

N,N-DI(POLYOXYETHYLENE)–4–DODECYLANILINE AS A CORROSION INHIBITOR FOR STEEL IN HYDROCHLORIC ACID SOLUTIONS

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We have used weight loss measurements, potentiodynamic polarization, and lectrochemical impedance spectroscopy to study the inhibiting effect of N,N-di(polyoxyethylene)–4–dodecylaniline, with different numbers of oxyethylene units, on corrosion of steel in a 1 M hydrochloric acid solution. We have determined that the studied compounds are good corrosion inhibitors. The inhibition efficiency increases with an increase in inhibitor concentration, an increase in the number of oxyethylene units in the chain of the inhibitor, and a decrease in temperature. The inhibiting effect of the studied compounds is due to blocking of the metal surface as a result of adsorption of the oxyethylene units of the inhibitor. The adsorption process is described by the Freundlich isotherm. The indicated compounds act as mixed (mostly anodic) corrosion inhibitors. We calculate some thermodynamic corrosion parameters.

Key words: corrosion inhibitor, N,N-di(polyoxyethylene)–4–dodecylaniline, iron, adsorption.

Iron and iron alloys are exposed to acids in industrial applications. Acids are used in chemical and electrochemical etching, surface cleaning by acid, including cleaning of oil refinery equipment, oil well acidizing and acid descaling [1]. Inhibitors are used to prevent corrosion of iron or steel or to reduce the corrosion rate in acid media [2].

Corrosion inhibitors have been developed based on organic compounds containing nitrogen, oxygen, sulfur, and multiple bonds, which facilitate adsorption on the surface of the metal [3]. Some organic compounds [4–10] display good corrosion inhibiting properties in acid media. These compounds protect the metal by being adsorbed on its surface. Adsorption is affected by the nature of the metal surface and the charge on it, the type of electrolyte, and the chemical structure of the inhibitor [11].

A very effective method for reducing the corrosion rate has been to add high molecular weight organic compounds, acting as surfactants, to the acid media. These compounds are effective inhibitors because their molecules contain long hydrocarbon chains, ensuring high coverage of the metal surface, and also multiple active

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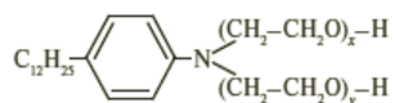
Table 1

Inhibitor concentration, M	IE, %	θ
Compound I		
$1 \cdot 10^{-5}$	71.22	0.712
$5 \cdot 10^{-5}$	78.65	0.786
$1 \cdot 10^{-4}$	85.22	0.852
$5 \cdot 10^{-4}$	89.28	0.893
$1 \cdot 10^{-3}$	91.33	0.913
$5 \cdot 10^{-3}$	94.12	0.941
Compound II		
$1 \cdot 10^{-5}$	74.32	0.743
$5 \cdot 10^{-5}$	79.98	0.800
$1 \cdot 10^{-4}$	87.64	0.876
$5 \cdot 10^{-4}$	90.83	0.908
$1 \cdot 10^{-3}$	93.14	0.931
$5 \cdot 10^{-3}$	95.66	0.957
Compound III		
$1 \cdot 10^{-5}$	76.27	0.763
$5 \cdot 10^{-5}$	81.24	0.812
$1 \cdot 10^{-4}$	88.93	0.889
$5 \cdot 10^{-4}$	92.26	0.923
$1 \cdot 10^{-3}$	94.66	0.947
$5 \cdot 10^{-3}$	96.84	0.968

centers responsible for adsorption. It has been shown [12–16] that ionic and non-ionic surfactants are good corrosion inhibitors for iron and steel in acid media.

The aim of this work was to use weight loss measurements, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS) to study the inhibiting effect of N,N-di(polyoxyethylene)-4-dodecylaniline, with different numbers of oxyethylene units, on corrosion of iron electrodes in a 1 M hydrochloric acid solution.

The hydrochloric acid solution was prepared using 37% hydrochloric acid (analytical reagent grade) and twice-distilled water. A steel electrode of the following composition (%) was used: carbon, 0.052; manganese, 0.189; sulfur, 0.011; phosphorus, 0.008; silicon, 0.011; aluminum, 0.039; nitrogen, 0.0001; chromium, 0.0128; copper, 0.04; molybdenum, 0.024; nickel, 0.0293; the remainder was iron. The non-ionic surfactant N,N-di(polyoxyethylene)-4-dodecylaniline was synthesized from aniline and ethylene oxide (EO) in the presence of potassium hydroxide as the catalyst [17]. The general structural formula for the inhibitor is:



where $n = x + y$; $n = 4, 6$, and 8 for the studied compounds *I*, *II*, and *III*.

Weight loss measurement. $2 \times 2 \times 0.2$ cm electrodes were mechanically polished successively with different grades of emery paper (up to grade 1200), degreased in acetone, rinsed with twice-distilled water, and dried between filter paper and weighed. The specimens were then immersed in 100 mL of a 1 M hydrochloric acid solution with different inhibitor concentrations. After 6 h, the electrodes were removed from the solutions, rinsed with twice-distilled water, dried between filter paper, and weighed again. From the weight loss, we calculated the inhibition efficiency *IE* (in %) of the inhibitor and the coverage θ of the electrode surface by the inhibitor:

$$IE = \frac{W_{free} - W_{adid}}{W_{free}} \cdot 100$$

$$\theta = \frac{W_{free} - W_{adid}}{W_{free}}$$

where W_{free} and W_{adid} represent the weight loss (mg/cm^2) of the electrode in the absence of and in the presence of the inhibitor.

Potentiodynamic polarization measurement. We used a three-electrode electrochemical cell. The working electrode was made from 1×1 cm steel sheet. The electrodes were welded on one side to the copper wire used for connection to the power supply. The electrodes were embedded in a glass tube, held in place by epoxy resin. The reference and auxiliary electrodes were respectively the saturated calomel electrode (SCE) and platinum foil. The working electrode was immersed in 100 mL of a 1 M hydrochloric acid solution. The cathodic and anodic polarization curves were recorded using a GAMRY potentiostat/galvanostat at a scan rate of 2 mV/s. The experiments were run at a temperature of $30^\circ\text{C} \pm 1^\circ\text{C}$, maintained using a thermostat. The percentage inhibition efficiency was calculated from the equation:

$$IE = \left[1 - \frac{i_{corr.adid}}{i_{corr.free}} \right] \cdot 100$$

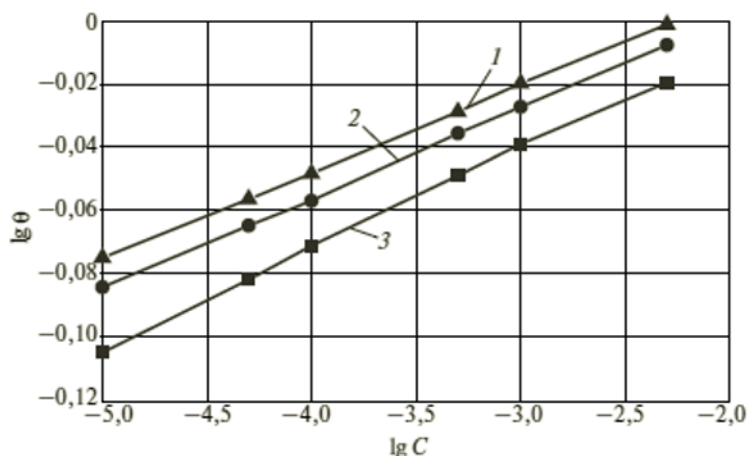


Fig. 1 Dependence of $\log \theta$ on $\log C$: 1) *I*; 2) *II*; 3) *III*.

where $i_{\text{corr,add}}$, $i_{\text{corr,free}}$ are the corrosion current densities respectively in the presence of and in the absence of the inhibitor.

Electrochemical impedance spectroscopy (EIS) was run using a Volta Lab (model PGZ-301) potentiostat with Voltammeter software. The measurements were made with an a.c. signal at open circuit potential 4 mV in the 100 kHz to 50 MHz frequency range.

Table 1 shows the inhibition efficiencies of compounds *I–III* and the surface coverage calculated from the weight loss measurements for steel electrodes in a 1 M hydrochloric acid solution. In all cases, as the inhibitor concentration increased, the weight loss decreased while the inhibition efficiency increased. This behavior can be explained by the increase in surface area occupied by the N,N-di(polyoxyethylene)-4-dodecylaniline as its concentration increases. It is generally assumed that the first step in the inhibition mechanism is adsorption of the inhibitor at the metal – solution interface. When we examine the results, we see that for the same inhibitor concentration, the inhibition efficiency decreases in the series:

Table 2

Temperature, °C	R_{cor} , mg/(cm ² ·min)	IE, %
<i>No inhibitor</i>		
30	0.620	–
40	0.841	–
50	1.050	–
60	1.283	–
70	1.526	–
<i>Compound I</i>		
30	0.054	91.29
40	0.099	88.23
50	0.176	83.23
60	0.253	80.28
70	0.349	77.13
<i>Compound II</i>		
30	0.043	93.06
40	0.082	90.25
50	0.149	85.81
60	0.225	82.46
70	0.326	78.69
<i>Compound III</i>		
30	0.033	94.67
40	0.070	91.68
50	0.125	88.09
60	0.196	84.72
70	0.303	80.14

$$III > II > I.$$

Basic information about the interaction between the inhibitor molecules and the metal surface can be obtained from the adsorption isotherm. We attempted to describe the θ values using different adsorption isotherms. The best results were obtained using the Freundlich isotherm:

$$\log \theta = \log K + n \log C,$$

where K is the equilibrium constant for the inhibitor adsorption process; C is the inhibitor concentration, M; n is a constant.

Fig. 1 graphically shows the $\log \theta$ vs. $\log C$ relationship for compounds *I–III*. The linear correlation indicates that adsorption of N,N-di(polyoxyethylene)-4-dodecylaniline on the steel surface is described by the Freundlich isotherm. The calculated values for the adsorption equilibrium constant for compounds *I*, *II*, and *III* are respectively 0.842, 0.822, and 0.776. The Gibbs free energy for adsorption, ΔG_{ads}^0 , was calculated from the equation:

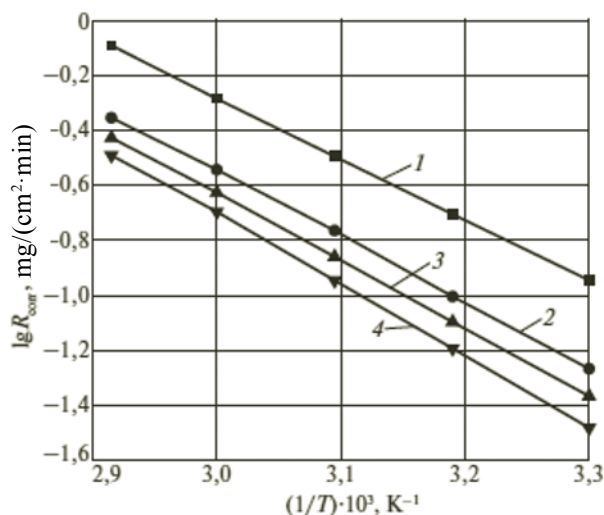


Fig. 2 Dependence of $\log R_{\text{corr}}$ on reciprocal temperature for the process of dissolution of steel in the absence of (*I*) and in the presence of inhibitor: 2) *I*; 3) *II*; 4) *III*.

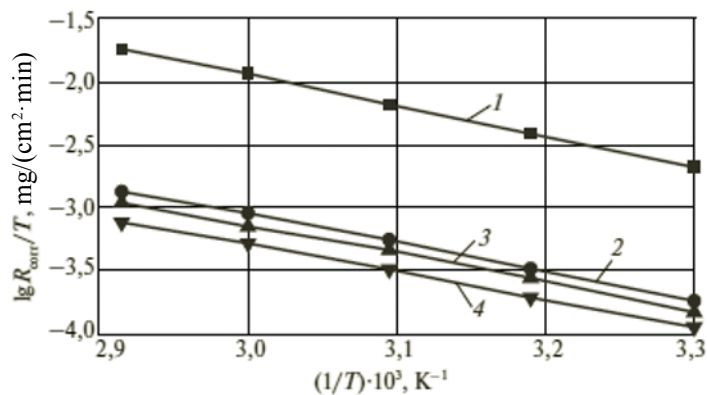


Fig. 3 Dependence of $\log(R_{\text{corr}}/T)$ on reciprocal temperature for the process of dissolution of steel in the absence of (*I*) and in the presence of inhibitor: 2) *I*; 3) *II*; 4) *III*.

$$K = (1/55.5) \exp(-\Delta G_{ads}^0 / RT)$$

where R is the universal gas constant and T is the absolute temperature.

The values of ΔG_{ads}^0 for compounds *I*, *II*, and *III* are respectively -9.52 , -9.46 , and -9.32 kJ/mol. The change in the adsorption free energy is associated with the water adsorption/desorption equilibrium, which makes a major contribution to the overall free energy of adsorption. The negative values of ΔG_{ads}^0 suggest that adsorption of the studied compounds on the steel surface is spontaneous.

The effect of temperature on the corrosion rate R_{corr} for steel and the inhibition efficiency of compounds *I–III* in a 1 M hydrochloric acid solution containing the studied compounds in a concentration of $1 \cdot 10^{-3}$ M were studied by measuring the weight loss in the temperature range $30^\circ\text{C}–70^\circ\text{C}$ (Table 2). We see that as the temperature rises, in the presence of all the inhibitors the weight loss and corrosion rate increase while the inhibition efficiency decreases. The corrosion rate was calculated from the Arrhenius equation:

$$R_{corr} = A \exp\left(-\frac{E_a}{RT}\right)$$

where E_a is the activation energy; A is the pre-exponential factor (or frequency factor).

Fig. 2 shows $\log R_{corr}$ vs. $1/T$ for a 1 M hydrochloric acid solution with no inhibitor and for the same solution containing the studied compounds in a concentration of $1 \cdot 10^{-3}$ M. The activation energy, calculated from the slope of the straight lines, is 32.46 kJ/mol for the solution with no inhibitor and respectively 37.29, 40.88, and 44.36 kJ/mol for solutions containing *I*, *II*, and *III*. As the temperature rises, we see an appreciable decrease in the inhibitor adsorption process and a corresponding increase in the corrosion reaction rate, since a larger surface of the metal is exposed to the acid [18].

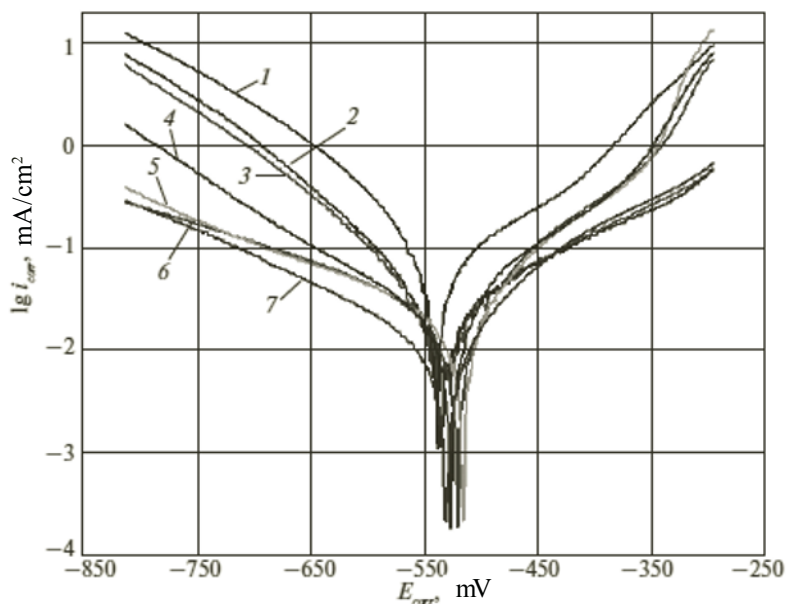


Fig. 4 Potentiodynamic polarization curves for an steel electrode in the absence of (*I*) and in the presence of inhibitor *III* in a concentration of (M) 2) $1 \cdot 10^{-5}$; 3) $5 \cdot 10^{-5}$; 4) $1 \cdot 10^{-4}$; 5) $5 \cdot 10^{-4}$; 6) $1 \cdot 10^{-3}$; 7) $5 \cdot 10^{-3}$.

The entropy of activation ΔS^0 and the enthalpy of activation ΔH^0 for corrosion of steel in a 1 M hydrochloric acid solution, in the presence of the studied compounds in a concentration of $1 \cdot 10^{-3}$ M, were calculated from the equation for the transition state [18]:

$$R_{corr} = (RT/Nh) \times \exp(\Delta G^0 / R) \cdot \exp(\Delta H^0 / RT)$$

where N is Avogadro's number; h is Planck's constant.

A plot of $\log(R_{corr}/T)$ vs. $1/T$ (Fig. 3) is a straight line with slope $\Delta H^0/2.303R$ and y -intercept $\log[(R/Nh) - (\Delta S^0/2.303R)]$. The ΔH^0 values, calculated from the slope of the straight lines, are 25.97 kJ/mol for the solution with no inhibitor and respectively 30.27, 34.88, and 36.25 kJ/mol for the same solution containing compounds *I*, *II*, and *III*. The different values of ΔH^0 for the studied compounds suggests that their structure affects the strength of their adsorption on the surface of the metal.

Table 3

Inhibitor concentration, M	Tafel constants, mV/10		E_{corr} , mV (standard calomel electrode)	i_{corr} , mA/cm ²	IE. %
	b_a	b_c			
<i>No inhibitor</i>					
	142	−155	−526	0.235	—
<i>Compound I</i>					
110^{-5}	225	−133	−530	0.064	72,16
510^{-5}	216	−125	−526	0.018	79,57
110^{-4}	202	−115	−523	0.037	84,25
$5-10^{-4}$	180	−122	−527	0.023	90,21
110^{-3}	155	−112	−527	0.018	92,34
510^{-3}	129	−106	−520	0.012	94,89
<i>Compound II</i>					
110^{-5}	218	−138	−533	0.055	76,95
510^{-5}	210	−120	−527	0.042	82,12
110^{-4}	197	−144	−526	0.027	88,51
$5-10^{-4}$	160	−160	−5015	0.021	91,06
110^{-3}	135	−112	−510	0.016	93,19
510^{-3}	120	−108	−514	0.011	95,31
<i>Compound III</i>					
110^{-5}	205	132	−535	0.055	76,59
510^{-5}	197	123	−530	0.041	82,55
110^{-4}	165	134	−525	0.025	89,36
$5-10^{-4}$	140	125	−527	0.016	93,19
110^{-3}	125	106	−520	0.012	94,89
510^{-3}	115	102	−518	0.009	96,17

The values of ΔS^0 , calculated from the y-intercept of the straight line, are $-215.22 \text{ J}/(\text{mol} \cdot ^\circ\text{K})$ for the solution with no inhibitor and respectively -226.32 , -232.18 , and $-243.66 \text{ J}/(\text{mol} \cdot ^\circ\text{K})$ for the solutions containing compounds *I*, *II*, and *III*. The negative values of ΔS^0 suggest that the activated complex is the rate-determining step. The order of the system increases on going from the initial reactants to the activated complex [19].

Fig. 4 shows the anodic and cathodic polarization curves for a steel electrode, obtained at 30°C in a 1 M hydrochloric acid solution in the absence of and in the presence of inhibitor *III* at different concentrations. Similar curves were obtained for compounds *I* and *II* (not shown). The corrosion parameters calculated for the studied inhibitors are shown in Table 3: anodic b_a and cathodic b_c Tafel constants, corrosion potential E_{corr} , corrosion current density i_{corr} , and percentage inhibition efficiency (*IE*). We see that the inhibitors shift the anodic and cathodic branches of the polarization curves toward lower current density. This suggests that the studied compounds inhibit both hydrogen evolution and steel dissolution [14].

As we see from Table 3, as the inhibitor concentration increases we observe the following behavior:

- The anodic and cathodic Tafel constants do not change significantly, and consequently the inhibitors affect both Tafel lines in both directions. From this it follows that a change in the inhibitor concentration does not affect the corrosion reaction mechanism. Therefore the inhibiting effect of the compounds is due to simple site blocking on the electrode surface [20], thus decreasing the surface area exposed to the aggressive medium.
- The corrosion potentials are shifted toward less negative values; the corrosion current density decreases while the inhibition efficiency increases.
- The studied compounds are mixed corrosion inhibitors (mostly anodic).
- The inhibition efficiency of the inhibitors, calculated from the polarization measurement, decreases in the following series:

$$III > II > I.$$

Fig. 5 shows the Nyquist plots for steel in a 1 M hydrochloric acid solution in the absence of (*I*) and in the presence of compound *III* in a concentration of (M): 2) $1 \cdot 10^{-5}$; 3) $5 \cdot 10^{-5}$; 4) $1 \cdot 10^{-4}$; 5) $5 \cdot 10^{-4}$; 6) $1 \cdot 10^{-3}$; 7) $5 \cdot 10^{-3}$.

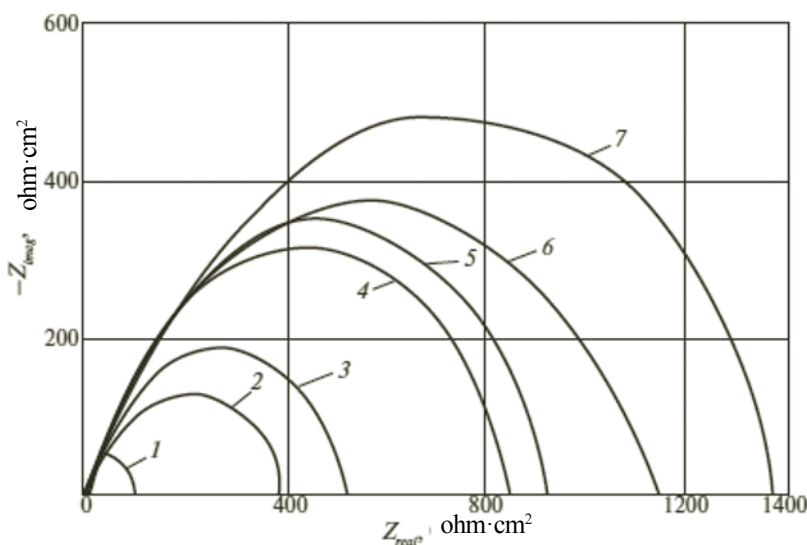


Fig. 5 Nyquist plots for carbon steel in a 1 M hydrochloric acid solution in the absence of (*I*) and in the presence of inhibitor *III* in a concentration of (M): 2) $1 \cdot 10^{-5}$; 3) $5 \cdot 10^{-5}$; 4) $1 \cdot 10^{-4}$; 5) $5 \cdot 10^{-4}$; 6) $1 \cdot 10^{-3}$; 7) $5 \cdot 10^{-3}$.

compounds *I* and *II* (not shown). The Nyquist plots represent semicircles; Z_{img} is the imaginary part of the impedance, Z_{real} is the real part of the impedance. The charge transfer resistance R_{ct} was calculated from the difference in impedance at low and higher frequencies [21]. In order to determine the double layer capacitance C_{dl} , we determined the frequency f at which the imaginary part of the impedance is maximum, i.e. $f(-Z'_{img})$. The value of C_{dl} was calculated from the equation:

$$f(-Z'_{img}) = \frac{1}{2TC_{dl}R_{ct}}$$

From the known charge transfer resistance, we calculated the percentage inhibition efficiency:

$$IE = \left[1 - \frac{R_{corr.add}}{R_{corr.free}} \right] \cdot 100$$

Table 4

Inhibitor concentration, M	R_{cp} , ohm/cm ²	C_d , μF/cm ²	IE, %
<i>No inhibitor</i>			
	112	125.4	—
<i>Compound I</i>			
$1 \cdot 10^{-5}$	282	36.1	71.21
$5 \cdot 10^{-5}$	420	26.5	78.86
$1 \cdot 10^{-4}$	682	21.6	82.77
$5 \cdot 10^{-4}$	855	11.45	90.86
$1 \cdot 10^{-3}$	990	8.58	93.15
$5 \cdot 10^{-3}$	1122	7.25	94.22
<i>Compound II</i>			
$1 \cdot 10^{-5}$	350	31.19	75.12
$5 \cdot 10^{-5}$	458	21.04	83.22
$1 \cdot 10^{-4}$	678	17.33	86.18
$5 \cdot 10^{-4}$	890	10.19	91.87
$1 \cdot 10^{-3}$	1012	7.75	93.82
$5 \cdot 10^{-3}$	1208	6.02	95.19
<i>Compound III</i>			
$1 \cdot 10^{-5}$	395	27.22	78.29
$5 \cdot 10^{-5}$	512	21.10	83.12
$1 \cdot 10^{-4}$	795	12.38	90.13
$5 \cdot 10^{-4}$	996	7.95	93.66
$1 \cdot 10^{-3}$	1082	6.12	95.12
$5 \cdot 10^{-3}$	1297	4.26	96.60

where $R_{ct,add}$, $R_{ct,free}$ are the charge transfer resistances respectively in the presence of and in the absence of the inhibitor.

The impedance parameters are given in Table 4. We see that as the inhibitor concentration increases, the charge transfer resistance increases while the double layer capacitance decreases. C_{dl} decreases because of adsorption of the inhibitors on the steel surface [22]. The inhibition efficiency of the inhibitors decreases in the series:

$$III > II > I.$$

The electrochemical impedance spectroscopy (EIS) results for the equivalent circuit used to model the steel/hydrochloric acid solution interface were explained earlier [23] for the process of corrosion of steel in an acid medium.

In aqueous media, non-ionic surfactants such as N,N-di(polyoxyethylene)-4-dodecylaniline significantly reduce the corrosion rate of steel because of their high adsorbability on the metal surface, forming a strong protective film. Adsorption usually occurs rapidly and accordingly the metal is protected from exposure to the aqueous medium. Furthermore, the adsorbed molecules can be incorporated into the oxide film and can react with it to form a protective three-dimensional network [24].

The inhibition efficiency of the considered compounds increases as the number of oxyethylene units in their molecules increases. These units facilitate adsorption due to the presence of an unshared electron pair. The inhibition efficiency of the considered compounds is different when calculated by different methods because of the different experimental conditions. However, the general trend in the variation in the inhibition efficiency in the series of compounds I–III is obvious.

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