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# Ionic Liquids based on Different Chain Fatty Acids as Green Corrosion Inhibitors for C-steel in Produced Oilfield Water

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**Abstract.** The work presents the investigation of fatty acids based ionic liquid as green corrosion inhibitors for C-steel in produced oilfield water. Different-chain length fatty acids C6, C12 and C18-1 are mixed with di-ethanol amine (DEA) to synthesize some ionic liquids and evaluated the corrosion rates on C-steel surface using weight loss method and potential time measurements. The prepared ionic solutions were shown to be effective in reducing the corrosion rates as sustainable corrosion inhibitors. The polar unit is indicated as the reaction center for the adsorption process. Generally, the inhibition efficiency increases with increase the inhibitor concentration.

**Keywords:** Carbon steel, ionic liquids, fatty acids, corrosion inhibitors, adsorption.

## 1. Introduction

Corrosion is an irreversible and spontaneous destruction of metal or alloy through chemical or electrochemical reaction with the environment [1]. Therefore, corrosion has drawn considerable academic and industrial attention [1-3]. The corrosion of carbon steel (C-steel) caused by the presence of produced oilfield water is considered a common problem across petroleum industries [4]. Produced oilfield water (also called formation water) exists in gas and oil reservoirs and it's rich in huge amounts of dissolved salts like chlorides and sulfates besides corrosive dissolved gases like CO<sub>2</sub> and H<sub>2</sub>S.

The research within the sector of green corrosion inhibitors has been addressed toward the goal of using cheap effective molecules at low or zero environmental impact. Green approach towards corrosion inhibition of carbon steel in produced oilfield water using ionic liquids which replaced the inhibitors supported toxic materials produced from petrochemical resources to reduce the pollution of the environment [5,6]. Moreover, the using of ionic liquids has attracted considerable attention because of their interesting properties like low volatility, non-inflammability, non-toxic nature, high thermal/chemical stability and their ability to adsorb on the metallic surface. The massive number of possible cation/ anion combinations allows an excellent variety of tunable interactions; also their unique properties and characteristics make it a promising and a favorable to work within several fields [7], their physicochemical properties, mainly, good electrical conductivity, solvent transport, and a comparatively wide electrochemical window [8]. Introducing different functional groups into cations, which provides an excellent deal of ILs with new structures, can markedly change its physicochemical properties, and it also affords more choices for applications of ILs in electrochemical devices.

The present work is to produce green corrosion inhibitors supported fatty acids ionic liquids such as hexanoic or caproic acid, lauric acid and oleic acid which are prepared by the reaction with diethanol amine. Moreover, the ability of the prepared materials to inhibit the corrosion of steel in oilfield water by applying different concentrations from the prepared inhibitors and tested it with different chemical techniques.



## 2. Experimental

### 2.1. Materials

#### 2.1.1. Chemical composition of carbon steel.

The experiments were performed using carbon steel with chemical composition that given in table 1.

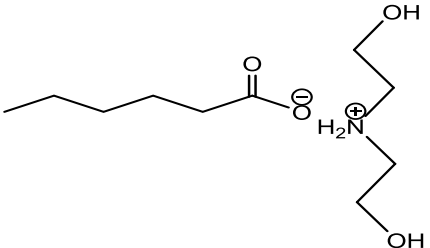
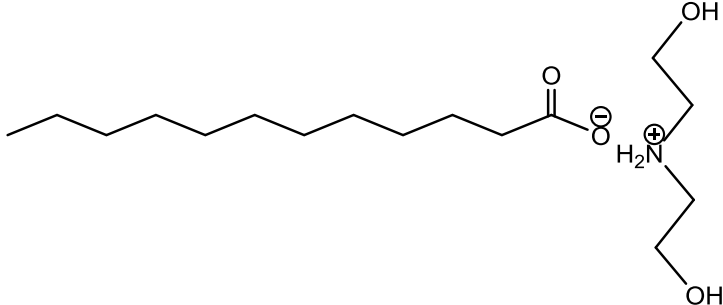
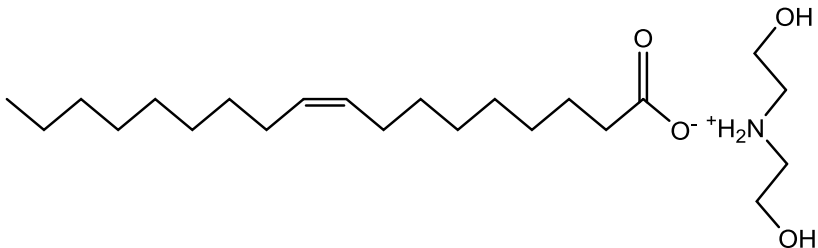
**Table 1.** Chemical composition of carbon steel by wt. %.

Chemical Composition	C	Mn	Si	S	P	Fe
wt. %	0.12	0.5	0.17	0.6	0.046	Rest

#### 2.1.2. Ionic liquids synthesis

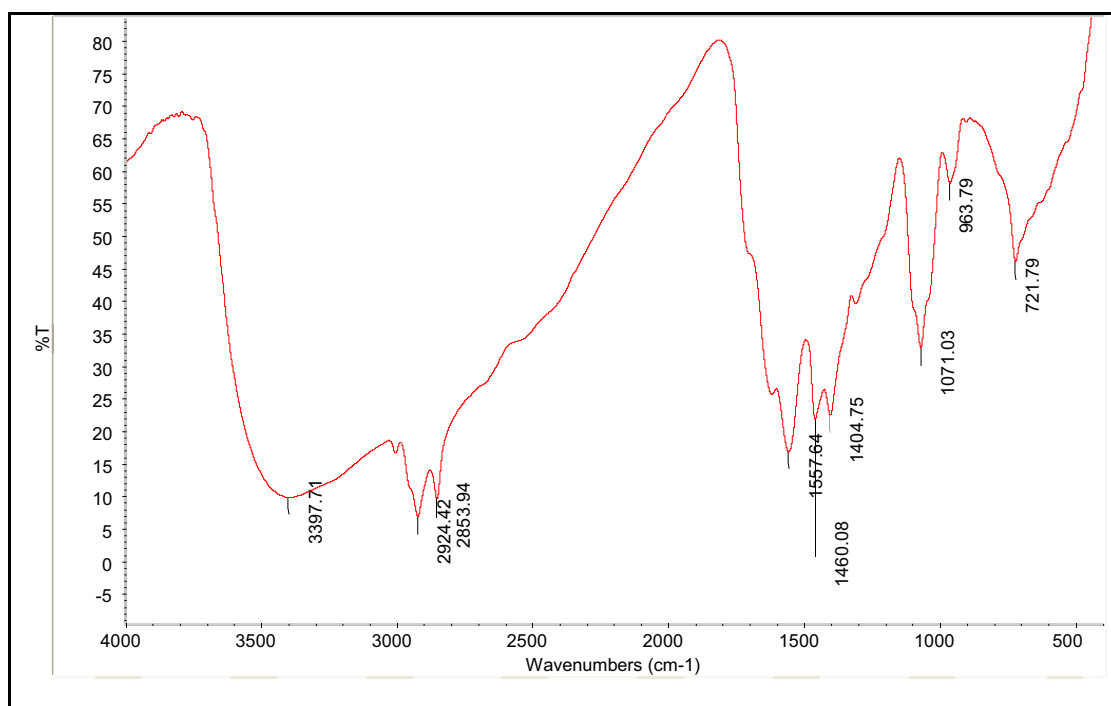
Diethanol amine, DEA (0.1 mole) was loaded into 100 ml two-necked round bottom flask on a magnetic stirrer. The system was equipped with a reflux condenser with vigorous stirring at constant rate under nitrogen. The same molar ratio of various fatty acids C<sub>6</sub>, C<sub>12</sub> and C<sub>18-1</sub> (0.1 mole) was added individually and drop wise to the system for about 15 minutes. The reaction mixture was allowed to stir for twenty-four hours. The product was treated with acetone, removed by evaporation under vacuum and dried for 24 hours at 45 °C. The molecular structures of the prepared ionic liquids as inhibitors are shown in table 2.

**Table 2.** Molecular structure of the prepared ionic liquids.

Chemical Name	Molecular Structure	M.wt
<b>IL-1: DEA-C<sub>6</sub></b> bis(2-hydroxyethyl)ammonium hexanoate		221.2
<b>IL-2: DEA-C<sub>12</sub></b> bis(2-hydroxyethyl)ammonium dodecanoate		305.46
<b>IL-3: DEA-C<sub>18-1</sub></b> bis(2-hydroxyethyl)ammonium oleate		387.6

### 2.1.3. FTIR analysis.

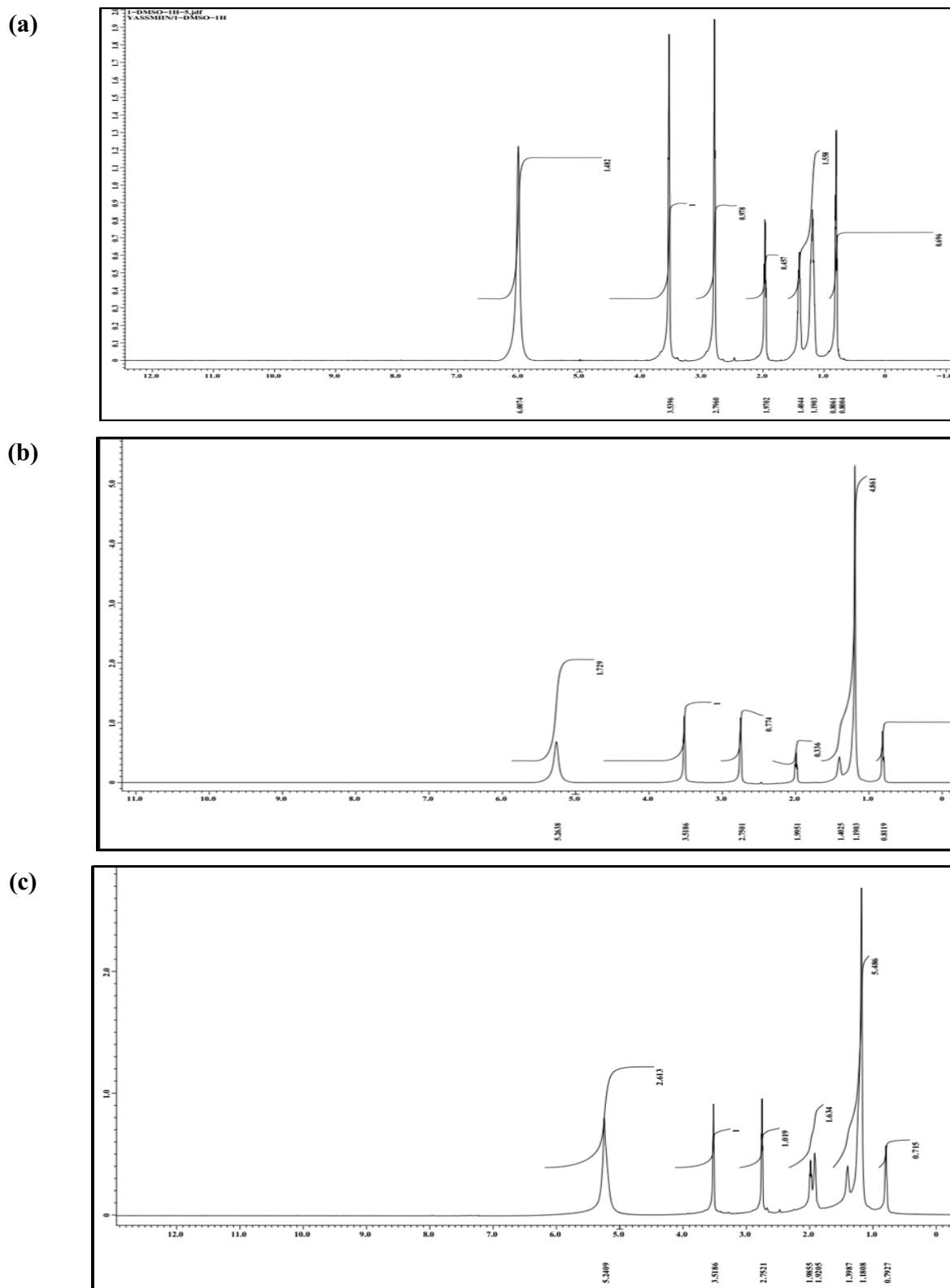
Infrared spectra of the three prepared ILs as represented in figure 1, show that each one of the three ILs exhibits a broad band at the range of 3100–3400  $\text{cm}^{-1}$ , assigned to  $\text{-NH}$  stretching vibration by a secondary amine. The strong vibrational bands in the range of 2800–2950  $\text{cm}^{-1}$  are assigned to C-H symmetric and asymmetric stretching vibrational motion of methylene and methyl groups within the alkyl chains of various ionic liquids. The presence of a sharp peak at 3006  $\text{cm}^{-1}$  in IL-3 of monounsaturated fatty acid (oleic acid) only is attributed to  $\text{=C-H}$  stretching [9]. The characteristic peaks of fatty acid-ionic liquids are ascertained by the presence of the symmetric and asymmetric stretching vibrations of the carboxylate group ( $\text{C=O}$  &  $\text{COO}$ ) for the three ILs at the range of 1552–1558  $\text{cm}^{-1}$ , 1624–1653  $\text{cm}^{-1}$  & 1450–1457  $\text{cm}^{-1}$ , respectively. Thermo Scientific Nicolet iS50 FTIR Spectrometer used in this work and performed at Chemistry Administration, Cairo, Egypt.



**Figure 1.** FTIR spectra of DEA-C<sub>18-1</sub> IL.

### 2.1.4. <sup>1</sup>H-NMR analysis.

The three ionic liquids nearly exhibit an equivalent chemical shifts as shown in figure 2(a,b,c), which, are often summarized as following: the peaks appeared at chemical shifts ( $\delta$ , ppm) of 0.79 – 0.81 are assigned to the terminal methyl group (t, 3H,  $\text{CH}_3$  -); ( $\delta$ , ppm) of 1.18–1.99 are assigned to (m, 2H,  $\text{CH}_2$  -); ( $\delta$ , ppm) of 2.75 – 2.79 are assigned to (P, 2H,  $\text{CH}_2$  -  $\text{CH}_2$  -  $\text{NH}_2^+$ ); ( $\delta$ , ppm) of 3.5 – 3.53 are assigned to (t, 1H,  $\text{CH}_2$  - OH) [10]. The downfield shift from  $\delta$ , ppm adequate to 5.24 just in case of IL3 of oleic acid to 5.3 in IL2 of lauric acid and eventually to 6.0 in the case of IL1 of hexanoic acid are due to the difference of alkyl chain length in each ionic liquid. The data of Nuclear Magnetic Resonance (<sup>1</sup>H NMR spectra) were performed using Joel 500 MHz, at National Research Center (NRC), Cairo, Egypt.



**Figure 2.** <sup>1</sup>H-NMR spectra of (a) DEA-C<sub>6</sub>, (b) DEA-C<sub>12</sub> and (c) DEA-C<sub>18-1</sub> ILs, respectively.

### 2.1.5. Preparation of solutions.

The oilfield formation water used in this work has the chemical analysis listed in table 3. It diluted by 50% with distilled water and used as an aggressive solution for all experiments. The prepared ionic liquids were prepared in molarity, M, by using the same concentration of formation water. All experiments were performed at room temperature, 25 °C, in equilibrium with the atmosphere.

**Table 3.** Chemical composition of oilfield formation water.

Test	Result
Chloride Salinity	19148 ppm
Ca <sup>++</sup>	561 ppm
Mg <sup>++</sup>	146 ppm
pH	7.5

## 2.2. Methods

### 2.2.1. Weight loss measurements.

The dimensions of carbon steel coupons 7.5 x 1.2 x 0.1 cm included two holes of radius 0.4 cm, with total surface area of 17.74 cm<sup>2</sup> of two sides used for weight loss measurements and obtained from the oil field region (the study area). Before all measurements, the exposed area was mechanically abraded with different grades of emery papers (280–1200). The specimens were completely immersed in 100 ml of the test solution from of 1 to 24 hours. The weight loss for each specimen is taken as the difference in weight before and after immersion using digital electronic balance with sensitivity of ±0.1 mg.

### 2.2.2. Potential time measurements.

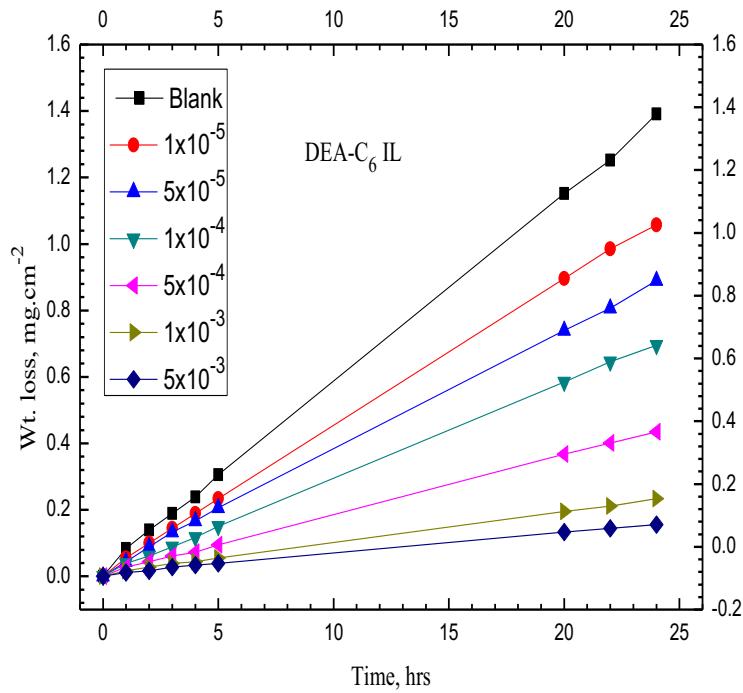
The potential time curves were performed by open circuit and record the potential shifting with and without different concentrations of ionic liquids for 3 hrs. The carbon steel electrode of geometric area 0.12 cm<sup>2</sup> is employed for the measurements and the saturated silver/silver chloride electrode (Ag/AgCl) used as a reference electrode for referring to all potentials.

## 3. Results and Discussion

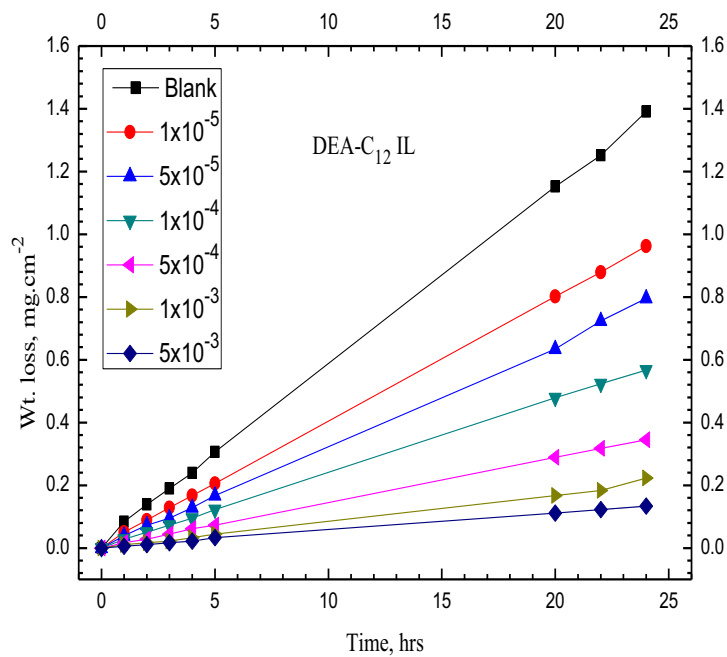
### 3.1. Weight loss measurements

The weight loss of carbon steel in diluted formation water by 50% with and without the addition of various concentrations of inhibitors was compiled in figures 3, 4 & 5. Inspection of those figures reveal that, the linear variation of weight loss with time in uninhibited and inhibited 50% formation water indicates the absence of insoluble surface films during corrosion i.e. the inhibitors are first adsorbed on the metal surface and thus impede corrosion either by merely blocking the reaction sites anodic and cathodic or by altering the mechanism of the anodic and cathodic processes [11].

The carbon steel surface is corroded by the aggressive Cl<sup>-</sup> and S<sup>2-</sup> anions which are existed in oilfield formation water. The corrosion rates calculated from the slope of the fit linear for wt. loss-time curves as shown in equation (1). It's clear that the corrosion rate decreased by adding increased concentrations from DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs to the aggressive solution, as shown in figure 6. The inhibition efficiencies (IE%) and surface coverage (θ) obtained from weight loss calculated from equations (2), (3), respectively. It is obvious that the IE increases with increasing the inhibitor concentration [12] in the following order: DEA-C<sub>18-1</sub> ILs > DEA-C<sub>12</sub> ILs > DEA-C<sub>6</sub> ILs. The inhibition efficiencies values are listed in table 4.



**Figure 3.** Plot of Wt. loss-time curves for carbon steel in different concentrations of DEA-C<sub>6</sub> IL.



**Figure 4.** Plot of Wt. loss-time curves for carbon steel in different concentrations of DEA-C<sub>12</sub> IL.

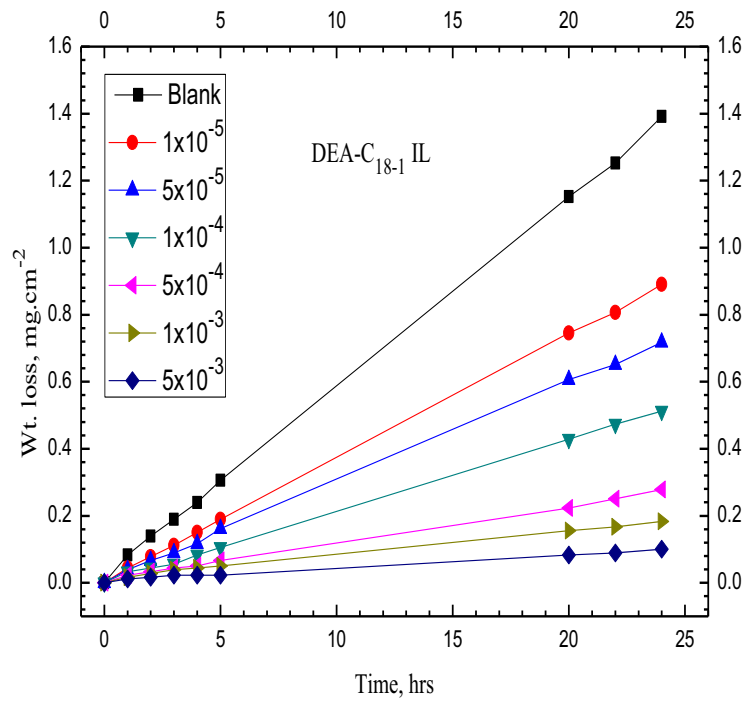


Figure 5. Plot of Wt. loss-time curves for carbon steel in different concentrations of DEA-C<sub>18-1</sub> IL.

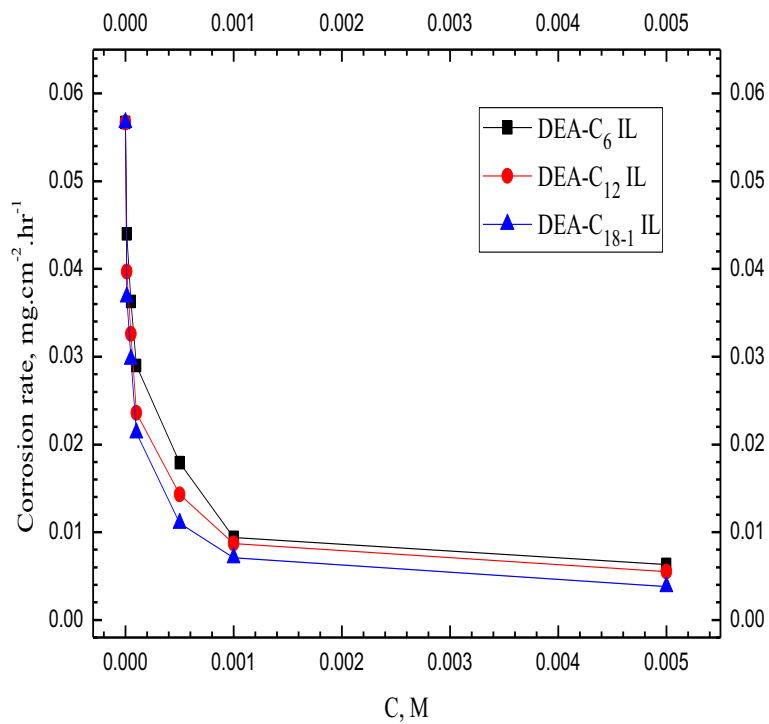


Figure 6. Corrosion rate ( $C_R$ ) for corrosion for carbon steel as a function of concentration of DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs with 50% oilfield formation water.



$$C_R = \frac{W_o - W_i}{W_o} \tag{1}$$

where  $W_o$  and  $W_i$  are the wt. losses in absence and presence of the inhibitor, respectively.

$$\%IE = \frac{CR_o - CR_i}{CR_o} \times 100 \tag{2}$$

$$\theta = \frac{CR_o - CR_i}{CR_o} \tag{3}$$

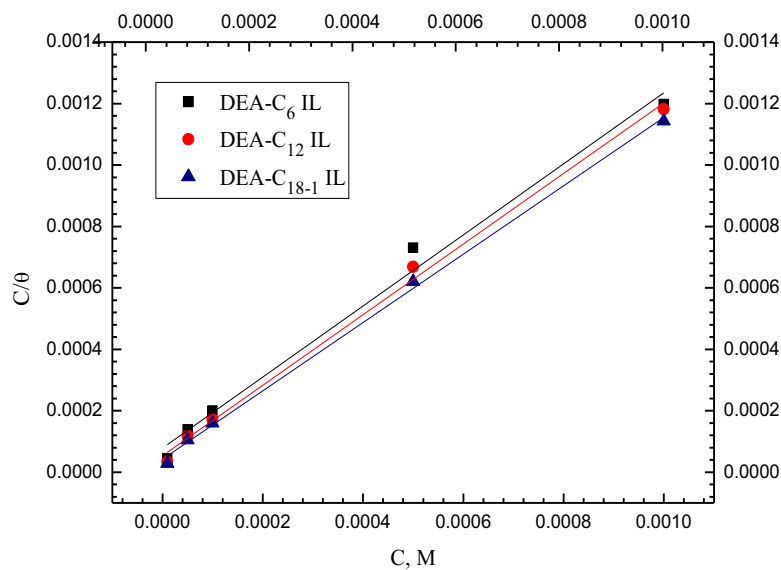
where  $CR_o$  and  $CR_i$  are the corrosion rates in the absence and presence of the inhibitor, respectively.

**Table 4.** Rate of corrosion ( $C_R$ ) and inhibition efficiency (IE) for corrosion of carbon steel in different ionic liquids.

C, M	DEA-C <sub>6</sub> IL		DEA-C <sub>12</sub> IL		DEA-C <sub>18-1</sub> IL	
	$C_R$ , mg.cm <sup>-2</sup> .hr <sup>-1</sup>	IE, %	$C_R$ , mg.cm <sup>-2</sup> .hr <sup>-1</sup>	IE, %	$C_R$ , mg.cm <sup>-2</sup> .hr <sup>-1</sup>	IE, %
Blank	56.7x10 <sup>-3</sup>	-	56.7x10 <sup>-3</sup>	-	56.7x10 <sup>-3</sup>	-
1x10 <sup>-5</sup>	44x10 <sup>-3</sup>	22.39	39.7x10 <sup>-3</sup>	29.98	36.8x10 <sup>-3</sup>	35.097
5x10 <sup>-5</sup>	36.3x10 <sup>-3</sup>		32.6x10 <sup>-3</sup>	42.5	29.7x10 <sup>-3</sup>	47.62
1x10 <sup>-4</sup>	29x10 <sup>-3</sup>	35.97	23.6x10 <sup>-3</sup>	58.38	21.3x10 <sup>-3</sup>	62.43
5x10 <sup>-4</sup>	17.9x10 <sup>-3</sup>	48.85	14.3x10 <sup>-3</sup>	74.78	11x10 <sup>-3</sup>	80.6
1x10 <sup>-3</sup>	9.4x10 <sup>-3</sup>	68.43	8.7x10 <sup>-3</sup>	84.65	7.1x10 <sup>-3</sup>	87.48
5x10 <sup>-3</sup>	6.3x10 <sup>-3</sup>	83.42	5.5x10 <sup>-3</sup>	90.3	3.8x10 <sup>-3</sup>	93.3

3.1.1. Adsorption isotherm.

Since corrosion is a spontaneous process, relative rate of corrosion among a given series of metals is related to the change in standard Gibb’s free energy  $\Delta G^\circ$ . A more negative value of  $\Delta G^\circ$  related with higher spontaneity of reaction [13-16]. It corroded in order to form stable corrosion products [17-19]. The values of the degree surface coverage  $\theta$  were evaluated at different concentrations of the DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs according to equation 4.



**Figure 7.** Langmuir isotherms for carbon steel in DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs.

$$C_{inh}/\theta = 1/K_{ads} + C_{inh} \quad (4)$$

where  $K_{ads}$  is the equilibrium constant of the adsorption/desorption process.

**Table 5.** Langmuir adsorption isotherm of DEA-fatty acids ILs on carbon steel in 50% oilfield formation water.

ILs inhibitors	Langmuir adsorption isotherm			
	$K_{ads}, M$	$\Delta G_{ads}^{\circ}, kJ mol^{-1}$	Slope	$R^2$
DEA-C <sub>6</sub>	$1.27 \times 10^4$	-30.58	1.15	0.987
DEA-C <sub>12</sub>	$1.89 \times 10^4$	-31.47	1.14	0.995
DEA-C <sub>18-1</sub>	$2.38 \times 10^4$	-32	1.11	0.998

Figure 7, represents the relationship between  $C/\theta$  and  $C$ . Straight lines were obtained with intercept of  $K_{ads}^{-1}$  with the slopes of  $\sim 1$ , suggesting that the adsorption of all ILs on the carbon steel surface follow Langmuir isotherm. From table 5, it can be seen the values of  $K_{ads}$  and the related free energy of adsorption  $\Delta G_{ads}^{\circ}$  that evaluated by the following equation:

$$\Delta G_{ads}^{\circ} = -2.303 RT \log (55.5 K) \quad (5)$$

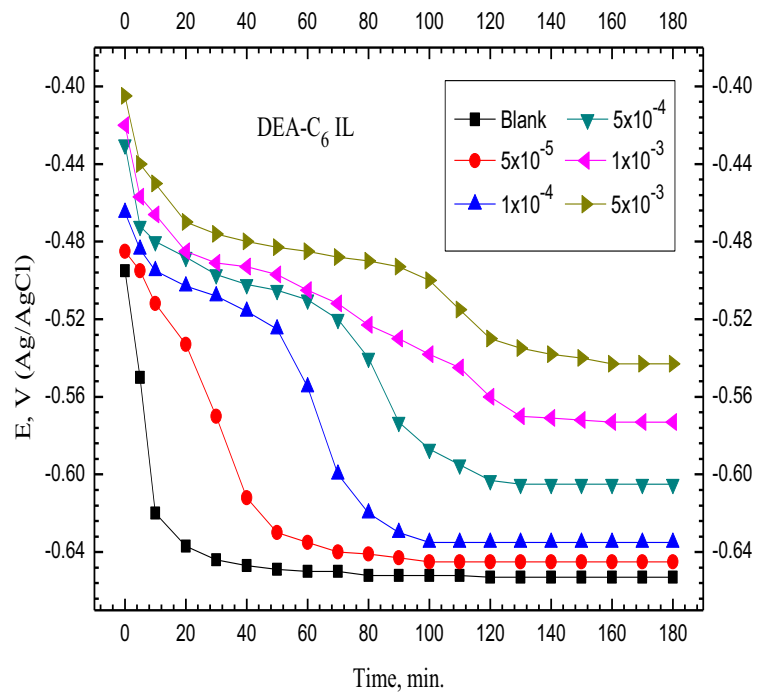
where,  $R$  is the universal gas constant,  $T$  is that the temperature (273 K) and  $K$  is that the equilibrium constant of adsorption. The value of 55.5 is that the concentration of water within the solution in mol/L. The negative values refer to the spontaneity of the adsorption process and therefore the film stability on the C-steel surface. In addition to, the high  $K_{ads}$  values indicate a fast adsorption process on the metal active sites. This phenomenon is due to the fast orientation of the high electronic density zone of the molecule located over the metal ion. The calculated values of  $\Delta G_{ads}^{\circ}$  ranged from  $-30.58$  to  $-32$   $kJ mol^{-1}$ , listed in table 5, suggesting competitive physisorption and chemisorption processes of the active constituents present in DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs on carbon steel surface [20].

### 3.2. Potential-time Curves

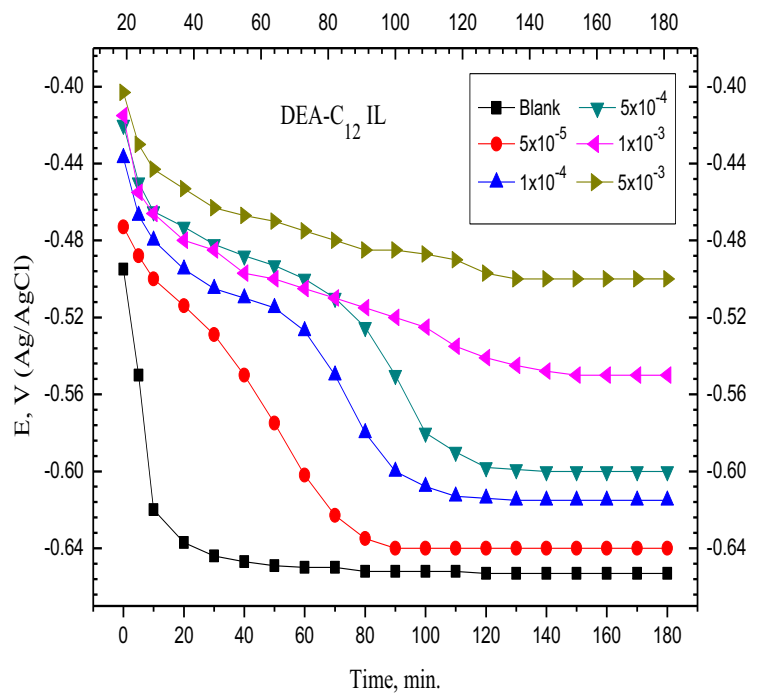
Corrosion studies by open circuit potential in 50% oilfield formation water with and without using DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs as corrosion inhibition of carbon steel shows the simulation curves has the same trend, figures 8, 9 & 10, respectively. At the beginning the potential decreases rapidly in case of aggressive solution up to 10 minutes of exposure and slow gradually by increasing concentrations of inhibitors then the potential remains constant and it results in a marked decrease in potential development. These data have been elucidated that two counter-acting processes happened, the first one being a breaking of the electrode surface adsorbed layer, It can be seen that carbon steel behavior either with and without inhibitor addition, and as a result delayed-action corrosion takes place moving the  $E_{OC}$  to more negative data till constancy. The steady-state potential proceeds towards less negative values with raising ILs concentrations. These potential data clarify that ILs having high efficiency as corrosion inhibitors. The variation of the steady state potential  $E_{st}$  with the logarithm of the molar concentration of ILs is shown in figure 11, whereby  $E_{st}$  varies with  $\log C$  according to the following equation:

$$E_{st} = a_1 + b_1 \log C_{inh} \quad (6)$$

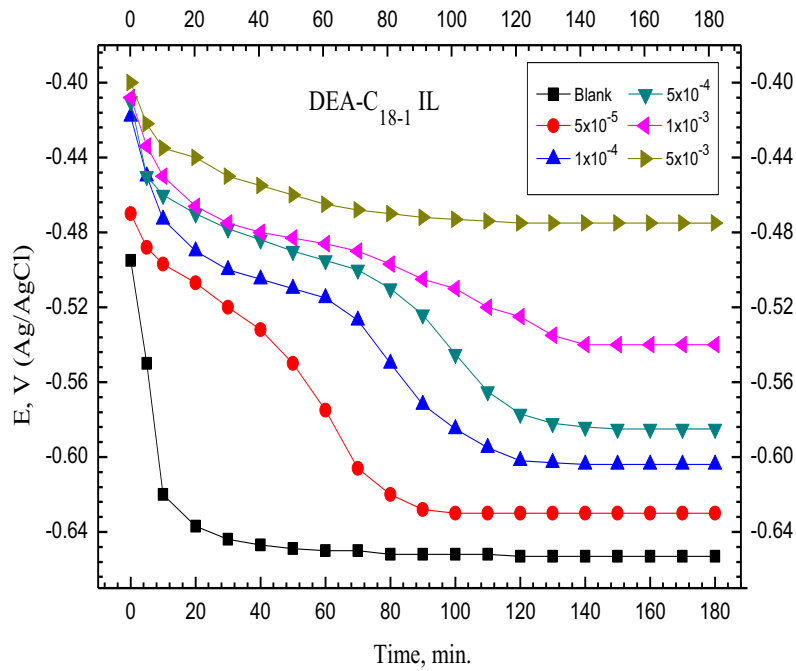
Where,  $a_1$  and  $b_1$  are constants depending on the nature of the metal and type of anions used.



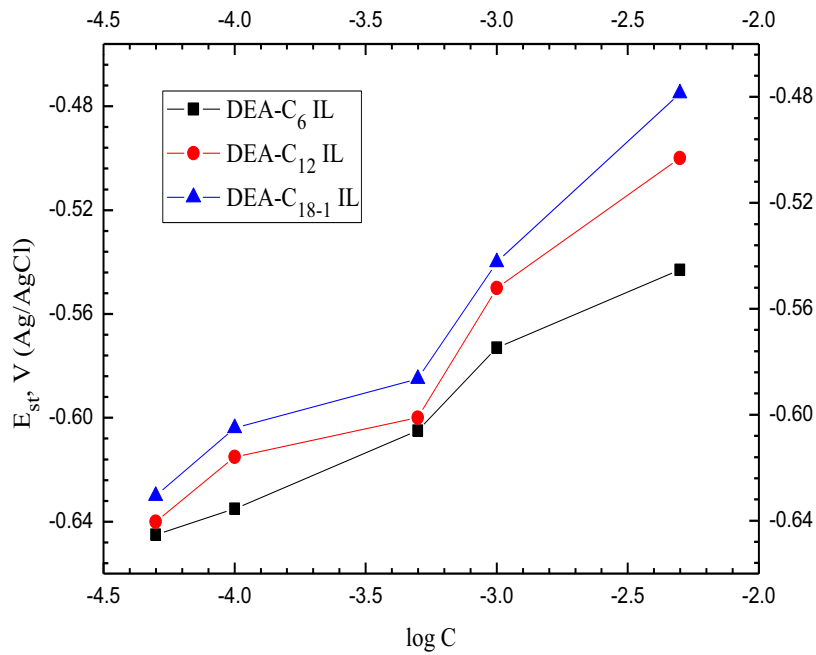
**Figure 8.** Plot of potential-time curves for carbon steel in different concentrations of DEA-C<sub>6</sub> IL.



**Figure 9.** Plot of potential-time curves for carbon steel in different concentrations of DEA-C<sub>12</sub> IL.



**Figure 10.** Plot of potential-time curves for carbon steel in different concentrations of DEA-C<sub>18-1</sub> IL.



**Figure 11.** The steady-state potential,  $E_{st}$ , as a function of  $\log C$  for carbon steel in DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs.

From the equation (6), it can be described a general phenomenon, regardless of whether the anodic reaction results in film destruction or film repair which was derived theoretically [21] on the basis of the specific adsorption of the anions on the surface of the metal or the metal oxide. The rate of oxide film destruction for carbon steel in absence and presence of DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs was concluded from the linear relationship between the open circuit potential of the C-steel and the logarithm of immersion time,  $t$ , for DEA-C<sub>12</sub> IL as in figure 12, and the relation:

$$E = a_2 - b_2 \log t \quad (7)$$

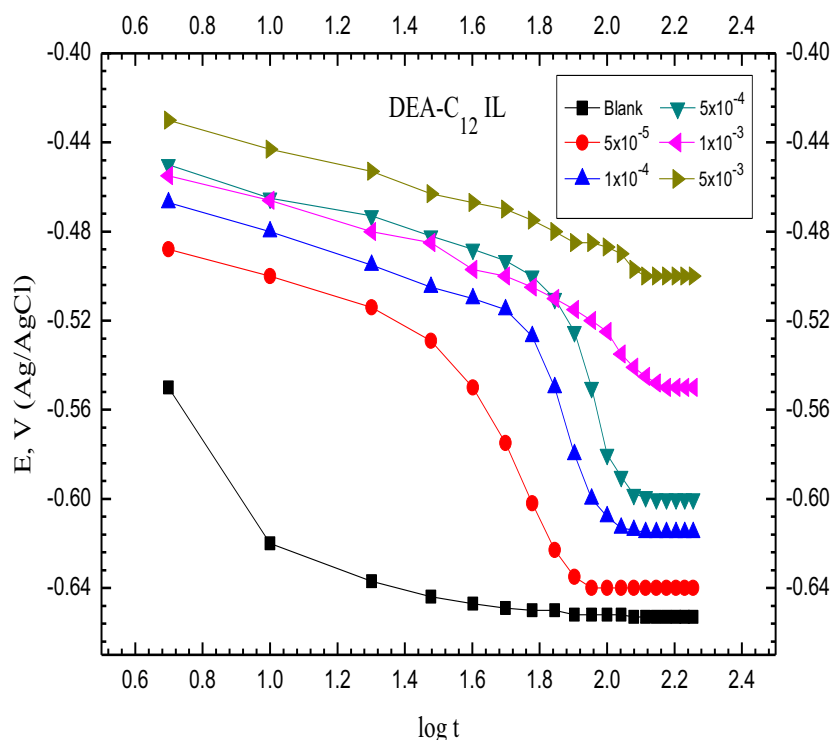
Where  $a_2$ , and  $b_2$  are constants for oxide film destruction. It has been suggested that the driving force for surface oxide film destruction or formation on the metal is the free energy change of the reaction between the metal and the test solution. This process is assumed to proceed via solid state mechanism by the influence of a sufficiently high electric field to cause ionic migration and hence sustain self-diffusion of metal ions through semiconducting oxide films [22]. Based on this notion a relationship between  $E$  and  $\log t$  has been theorized [23] as given by the following equation:

$$E = \text{const.} + 2.303 \delta / \beta \log t \quad (8)$$

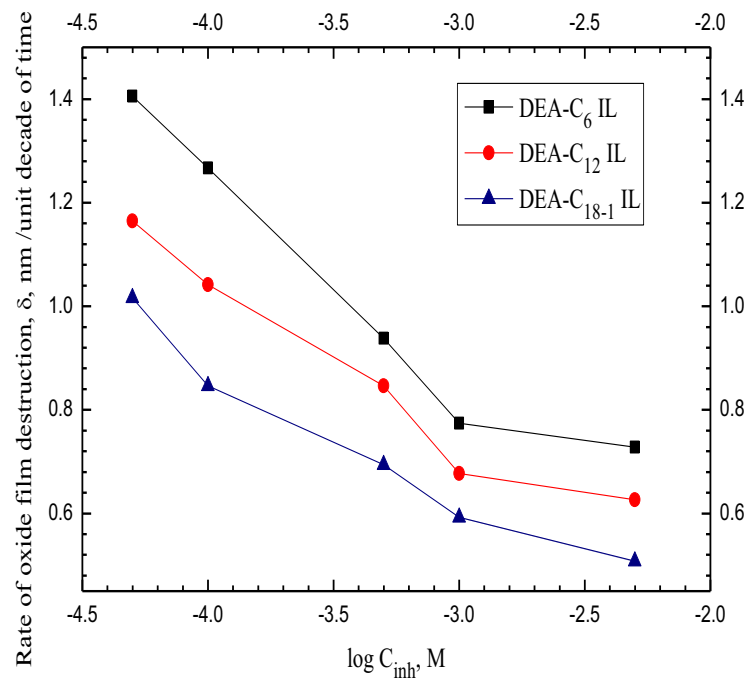
Where  $\delta$  represents the rate of oxide film thickening or destruction per unit decade of time, and  $\beta$  is a constant which is identified as [23, 24]:

$$\beta = (nF/RT) \alpha \delta \quad (9)$$

Where  $\alpha$  is the transfer coefficient similar to that encountered in normal electrochemical reactions [23, 24], ( $0 < \alpha < 1$ ) and  $\delta$  is the width of the energy barrier surmounted by the ion during transfer.



**Figure 12.** E-log  $t$  plots for carbon steel for different concentrations of DEA-C<sub>12</sub> IL.



**Figure 13.** Rate of oxide film destruction,  $\delta$ , as a function of  $\log C$  for carbon steel in DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs with 50% oilfield formation water.

**Table 6.** Rate of O-destruction,  $\delta$  (nm/unit decade of time) and the inhibition efficiency, IE (%) for potential time curves.

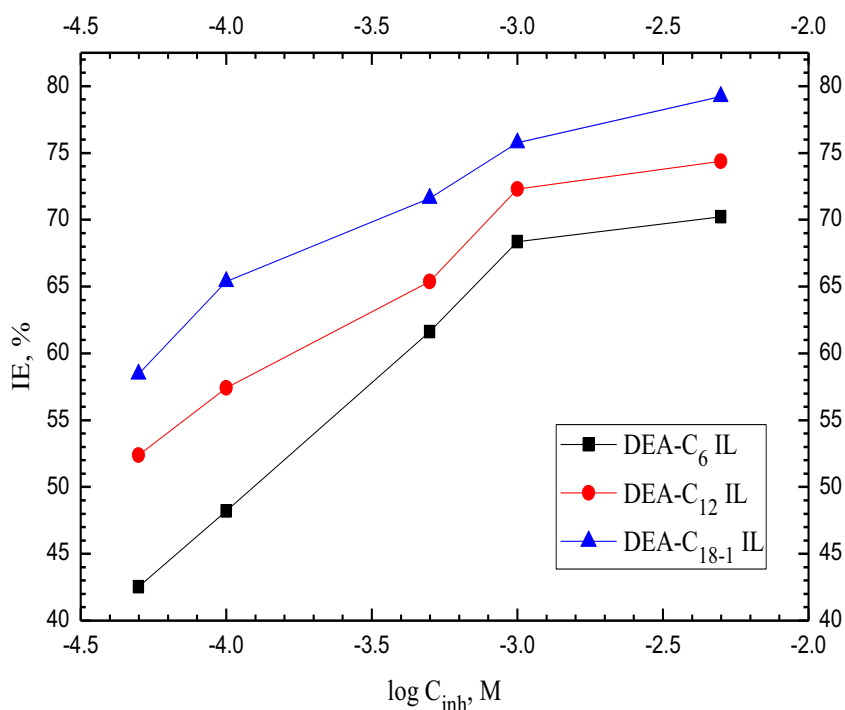
C, M	DEA-C <sub>6</sub> IL		DEA-C <sub>12</sub> IL		DEA-C <sub>18-1</sub> IL	
	$\delta$	IE	$\delta$	IE	$\delta$	IE
Blank	2.44	-	2.44	-	2.44	-
$5 \times 10^{-5}$	1.41	42.52	1.16	52.38	1.01	58.45
$1 \times 10^{-4}$	1.26	48.2	1.04	57.41	0.84	65.37
$5 \times 10^{-4}$	0.94	61.63	0.84	65.37	0.69	71.61
$1 \times 10^{-3}$	0.77	68.35	0.67	72.3	0.59	75.76
$5 \times 10^{-3}$	0.73	70.22	0.62	74.38	0.5	79.22

From the values of the slopes of the straight lines of figure 12, as in DEA-C12 IL, relating the variation of E with  $\log t$ , one readily calculate the values of the rate of oxide destruction,  $\delta$  in nm/unit decade of time to different concentrations of DEA-C6, DEA-C12 and DEA-C18-1 ILs as shown in figure 13. It is found that the rate of O-destruction decreases with increasing the concentration of all ionic liquids inhibitors, as demonstrated in table 6. Also, the inhibition efficiency is calculated from the equation:

$$IE = [1 - (\delta_{inh}/\delta_o)] \times 100 \quad (10)$$

where  $\delta_o$  and  $\delta_{inh}$  are the rates of oxide film destruction of the passive oxide film on the carbon steel surface in absence and presence of inhibitor, respectively. The values of the percentage inhibition

efficiency, IE, are computed as a function of ionic liquids concentrations and shown in figure 14. It increases of the order DEA-C18-1 ILs > DEA-C12 ILs > DEA-C6 ILs.



**Figure 14.** Inhibition efficiency, IE, for carbon steel in DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ILs with 50% oilfield formation water.

### 3.3. Mechanism of Corrosion Inhibition

Literature survey reveals that several synthetic ionic liquids have been used as effective corrosion inhibitors for mild and carbon steel in various electrolytic media [20,25]. Two generic species DEA molecules in combination with fatty acids have been tested according to this methodology as ionic liquids. The ionic liquid under taken in the study inhibits metallic corrosion by adsorbing on the surface as resulted previously which the mechanism obeyed Langmuir adsorption isotherm [20]. This suggests that the metal surface exposed to the aggressive formation water is positively charged. The positively charged surface can attract  $Cl^-$  and  $S^{2-}$  ions, which in turn will attract DEA molecules species through electrostatic interactions. This causes the adsorption of a DEA species on the metal surface, which prevents carbon steel to ionized from entering the aggressive formation water. The fatty acid ionic liquids as lubricants interact with steel surface under the stress and prepare the thin film of low shear-strength, which reduces the friction and protect the surface against the undesirable wear [9]. The syntheses of DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> ionic liquids are confirmed by FTIR and NMR analyses. The viscosity of these ionic liquids increases with increasing the chain length of fatty acid anions and was attributed to increasing van der Waals interaction between the associated methylene units. The introduction of unsaturation sites in fatty acid anions significantly diminished the van der Waals interaction owing to steric constraint and reduced the viscosity. The inherent polar nature of fatty acid anions facilitates their interaction promptly with steel surface under the boundary lubrication and forms the stable chemical thin film of ionic liquids which reduces both the friction and the wear. Further, significantly low cost, environmentally-friendly nature, renewable and sustainable resources of fatty acid precursors promise immense potential of these ionic liquids for various applications.

#### 4. Conclusions

The investigated ionic liquids of DEA-C<sub>6</sub>, DEA-C<sub>12</sub> and DEA-C<sub>18-1</sub> showed excellent alternative corrosion inhibition with eco-friendly properties for the protection of carbon steel in 50% oilfield formation water, which was confirmed by wt. loss and potential time curves.

The inhibition mechanism was attributed to the strong adsorption ability of this ionic liquid to form a protective layer on the metal surface, reducing the active sites and the steel dissolution at the anodic sites by the aggressive environment.

Prepared ionic liquids gave a highly inhibition efficiency at rather low concentrations (i.e.  $5 \times 10^{-5}$ ).

This was further confirmed by several parameters which calculated in this study as  $K_{ads}$ ,  $\Delta G_{ads}$  and  $\delta$  values.

The performance of corrosion inhibition of two techniques is in agreement with each other ranged of the order: DEA-C<sub>18-1</sub> ILs > DEA-C<sub>12</sub> ILs > DEA-C<sub>6</sub> ILs.

#### 5. References

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