sulfating syntheses were performed. The Sulfating process was done in 500 ml ground glass three-neck flask equipped with a mechanical stirrer, a thermometer with a temperature controller, were charged

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with 100 gm fatty acid and then H<sub>2</sub>SO<sub>4</sub> was added drop by drop at the reaction temperature in the range from 343 to 348 K with stirring for 7 h. The product is sulfated fatty acid monoethanolamine amide. Five types from surfactants were synthesized in high purity by the following composition: [R-CH-(OSO<sub>3</sub>M)-CONH-CH<sub>2</sub>-CH<sub>2</sub>-OH] (where M = Na, K, NH<sub>4</sub>, -NH-CH<sub>2</sub>-CH<sub>2</sub>-OH and -N-(CH<sub>2</sub>-CH<sub>2</sub>-OH)<sub>2</sub>). List of the synthesized surfactants are shown in **Table 1**. The chemical structure of the synthesized surfactants was characterized by using FT-IR, Spectrum BX spectrometer using KBr disks.

## 2.2. Chemical Composition of Mild Steel Alloy

Mild steel was used for this study has the following composition: C 0.18%, Si 0.17%, Mn 0.70%, P 0.011%, S 0.03%, Ni 0.01%, Cr 0.01% and Fe balance.

## 2.3. Corrosion Measurements

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 inhibitors solutions were prepared using a mixture from distilled water and *alcohol* in a different ratio.

The measurements were performed on the rotating cylinder electrode as a working electrode. This working electrode was used for one time without polishing. The reference electrode was Ag/AgCl Electrode to which all potentials are referred.

The extrapolation of cathodic and anodic Tafel lines was carried out in a potential range ±100 mV with respect to corrosion potential ( $E_{\text{corr}}$ ) at scan rate of 1 mV/s. To remove any surface contamination and air formed oxide, the working electrode was kept at -1500 mV (Ag/AgCl) for 5 min in the tested solution, disconnected shaken free of adsorbed hydrogen bubbles and then cathodic and anodic polarization was recorded. ACM Gill AC instrument connected with a personal computer was used for the measurements.

**Table 1.** List of the prepared surfactants includes, code number, name and structure

Code number of the inhibitor	Name and abbreviation	Structure	molecular weight (g/mol)
C <sub>I</sub>	Sodium salt of sulfated fatty acid monoethanolamine amide (SSMA)	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R}-(\text{CH}_2)_8-\text{CH}-(\text{CH}_2)_7-\text{C}-\text{NH}-\text{CH}_2-\text{CH}_2-\text{OH} \\   \\ \text{O}-\text{S}-\text{O}^-\text{Na}^+ \\ \parallel \\ \text{O} \end{array}$	410
C <sub>II</sub>	Potassium salt of sulfated fatty acid monoethanolamine amide (PSMA)	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R}-(\text{CH}_2)_8-\text{CH}-(\text{CH}_2)_7-\text{C}-\text{NH}-\text{CH}_2-\text{CH}_2-\text{OH} \\   \\ \text{O}-\text{S}-\text{O}^-\text{K}^+ \\ \parallel \\ \text{O} \end{array}$	426
C <sub>III</sub>	Ammonium salt of sulfated fatty acid monoethanolamine amide (ASMA)	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R}-(\text{CH}_2)_8-\text{CH}-(\text{CH}_2)_7-\text{C}-\text{NH}-\text{CH}_2-\text{CH}_2-\text{OH} \\   \\ \text{O}-\text{S}-\text{O}^-\text{NH}_4^+ \\ \parallel \\ \text{O} \end{array}$	404
C <sub>IV</sub>	Sulfated fatty acid monoethanolamine amide - monoethanolamine complex (MCMA)	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R}-(\text{CH}_2)_8-\text{CH}-(\text{CH}_2)_7-\text{C}-\text{NH}-\text{CH}_2-\text{CH}_2-\text{OH} \\   \\ \text{O}-\text{S}-\text{O}^-\text{NH}_2-\text{CH}_2-\text{CH}_2-\text{OH} \\ \parallel \\ \text{O} \end{array}$	436
C <sub>V</sub>	Sulfated fatty acid monoethanolamine amide - diethanolamine Complex (DCMA)	$\begin{array}{c} \text{O} \\ \parallel \\ \text{R}-(\text{CH}_2)_8-\text{CH}-(\text{CH}_2)_7-\text{C}-\text{NH}-\text{CH}_2-\text{CH}_2-\text{OH} \\   \\ \text{O}-\text{S}-\text{O}^-\text{NH}-(\text{CH}_2-\text{CH}_2-\text{OH})_2 \\ \parallel \\ \text{O} \end{array}$	485

LPR test has been performed in brine saturated with CO<sub>2</sub> at 323 K, in turbulence fluid stream during 20 hours. The prepared 1% NaCl was stirred by a magnetic stirrer for 60 min in 4000 ml glass beaker. The prepared solution poured into the 4 glass beakers (1000 ml for each one). Then these beakers were placed on a heater at 50°C for 1 hour under a pressure of 0.9 bars. The solution was saturated with carbon dioxide (pH=5.7). 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<sub>2</sub> under pressure of 0.9 bar until the end of the experiment.

The potential of the working electrode was varied by a CoreRunning programme (Version 5.1.3.) through an ACM instrument Gill AC. The CoreRunning programme converts a corrosion current in mA/cm<sup>2</sup> to a corrosion rate in mm/year. A cylindrical mild 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.

#### 2.4. Surface Tension Measurements

The surface tension ( $\gamma$ ) of the investigated surfactants was measured using (DuNouy Tensiometer, Kruss Type 8451) for various concentrations of the prepared surfactants.

### 3. Results and Discussion

#### 3.1. Linear Polarization Corrosion Rate (LPR Bubble Test)

The linear polarization resistance corrosion rate test involved evaluating the corrosion of mild steel in CO<sub>2</sub>-saturated 1 % NaCl solution at 323 K. Fig. 1 a, b, c and d show that, the change in corrosion rate (CR) with time for mild steel in CO<sub>2</sub>-saturated 1 % NaCl solution containing different concentrations for surfactants (a) C<sub>I</sub>, (b) C<sub>II</sub>, (c) C<sub>III</sub> and (d) C<sub>V</sub> at 323 K. 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 addition of the inhibitor. The initial corrosion rate, without inhibitor was measured to be between 4.18 and 4.9 mm y<sup>-1</sup>. From Fig. 1 it can be observed that, in the baseline test (no inhibitor addition), CO<sub>2</sub> corrosion rate steadily increase with time since the corrosion process leaves iron carbide (Fe<sub>3</sub>C) left on the mild steel surface, which increases surface area of cathodic reaction and further increases the corrosion rate [17-19]. The increase in the corrosion rate had been attributed to the decrease in pH value from 6.77 to 5.70 (more acidic) after saturation with CO<sub>2</sub>. The inhibition

efficiency ( $\eta\%$ ) and surface coverage ( $\theta$ ) were calculated according to the following equations [3]:

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

$$\theta = \frac{CR_0 - CR_i}{CR_0} \quad (2)$$

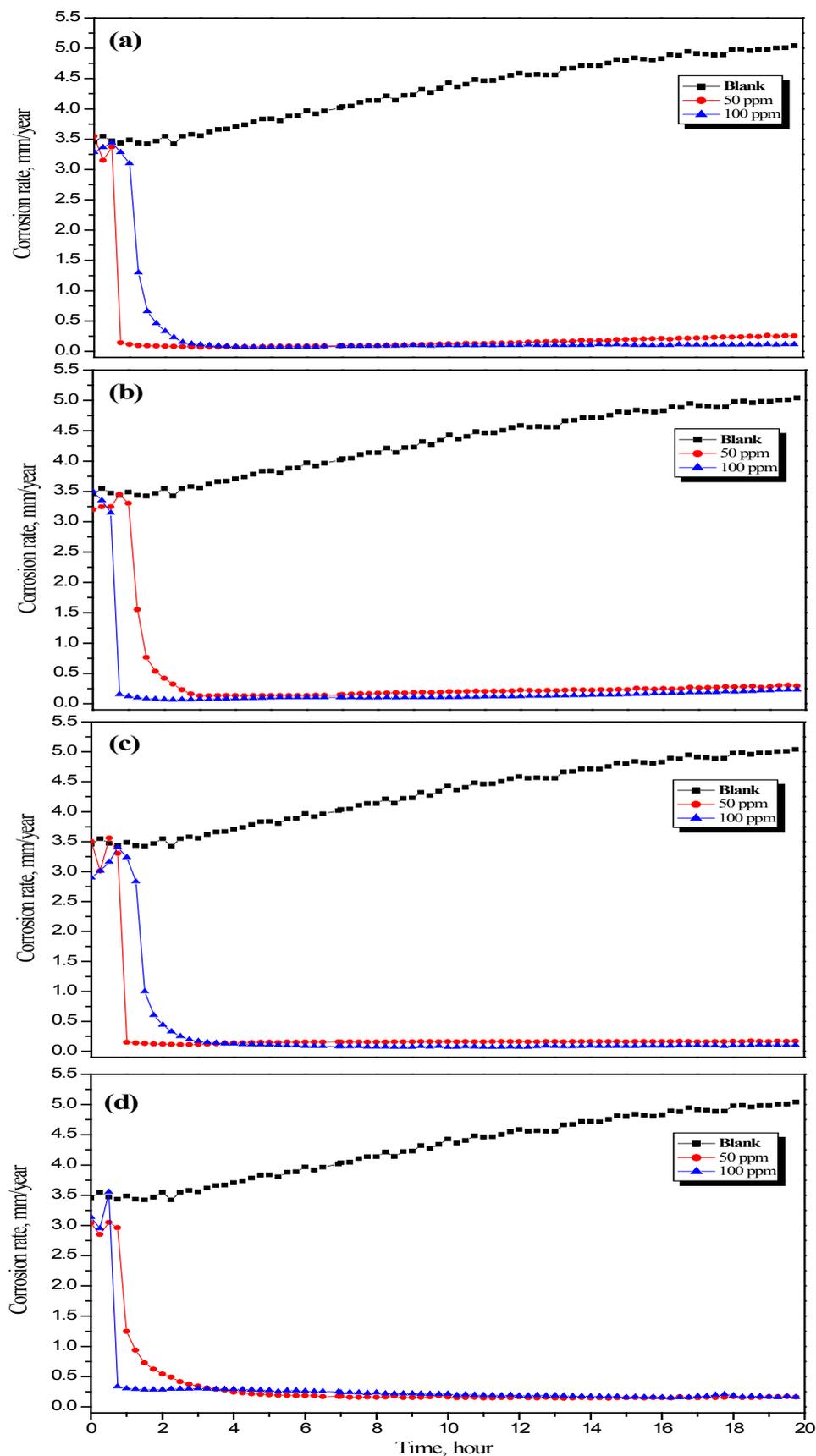
Where CR<sub>0</sub> is the corrosion rate without inhibitor and CR<sub>i</sub> is the corrosion rate when inhibitor is present. It can be seen that, the results of this series of experiments showed that, the addition of the studied surfactants greatly decreased the corrosion rate of mild steel as the inhibitor concentration increased in the given corrosive media. The corrosion rates in the presence of 100 ppm of C<sub>III</sub> decrease from 5.037 to 0.105 mm/yr and stabilize at this level. It is found that corrosion rate remains steady until the end of experiment. The inhibition efficiencies are 97.73%, 97.71%, 97.91%, 96.84% and 90.84% for inhibitors C<sub>I</sub>, C<sub>II</sub>, C<sub>III</sub>, C<sub>IV</sub> and C<sub>V</sub> respectively. This means that these surfactant inhibitors have a good ability to form protective layers on the metal surface, even in the presence of small concentrations.

Figure 2 shows the variation of the corrosion rate with time for C1018 mild steel in CO<sub>2</sub>-saturated brine in the presence of 100 ppm from different inhibitors at 323 K. This plot indicates that, the presence of all surfactants decreases the rate of corrosion. However, the maximum decrease in the corrosion rate was observed for inhibitor (C<sub>III</sub>) and the inhibition efficiency data showed that C<sub>III</sub> has greater interaction with mild steel compared to other compounds.

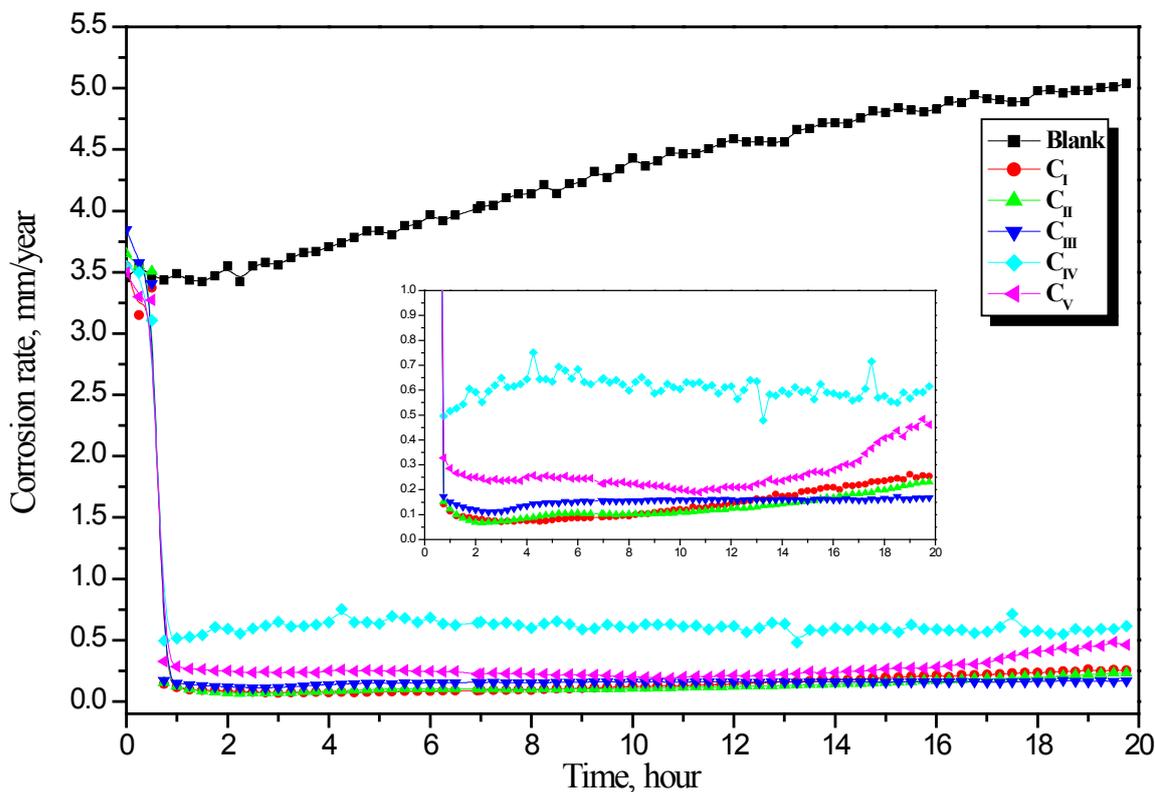
Table 2 shows the calculated values of corrosion rates, surface coverage and the inhibition efficiencies in the absence and presence of different concentrations of investigated inhibitors at 323 K. The data exhibited that, the corrosion rates, surface coverage and the inhibition efficiencies were found to depend on the concentrations of the inhibitors. The corrosion rate (CR) was decreased, and the surface coverage and inhibition efficiencies ( $\eta\%$ ) were increased with the increase of the inhibitor concentrations. The results also indicated that the percentage inhibition efficiency ( $\eta\%$ ) of the inhibitor (C<sub>III</sub>) was greater than that of the other inhibitors. The inhibition efficiency of the surfactant inhibitors decreased in the following order: C<sub>III</sub> > C<sub>I</sub> > C<sub>II</sub> > C<sub>IV</sub> > C<sub>V</sub> (at 100 ppm dose).

#### 3.2. Potentiodynamic Polarization Measurements

The effect of addition of the prepared surfactants of fatty acid derivatives on the anodic and cathodic polarization curves for mild steel in CO<sub>2</sub>-saturated brine solution at 323K was studied. The effect of increased concentration of compound C<sub>I</sub> is shown in Fig. 3 as an example of the studied surfactants. Similar results were obtained for the other inhibitors (not shown). The dependence of the degree of surface coverage ( $\theta$ ) and the inhibition efficiency ( $\eta\%$ ) on the concentration of the inhibitor were calculated using [16]:



**Figure 1.** Variation of the Corrosion rate with time for carbon steel in CO<sub>2</sub>-saturated 1 % NaCl solution containing different concentrations of inhibitors (a) C<sub>I</sub>, (b) C<sub>II</sub>, (c) C<sub>III</sub> and (d) C<sub>V</sub> based on palm oil at 323 K



**Figure 2.** Variation of the Corrosion rate with time for carbon steel in CO<sub>2</sub>-saturated 1% NaCl solution containing 100 ppm of different inhibitors at 323 K

**Table 2.** The corrosion parameters obtained from LPR corrosion rate measurements for mild steel electrode in CO<sub>2</sub>-saturated brine in the absence and presence of various concentrations of surfactants obtained based on palm oil at 323 K

Inhibitors	Concentration, ppm	Corrosion rate (mm/year)	Surface coverage $\theta$	The inhibition efficiency, $\eta$ %
Absence	0.0	5.037	-----	-----
C <sub>I</sub>	50	0.254	0.949	94.95
	100	0.114	0.977	97.73
C <sub>II</sub>	50	0.230	0.977	95.43
	100	0.115	0.954	97.71
C <sub>III</sub>	50	0.168	0.966	96.66
	100	0.105	0.979	97.91
C <sub>IV</sub>	50	0.162	0.967	96.78
	100	0.159	0.968	96.84
C <sub>V</sub>	50	0.875	0.826	82.62
	100	0.461	0.908	90.84

$$\eta\% = \frac{I_{uninh.} - I_{inh.}}{I_{uninh.}} \times 100 \quad (3)$$

$$\theta = \frac{I_{uninh.} - I_{inh.}}{I_{uninh.}} \quad (4)$$

where  $I_{uninh.}$  and  $I_{inh.}$  are the corrosion current densities determined by the intersection of the extrapolated Tafel lines and the corrosion potential for mild steel in uninhibited and inhibited acid solution, respectively. The following electrochemical parameters; cathodic and anodic Tafel

slopes ( $b_a$  and  $b_c$ , respectively), corrosion potential ( $E_{corr.}$ ), corrosion current density ( $I_{corr.}$ ), degree of surface coverage ( $\theta$ ) and the percentage inhibition efficiency ( $\eta\%$ ) were calculated. From the results given in **Figs. 4 a, b** and **5**, it is clear that, as the concentration of the inhibitor increase, the following could be observed.

1) The  $E_{corr}$  values of all synthesized surfactants were shifted slightly toward both cathodic and anodic directions and did not show any definite trend in CO<sub>2</sub>-saturated brine (**Fig. 4b**). This may be contributed to the mixed-type behavior of the studied inhibitors. It can be observed, the

shift in  $E_{\text{corr}}$  that is characteristic of anodic and anodic/cathodic inhibitor [20].

II) The corrosion current density ( $I_{\text{corr}}$ ) decreases as the concentration of inhibitors is increased (Fig. 3a). These results suggest that retardation of the electrodes processes occurs, at both cathodic and anodic sites, as a result of coverage of these sites by surfactant molecules. However, the maximum decrease in  $I_{\text{corr}}$  was observed for C<sub>III</sub>. This could be attributed to the increase of the number of active sites, the electron densities and the molecular size [21].

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

IV) The inhibition efficiency ( $\eta\%$ ) increases as the concentration of inhibitors is increased (Fig. 5). The increase of inhibitor efficiency with increasing the concentration can be interpreted on the basis the adsorption amount and the coverage of inhibitor molecules, increases with increasing concentration [22].

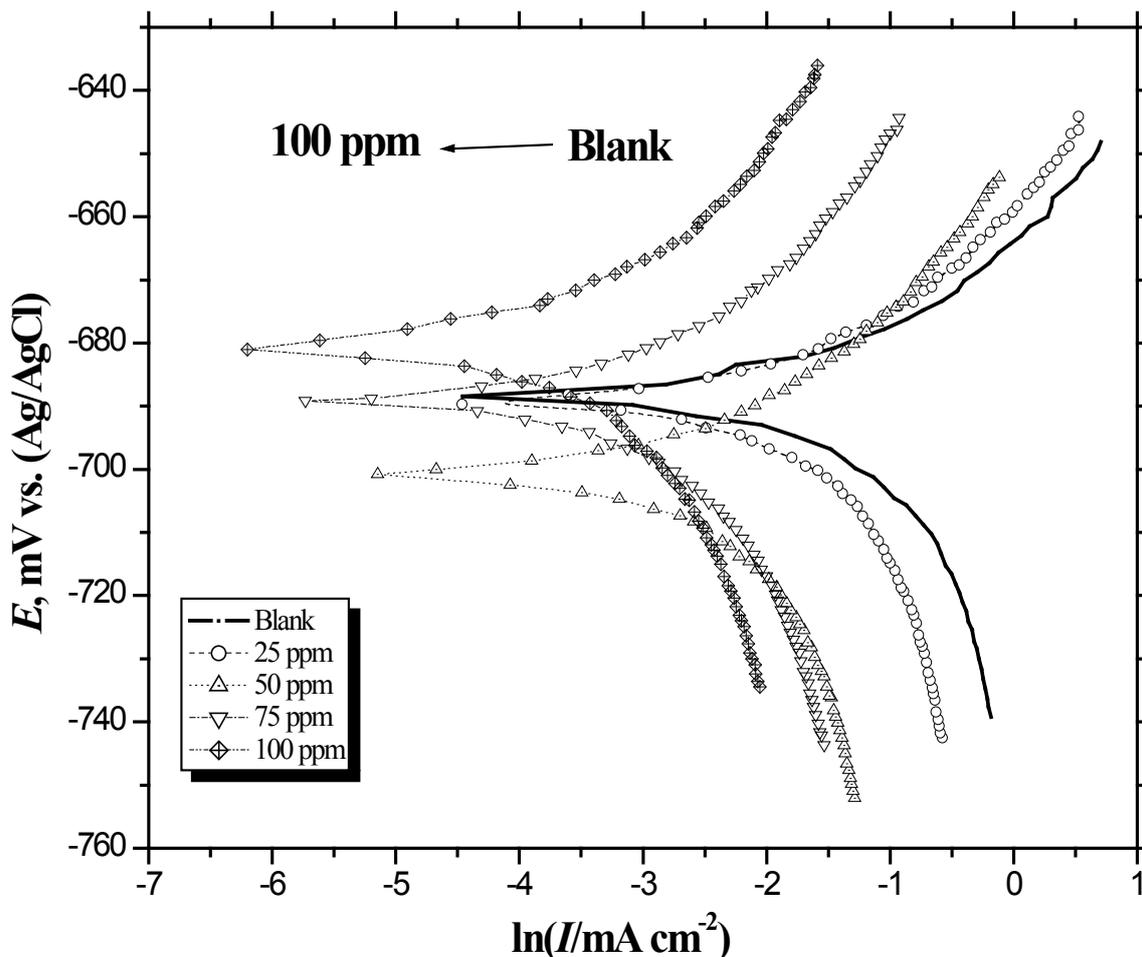
V) The values of  $\eta\%$  of the five tested compounds decrease in the following order:

$$C_{\text{III}} > C_{\text{I}} > C_{\text{II}} > C_{\text{IV}} > C_{\text{V}} \text{ (at 100 ppm dose)}$$

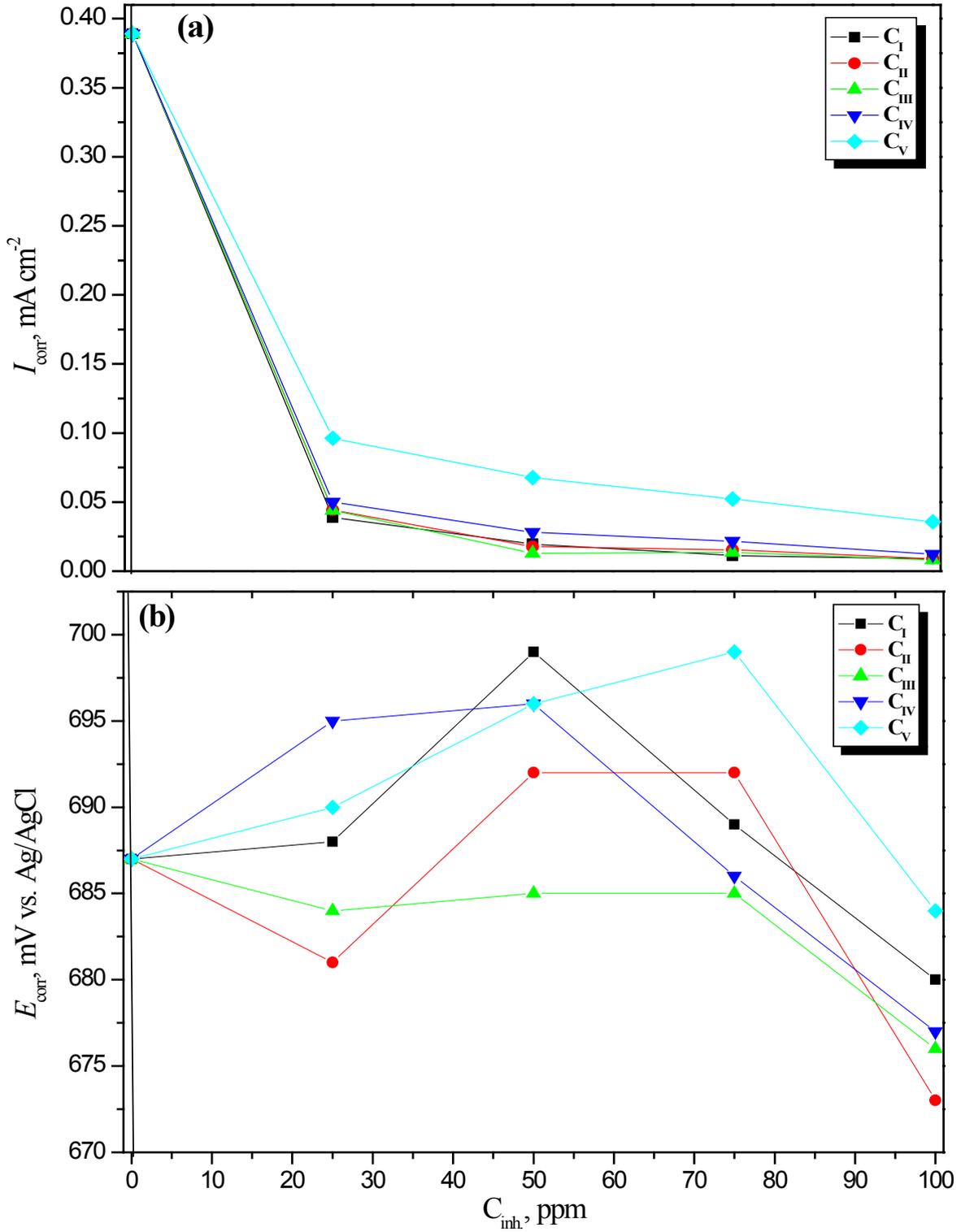
The inhibition of mild steel is attributed to the adsorption of surfactant molecules on the steel surface by the hydrophilic head groups. The hydrophobic chain may be oriented towards the aqueous medium and may be arranged horizontally to the steel surface [23].

The values of surface tension ( $\gamma$ ) were measured at 298 K for various concentrations of the prepared inhibitor C<sub>I</sub>. The relation between surface tension ( $\gamma$ ) and the logarithm of concentration ( $\log C$ ) is shown in Fig. 6. The intercept of the two straight lines designates the critical micelle concentration (CMC).

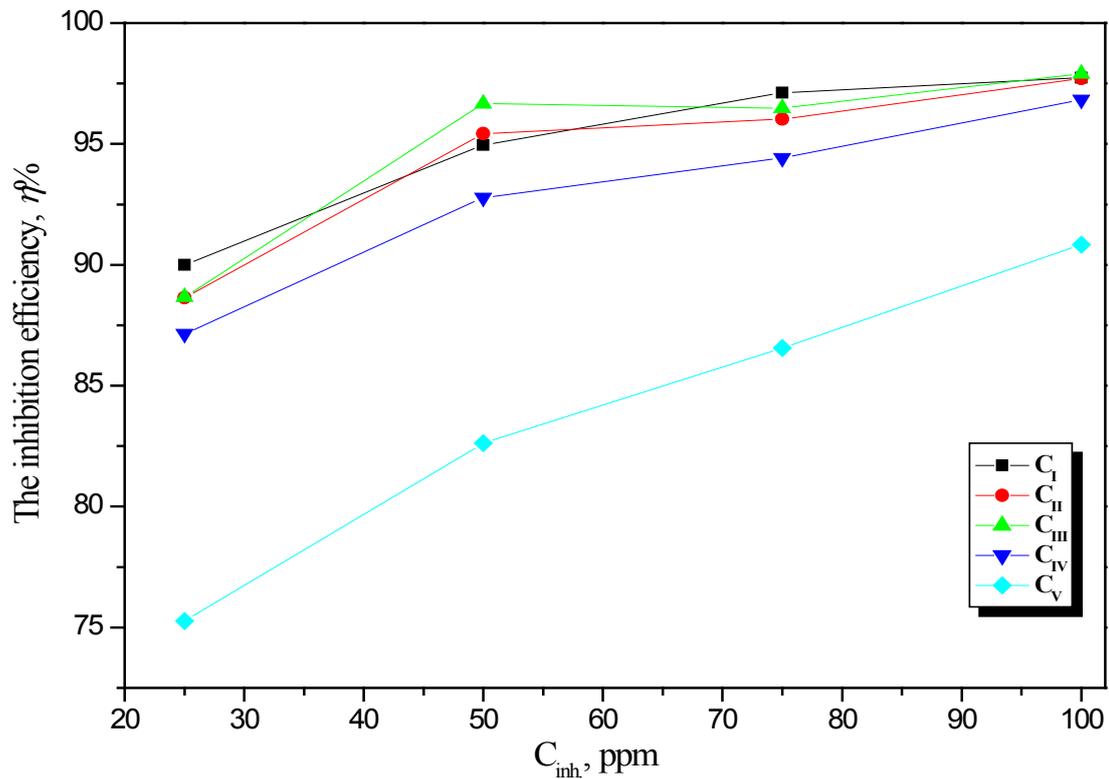
The obtained results of surface active parameters (The effectiveness, maximum surface excess and the minimum surface area per molecule) at 298 K indicate that the prepared compound gives large reduction of surface tension at CMC, so that, the prepared surfactants acts as effective corrosion inhibitors for mild steel in CO<sub>2</sub>-saturated 1% NaCl.



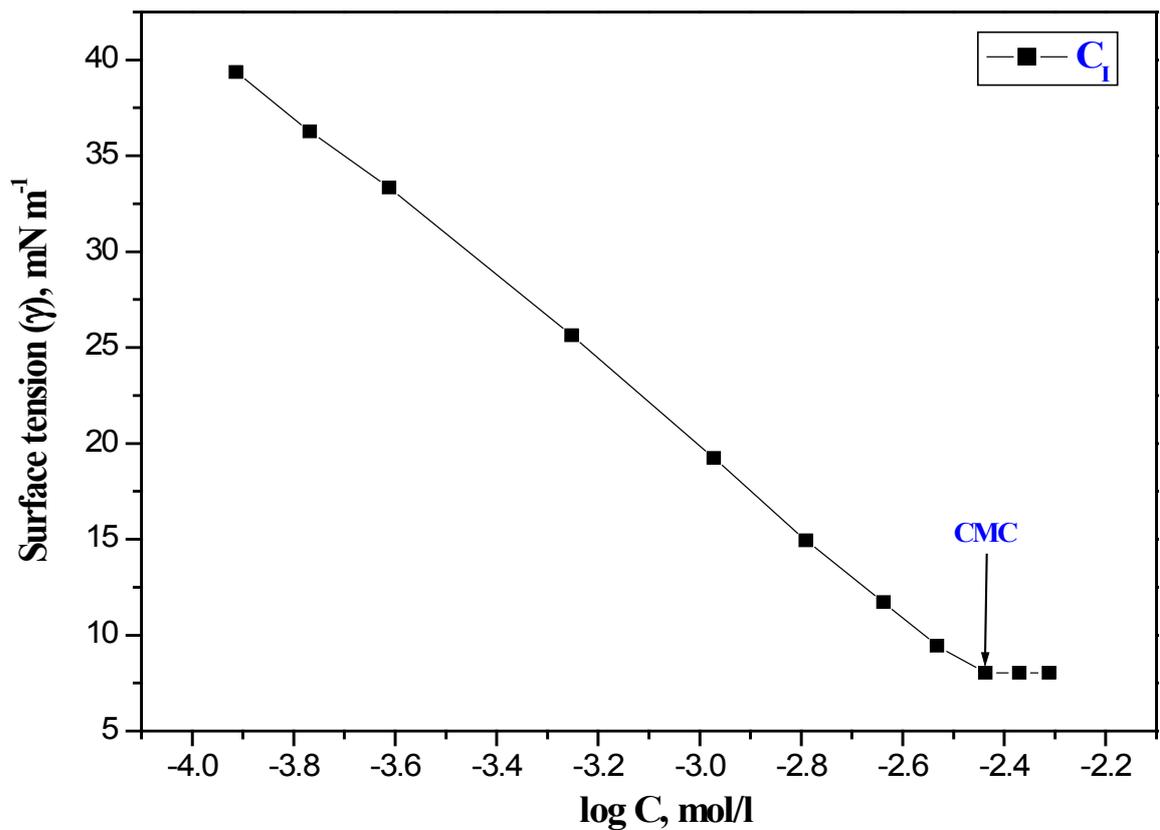
**Figure 3.** Potentiodynamic polarization plots of carbon steel electrode obtained in CO<sub>2</sub>-saturated 1% NaCl solution containing different concentration of inhibitor (C<sub>i</sub>) at 323 K



**Figure 4.** Comparison between (a) corrosion current density,  $I_{corr}$  and (b) corrosion potential,  $E_{corr}$ , of mild steel alloy in  $CO_2$ -saturated solution containing different concentrations of the prepared surfactants at 323



**Figure 5.** Comparison between inhibition efficiency of mild steel alloy in CO<sub>2</sub>-saturated solution containing different concentrations of the prepared surfactants at 323 K



**Figure 6.** Variation of surface tension vs. log C of the inhibitor C<sub>I</sub> at 298 K

### 3.3. Adsorption Isotherms and Thermodynamic Parameters for the Corrosion process

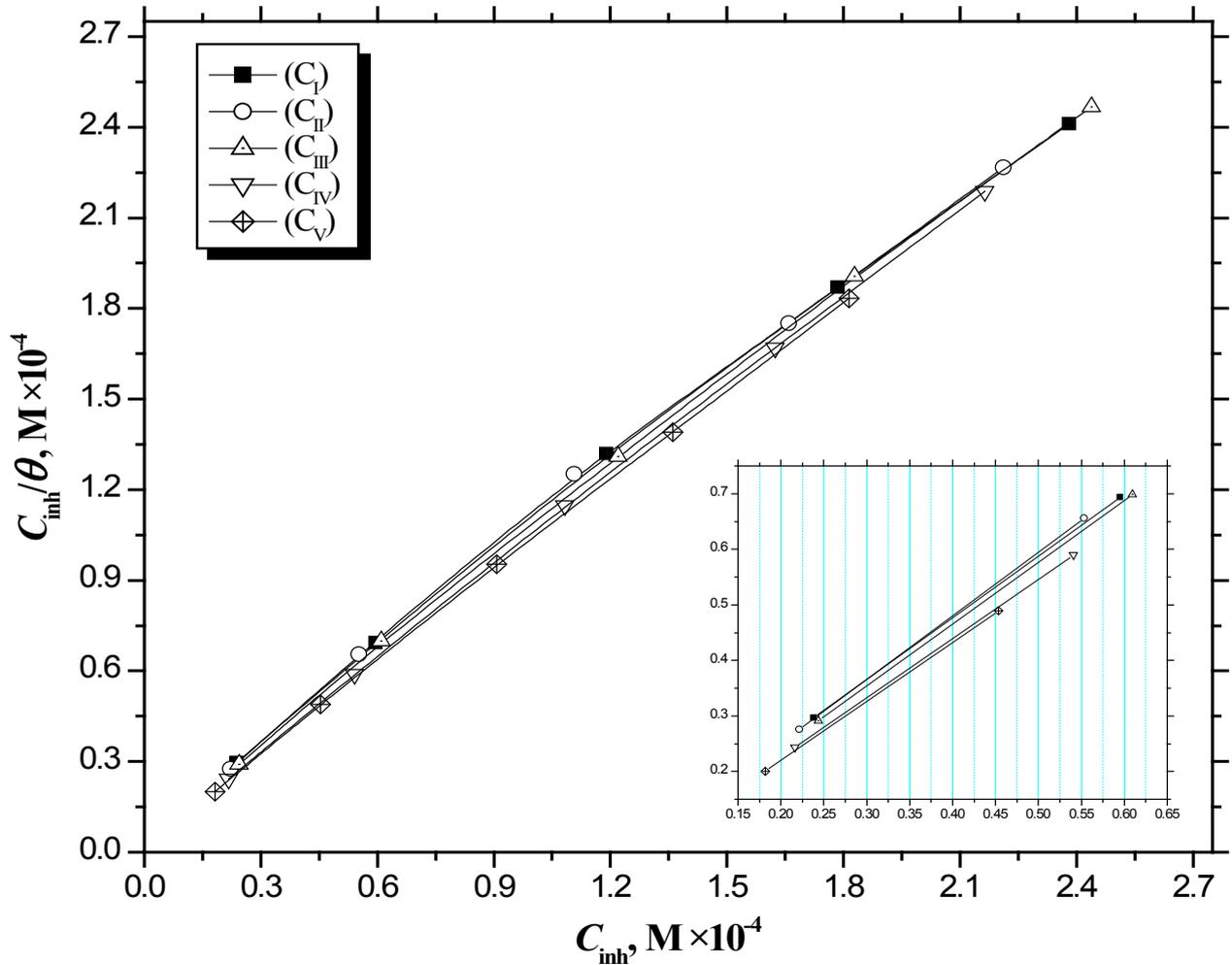
The values of the degree surface coverage  $\theta$  were evaluated at different concentrations of the prepared surfactant compounds in CO<sub>2</sub>-saturated solution at 323 K. The values of  $\theta$  have been used to explain the best isotherm to determine the adsorption process. For obtaining the best description of adsorption behavior of the inhibitor, attempts were made to fit  $\theta$  values to various isotherms including Frumkin, Temkin, Freundlich and Langmuir adsorption isotherms. By far the best fit was obtained with the Langmuir isotherm and can be represented using the following equation [1]:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad (5)$$

where  $C_{inh}$  is the inhibitor concentration,  $K_{ads}$  is the adsorption equilibrium constant and  $\theta$  is the surface coverage. **Figure 7** shows the plots of  $C_{inh} / \theta$  against inhibitor concentrations ( $C_{inh}$ ) at 323 K and the expected linear relationship is obtained for all surfactants with excellent correlation coefficients ( $R^2$ ) (**Table 3**), confirming the validity of this approach. The slopes of the straight lines are unity, suggesting that adsorbed surfactant molecules form monolayer on the mild steel surface and there is no interaction among the adsorbed inhibitor molecules.

The values of  $K_{ads}$  obtained from the Langmuir adsorption isotherm are listed in Tables 3, together with the values of the Gibbs free energy of adsorption ( $\Delta G_{ads}^o$ ) calculated from [16]:

$$K_{ads} = \frac{1}{55.5} \exp\left(-\frac{\Delta G_{ads}^o}{RT}\right) \quad (6)$$



**Figure 7.** Langmuir adsorption isotherm ( $C_i/\theta$  vs.  $C_i$ ) fitting of the obtained potentiodynamic polarization data for mild steel in CO<sub>2</sub> saturated brine containing various concentrations of inhibitors at 323 K

where  $R$  is the universal gas constant,  $T$  is the thermodynamic temperature and the value of 55.5 is the concentration of water in the solution [2]. The high values of  $K_{ads}$  for studied surfactants indicate stronger adsorption on the mild steel surface in CO<sub>2</sub>-saturated solution. It is also noted that, the high values of  $K_{ads}$  for surfactant C<sub>III</sub> indicate stronger adsorption on the mild steel surface than the other surfactants. The value and sign of  $\Delta G_{ads}^o$  provide information about the mechanism of the inhibitor molecules adsorption at the metal surface. The negative values of  $\Delta G_{ads}^o$ , are consistent with the spontaneity of the adsorption process and the stability of the adsorbed layer on the mild steel surface. Generally, values of  $\Delta G_{ads}^o$  up to -20 kJ mol<sup>-1</sup> are consistent with physisorption, while those around -40 kJ mol<sup>-1</sup> 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 [1-3, 16]. In the present study, the values of  $\Delta G_{ads}^o$  obtained for studied surfactants on mild steel in CO<sub>2</sub>-saturated solution more than -40 kJ mol<sup>-1</sup> (**Table 3**). These results indicate that the adsorption mechanism of surfactants on mild steel in CO<sub>2</sub> saturated solution is typical chemisorptions at the studied temperature.

**Table 3.** Thermodynamic parameters for the adsorption of the studied inhibitors on mild steel electrode in CO<sub>2</sub>-saturated 1% NaCl solution

Inhibitors	Slope	Regression coefficients, $R^2$	$K_{ads}$ , $M^{-1} \times 10^4$	$\Delta G_{ads}^o$ (KJ mol <sup>-1</sup> )
C <sub>I</sub>	1.02	0.9976	8.23	-42.14
C <sub>II</sub>	1.01	0.9993	8.64	-43.99
C <sub>III</sub>	1.04	0.9992	11.99	-45.74
C <sub>IV</sub>	1.06	0.9973	11.34	-43.79
C <sub>V</sub>	1.08	0.9992	8.24	-44.62

## 4. Conclusions

1. The synthesized surfactants of fatty acids derivatives are considered as good inhibitors for mild steel corrosion in CO<sub>2</sub>-saturated brine.
2. The inhibition efficiency was found to increase by increasing the inhibitor concentrations. The inhibition efficiencies are 97.73%, to 97.71%, 97.91%, 96.84% and 90.84% for inhibitors C<sub>I</sub>, C<sub>II</sub>, C<sub>III</sub>, C<sub>IV</sub> and C<sub>V</sub> respectively at 100 ppm.
3. The  $E_{corr}$  values of all synthesized surfactants were shifted slightly toward both cathodic and anodic directions and did not show any definite trend in CO<sub>2</sub>-saturated brine. This may be contributed to the mixed-type behavior of the studied inhibitors.
4. The inhibitive action of surfactants compounds is due to the adsorption on the steel surface.
5. The adsorption of cationic surfactants compounds on

the steel surface follows Langmuir adsorption isotherm.

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