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Synthesis of some Polymers Containing Heterocyclic Rings Corrosion Inhibitors of Mild Steel

A.A. Aly[1], A.A. Khalil[1], M.N. Ismail[2], Sh. A. Abd El-Kader[3] and M.M. Eldesouky[1]*

[1]Chemistry Department, Faculty of Science, Benha University, Benha, [2]Polymer Department, National Research Centre, Dokki, Giza, p.o.12622 and. [3]Basic Engineering Science Department, Faculty of Engineering, Benha University, Benha, Egypt.

A VARIETY of some polymers has been synthesized from the reaction of hydroxyquinazoline derivatives and amino quinazoline derivatives with methacrylic acid in the presence of N, N-dicyclo hexyl carbodimide (DCCI). The new monomers was polymerized in dimethyl form amide solution using azobis isobutyronitrile initiator. The new polymers were investigated for the corrosion inhibition properties for mild steel corrosion in Hcl solution. The result of these techniques indicated that the inhibition efficiency increased with the concentration of inhibitor and with decreasing the temperature; some thermodynamic parameters are calculated and explained for the tested systems from the data obtained at different temperatures.

Keywords: Hydroxyquinazoline derivatives, Aminoquinazoline derivatives, Monomer, Polymer, Steel, Hcl, Potentiodynamic polarization

Synthetic polymers are reported to have a wide range of activities such as anti-inflammatory[1], antifungal[2], anticonvalsant[3], antimalarial [4] and sedative[5]; some of these compounds are identified as drugs that are used as diuretics, were vasodilator and antihyper-tensive agents[6]. We report here the synthesis of new polymers with potentially wide spectrum of biological reposes.

Steel is widely used in industries and machinery and many other fields[12], the efficiency of an organic compound as an inhibitor depends on its ability to get adsorbed on the metal surface by replacing water molecule from metal surface[13], generally the compounds containing hetero atoms like O, N, S and P are found to work as very effective corrosion inhibitors. The efficiency of these compounds depends upon electron density present around the hetero atoms, the number of adsorption active centers in the molecule and their charge density, molecule size, mode of adsorption and formation of metallic complexes [14-27].

Experimental

Melting points are uncorrected. IR spectra in KBr were recorded on a perkin Elmer 298 spectrophotometer. ¹HNMR spectra were obtained on a varian Gemini 200 MHZ instrument using TMS as internal reference with chemical shifts expressed as ppm.

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*Mahmoud Eldesouky@yahoo.com

N-methacryl-amide (3-(4-hydroxy-quina-zolin-2-yl) -3- (naphthalene -2- yloxy) -1- phenylporpan) -1- one (4).

A mixture of (1) (0.01mol) and (0.01 mol) methacrylic acid in the presence of (0.01 mol) N,N- dicyclohexyl-carbodimide (DCCI) and methylene chloride (50/ml), the reaction mixture was found in ice bath and stirred for 8 hr, the formed solid was filterated to remove methylene chloride. The residue obtained (4), Yield, 56% m.p. 200-202 °C, IR: 3742 cm^{-1} (OH), 3324 cm^{-1} (CH aromatic), 3059 cm^{-1} (CH aliphatic), 1626 cm^{-1} (CO amide); ^{1}HNMR: (DMSO- d6) = 1.83 (s, 3H, CH$_3$), 5.5 (non identical protons, s, = CH$_2$), 7.42 (m, 16H, ArH).

N-methacrylamide (3-amino-1- (1-naphthalen-2-yloxy) -3- oxo -3-phenylpropyl) quinazolin -4(3H)-one (5)

A mixture of (2) (0.01 mol) and (0.01 mol) methacrylic acid in the presence of (0.01 mol) N, N-dicyclohexyl-carbodimide (DCCI) and methylene chloride (50)(ml); the reactions mixture was found in ice bath and stirred for 8 hr, the formed solid was filterated to remove mehylene chloride the residue obtained (5), Yield. 73% m.p. 165-167°C; IR: = 3326 cm^{-1} (NH), 3117cm^{-1} (CH aromatic), 2927 cm^{-1} (CH aliphatic), 1622 cm^{-1} (CO amide); ^{1}HNMR: (DMSO-d6) = 1.83 (s, 3H, CH$_3$), 5.38- 5.96 (non identical protons, s,= CH$_2$), 7.15 – 8.63 (m, 16H, ArH).

N-methacryl-amide (3-hydroxyl-1-(1-(naphthalene-2- yloxy) -3- oxo-3-phenylpropyl quinazolin- 4(3H)-one (6)

A mixture of (3) (0.01 mol) and (0.01 mol) methacrylic acid in the presence of (0.01 mol) N, N-dicyclohexyl carbodimide (DCCI) and methylene chloride (50)(ml); the reactions mixture was found in ice bath and stirred for 8 hr, the formed solid was filterated to remove methylene chloride, the residue obtained (6). Yield 51%, m.p. 170-172°C; IR: = 3901 cm^{-1} (OH), 3747 cm^{-1} (CH aromatic), 3327 cm^{-1} (CHaliphatic), 1625 cm^{-1} (CO amide); ^{1}HNMR: (DMSO-d6) = 1.83 (s, 3H, CH3), 5.95 (non identical protons, s, = CH2), 7.95 (m, 16H, ArH).

N-methacrylamide (3-(4-hydroxy quinazolin -2- yl) -3- (naphthalene -2- yloxy) -1- phenylpropan -1- one polymer (7)

N- methacrylamide (3- (4-Hydroxy quina zolin-2-yl)-3-(naphthalene -2-yloxy) -1- phenylpropan -1-one (4) was polymerized in dimethyl form-amide solution (1 mol%) using azobis-isobutyronitrile initiator at 70°C. The precipitated polymer was collected by filtration and purified-by precipitation from methanol, washed, dried and weighed, to give (7). Yield, 55%; IR: 3855 cm^{-1}, (oH) 3427 cm^{-1} (CH aromatic), 3064 cm^{-1} (CH aliphatic), 1655 cm^{-1} (CO amide); ^{1}HNMR: (DMSO-d6) = 2.5 (attributed to DMSO), 3.3 (attributed to H$_2$O), 2.8 (s, 2H, CH$_2$), 7.95 (m, 16H, ArH).

N-methacryl amide 3- amine -1- (1- (naphthalene -2- yloxy) -3- oxo -3- phenyl propyl) quinazolin - 4 (3H) – one) (polymer (8)

N-methacryl-amide (3-amino-1- (1-naphthalen -2- yloxy) -3- oxo -3- phenyl propyl) quinazolin-4(3H)-one monomer (5) was polymerized in dimethyl form amide solution (1mol%) using azobis isobutyronitrile initiator at 70°C. The precipitated polymer was collected by filtration and purified-by precipitation from methanol, washed, dried and weighed, to give (8). Yield, 52%; IR: = 3424 cm^{-1} (NH), 3063 cm^{-1} (CH aromatic), 2927 cm^{-1} (CH aliphatic) 1661 cm^{-1} (CO amide); ^{1}HNMR: (DMSO-d6) = 1.9 (s, 3H, CH$_3$), 2.8 (s, 2H, CH$_2$), 7.05-7.49 (m, 16H, ArH).

N- methacryl amide (3-hydroxy -1- (1-(naphthalen -2- yloxy) -3-oxo-3-phenyl propyl quinazolin-4 (3H) -one polymer (9)
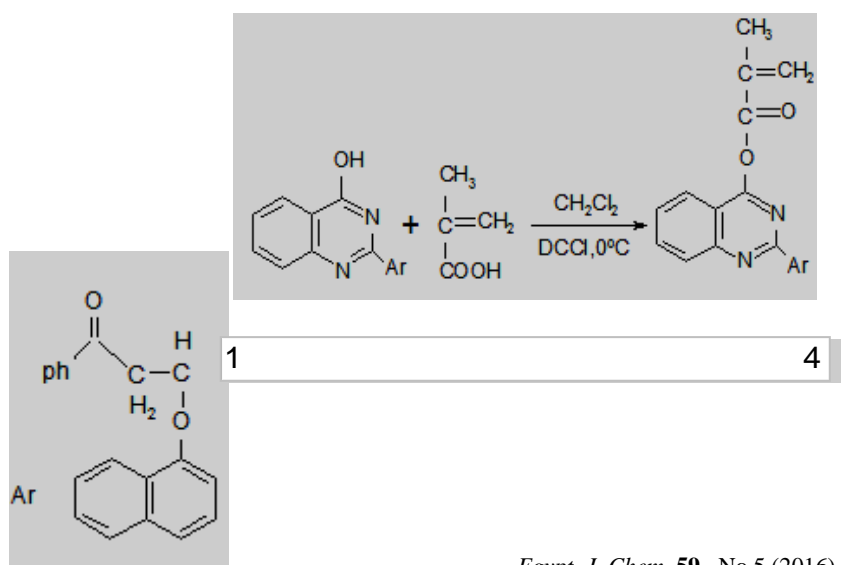
N- methacryl-amide (3-Hydroxy-1-(1-naphthalen-2-yloxy)-3-oxo-3-phenylpropyl-quinazolin- 4(3H)-one (6) was polymerized in dimethyl form-amide solution (1mol%) using azobis-isobutyronitrile initiator at 70°C. The precipitated polymer was collected by filtration and purified- by precipitation from methanol, washed, dried, and weighed to give (9). Yield 50%; IR: = 3743 cm^{-1} (OH), 3418 cm^{-1} (CH aromatic), 3061 cm^{-1} (CH aliphatic), 1622 cm^{-1} (CO amide); ^{1}HNMR: (DMSO-d6) = 1.7 (s, 3H, CH_3), 2.8 (s,2H, CH_2), 7.5 (m, 16H, ArH).

Steel electrode is sealed in a raldite resin with exposed surface area of 0.3695 cm^2. The electrode which is composed of C, (0.1%), Mn (0.4%), p (0.06%), S (0.0267%) and Fe rest. The electrode, surface was polished with different grades of emery papers 600 and 1200, degreased with acetone and rinsed with distilled water, the aggressive solutions of 0.1 M HCl were, prepared by dilution of analytical grade 34% HCl with distil distilled water.

Potentiodynamic polarization curves are preformed using economical potentiostat voltalab 21, PGP 201. The electrolytic cell was three necked glass bottle fitted with a platinum counter electrode, a saturated calomel electrode (SCE) as reference electrode and the working steel electrode.

Results and Discussions

The reaction of 3- (4-Hydroxy-quinazolin -2- yl) -3-(naphthalen-2-yloxy) -1- phenyl-propan -1- one (1) with methacrylic acid in the presence of N, N-dicyclohexyl-carbodimide (DCCI) afforded N-methacryl amide (3- (4- hydroxy quinazolin- 2-yl) -3- (naphtalen -2- yloxy) -1- phenyl propan -1- one (4), the structure of compound (4) was supported by elemental analysis and compatible spectroscopic data. Its IR spectrum showed absorption bands at 3742 cm^{-1} (OH), 3324 cm^{-1} (CH aromatic) 1626 cm^{-1} (CO amide).^{1}HNMR spectrum δ= 1.83 ppm due to CH_3, 5.5 ppm due to $CH_2 = C$.

N- methacryl amide (3-(4-hydroxy quinazolin -2-yl)-3- (naphtalen-2-yloxy) -1- phenyl propan -1- one (4) was polymerized in dimethyl form amide solution using azobis isobutylronitrile initiator afforded polymer of N- methacryl-amide (3-(4- hydroxy quinazolin-2-yl) -3-(naphthalene-2-yloxy)-1-phenyl-propan-1-one   polymer (7). The structure of compound 7 was confirmed from IR spectrum 3855 cm^{-1} (OH), 3427 cm^{-1} (CH aromatic). ^{1}HNMR spectrum δ = 2.5 ppm due to DMSO, 7.95 ppm due to ArH.
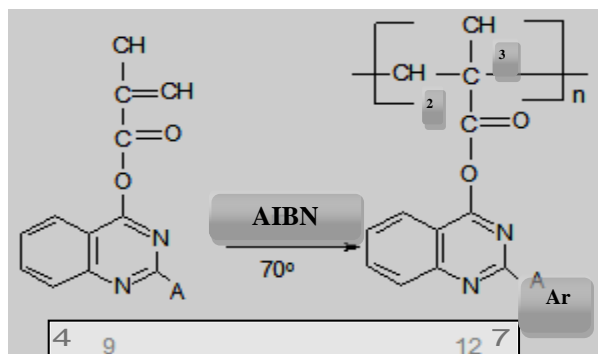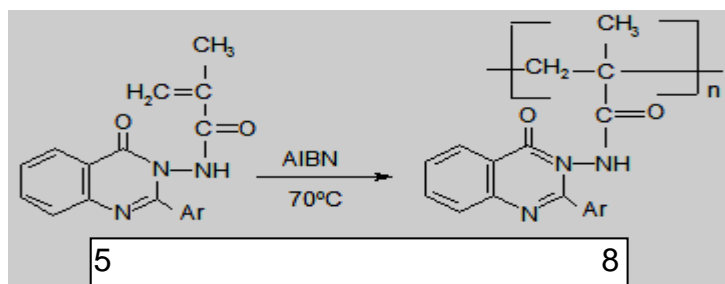

The reaction of 3- amino -1 (1-(naphthalene -2- yloxy) -3- oxo -3- phenyl-propyl) quinazolin- 4(3H) - one (2) with methacrylic acid in the presence of N, N-dicyclohexyl carbodimide afforded N- methacryl-amide (3- amino- 1- (1- (naphthalene-2-yloxy) -3- oxo -3- phenyl-propyl)-quinazolin-4(3H)-one monomer (5). The structure of compound (5) was established from IR- spectrum. 3326 cm^{-1}(NH), 3117cm^{-1} (CH aromatic), 1622 cm^{-1} (CO amide), ^{1}HNMR δ = 1.8 ppm due to CH$_3$, 5.98 ppm due to C= CH$_2$, 8.6 ppm due to ArH.
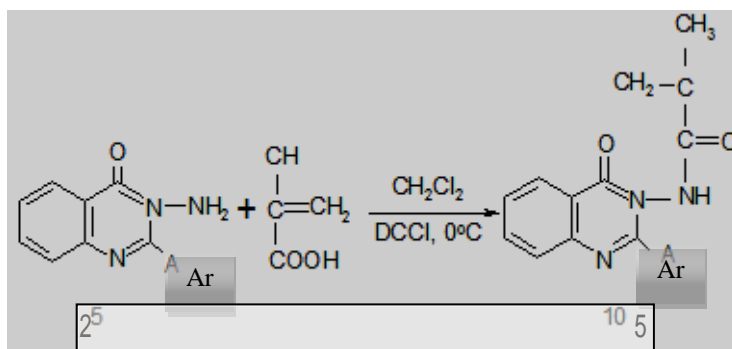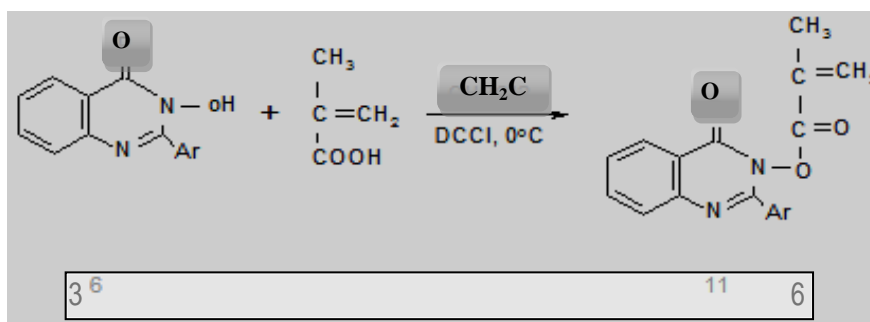

N-methacryl amide (3-amino -1- (1- (naphthalene -2- yloxy) -3- oxo -3- phenyl propyl) quinazolin -4(3H)-one monomer (5) was polymerized indimethyl formamide solution using atobis isobutyronitrile initiator afforded polymer of N-methacrylamide (3-amino -1- (1-(napthalen -2-yloxy) -3- oxo -3- phenylpropyl) quinazolin -4 (3H) -one (8). The   structure of compound (8) was confirmed from IR spectrum at 3424   cm^{-1} (NH), 3063 cm^{-1} (CH aromatic), 1661 cm^{-1} (CO amide). ^{1}HNMR spectrum δ = 1.9 ppm due to CH$_3$, 2.8 ppm due to CH$_2$, 7.05 ppm due to ArH.

The reaction of 3- hydroxy -1- (1- (naphthalene -2- yloxy) -3- oxo -3- phenyl-propyl-quinazolin -4(3H) – one (3) with methacrylic acid in presence of N, N-dicyclohexyl carbodimide (DCCI) afforded N-methacrylamide (3-Hydroxy-1- (1-(napthalen -2- yloxy)-3-oxo-3-phenyl-propyl quinazolin-4 (3H) - one monomer (6). The structure of compound (6) was established from IR spectrum 3901 cm^{-1} (OH), 3747 cm^{-1}(CH aromatic), 1625 cm^{-1} (CO amide) and ^{1}HNMR δ= 1.8 ppm due to CH$_3$, 5.9 ppm due to C=CH$_2$ 7.9 ppm due to ArH.

N-methacryl-amide (3-hydroxy-1-(1- (naphthalene -2-yloxy) -3- oxo -3- phenylpropyl quinazolin -4 (3H)-one (6) was polymerized in dimethyl form-amide solution using azobis isobutyronitrile initiator afforded of N-methacryl amide (3-hydroxy -1- (1-(naphthalen-2-yloxy) -3- oxo -3- phenyl-propyl quinazolin-4 (3H) - one (9). The structure of compound 9 was confirmed from IR-spectrum 3743 cm^{-1} (OH), 3418 cm^{-1} (CH aromatic), 1622 cm^{-1} (CO amide) and ^{1}HNMR spectrum δ 1.7 ppm due to CH$_3$, 2.8 ppm due to CH$_2$, 7.5 ppm due to ArH.
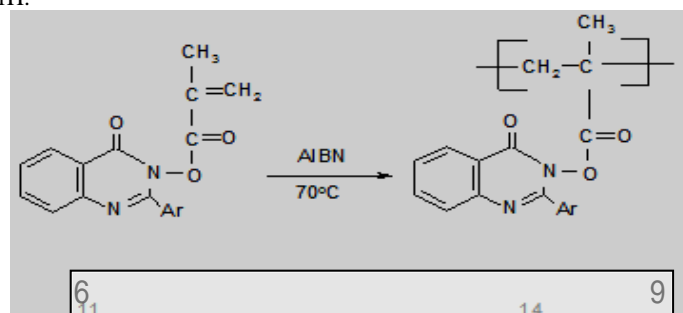
The structures of the synthesized compounds were assigned on the basis of elemental analysis and spectral data (cf, Experimental).

Potentiodynamic polarization curves of mild steel in 0.1 M HCl in the absence and presence of different concentration polymers are given in Fig.1. The potentiodynamic polarization parameters, *i.e.* corrosion potential (E_{corr}), anodictafel slopes (βa), cathodic tafel slopes (βc) corrosion current density (I_{corr}) and inhibition efficiency (%IE) are showed. The corrosion current density (I_{corr}) is calculated by the extra polation of anodic and cathodic tafel lines with the steady state corrosion potential (E_{corr}). The inhibition efficiency was calculated using the following equation:

$$IE = 1 - I_{add} / I_{free} \times 100$$

TABLE 1. Corrosion parameters of steel electrode in 0.1 M HCl solution containing different concentration of inhibitors.

Medium	β_a mV dec^{-1}	β_C mV dec^{-1}	-E_{corr} mVvs SCE	I_{corr} mA cm-2	%IE
Free HCl	88	98	530	0.7069	0.0
200 ppm comp (7)	103	123	542	0.5825	17.59
400ppm comp (7)	80	295	534	0.2902	59.9
600 ppm comp (7)	62	102	528	0.2591	63.3
800 ppm comp (7)	97	107	518	0.2226	68.5
1000 ppm comp (7)	71	96	525	0.1355	80.8
200 ppm comp (8)	84	143	517	0.3073	56.5
400 ppm comp (8)	63	133	529	0.1228	82.6
600 ppm comp (8)	145	172	515	0.0995	85.9
800 ppm comp (8)	207	251	469	0.0382	94.6
1000 ppm comp (8)	97	113	556	0.0336	95.2
200 ppm comp (9)	79	92	534	0.5140	27.32
400 ppm comp (9)	62	101	530	0.4329	38.7
600 ppm comp (9)	81	105	527	0.4264	39.6
800 ppm comp (9)	74	179	523	0.3963	43.9
1000 ppm comp (9)	122	121	503	0.0107	98.5

Adsorption isotherm

The degree of surface coverage (Θ) of steel surface by the adsorbed of polymers is calculated at constant potential using the following equation:

$\Theta = 1 - (I_{add}/I_{free})$ where I_{free} and I_{add} are the corrosion current densities in absence and presence of the additives compounds, respectively. The degree of surface coverage is found to increase with increasing concentration of the additives. Plotting of C/ Θ against concentration of inhibitor (C) gives straight lines with unit slopes. This indicates that the adsorption of the inhibitors takes place following Langmuir adsorption isotherm (Fig.2). This isotherm postulates that there is no interaction between the adsorbed molecules and the adsorption between the adsorbed molecules and the adsorption free energy independent of Θ value.
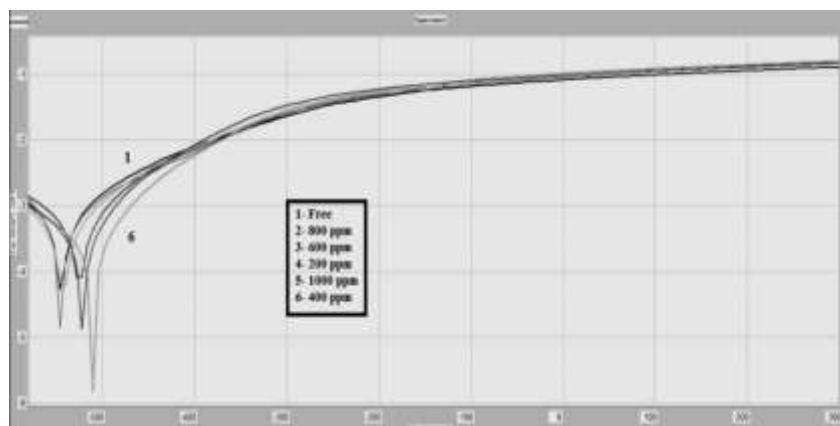
Fig. 1. Anodic and cathodic polarization curves of steel electrode in 0.1 M HCl solution containing different concentration of comp. 7-9 at 25°C.
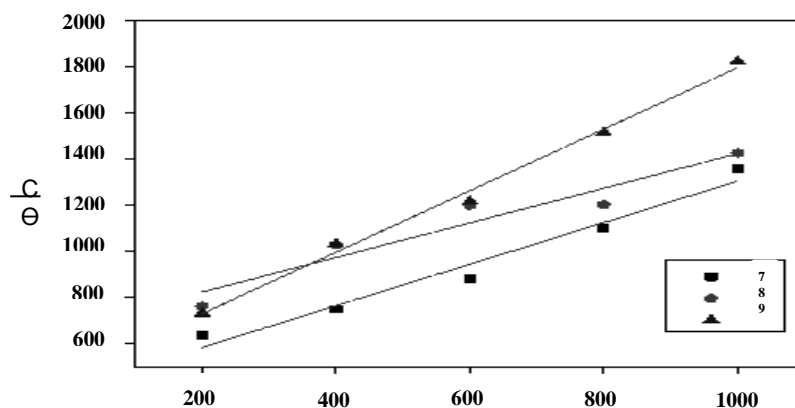

Fig. 2. Langmuir adsorption isotherm drawn using (ppm) concentrations of compounds (7-9) in 0.1 M HCl solution.

Effect of temperature

The effect of temperature of the corrosion parameters such as I_{corr}, E_{corr} and IE was studied in 0.1 M HCl solution, devoid and containing 1000 ppm of inhibitors over the temperature ranges of 25-65 °C. The results showed that the variation of temperature had almost effect on the shape of the polarization curves. The data listed in Table 2 show that, E_{corr} shifted to less negative values whereas the values of I_{corr} increased with the increase in temperature. This indicates the accelerating effect of rising temperature on the corrosion reaction on the other hand, the increase in temperature due to the decrease in the inhibition efficiencies of all the tested compounds.

The corrosion reaction is regarded as a rate process which in given by Arrhenius equation.

$$\text{Log } I_{corr} = \log A - E_a/ 2.303 \, RT$$

where I_{corr} represents the rate of corrosion reaction.

A is Arrhenius factor and E_a is the apparent activation energy of the corrosion reaction. Plotting the log ($\frac{Icorr}{T}$) versus $\left(\frac{1}{T}\right)$ gave straight lines in Fig 3.

TABLE 2. Effect of temperature on the corrosion parameters of steel electrode in 0.1 M HCl + 1000 ppm of inhibiters.

| T (K)	- E_corr mV vs SCE	I_corr mA cm-2	%IE
0.1 M Hcl			
298	530	0.7069	
308	540	0.7345	
318	517	0.8009	
328	532	0.8335	
338	525	0.9658	
0.1 M Hcl + comp (7)			
298	525	0.1355	80.8
308	524	0.2188	70.2
318	535	0.3777	52.8
328	529	0.4633	44.4
338	522	0.6360	34
0.1 M Hcl + comp (8)			
298	556	0.0336	95.2
308	543	0.1518	79.3
318	495	0.1803	77.5
328	525	0.2454	70.6
338	514	0.3934	59.3
0.1 M Hcl + comp (9)			
298	503	0.0107	98.5
308	497	0.3184	65.7
318	518	0.4226	47.2
328	529	0.6414	31
338	531	0.6603	23
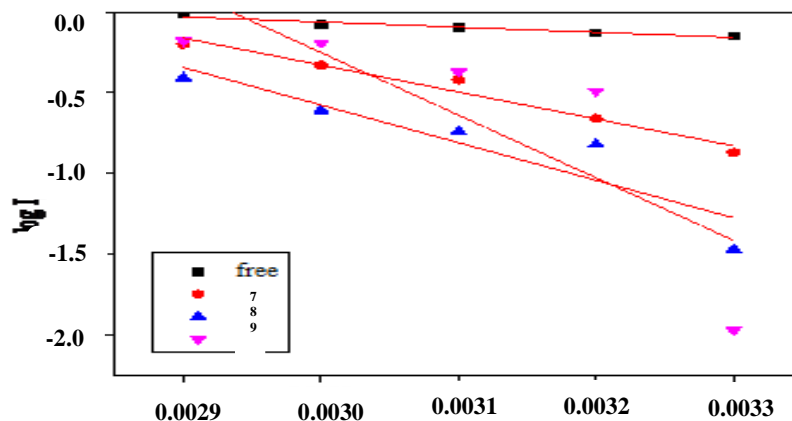
Fig. 3. Arrhenius plots of steel corrosion in 0.1 M HCl in absence and presence of 1000 ppm of compounds (7-9).

The other activation parameters were calculated using the transition state equation:

$$Log \frac{I corr}{T} = \left[Log \left(\frac{R}{hN}\right) \right] + \left[\frac{\Delta S*}{2.303R} \right] - \frac{\Delta H*}{2.303 RT}$$

where, R is the universal gas constant (8.314j/ mol.k), N is the Avogadro's number (6.02×10^{23}), h is the plank's constant ($6.62\times10^{34}m^2kg/s$) where $\Delta S*$ and $\Delta H*$ are the entropy and the enthalpy change of activation corrosion energies for the transition state complex, respectively, plotting log ($\frac{Icorr}{T}$) versus $\left(\frac{1}{T}\right)$ gives straight lines (Fig. 4).

$$Log \frac{Icorr}{T}$$

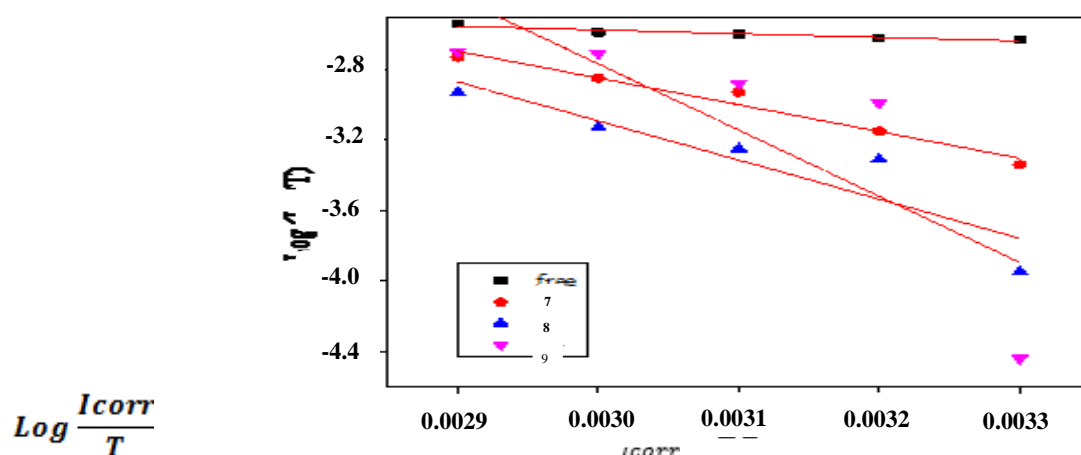**Fig. 4. The relation between log $\frac{icorr}{T}$ and 1/T for steel electrode in 0.1 M HCl in absence and presence of presence of 1000 ppm of compounds (7-9).**

Plotting log $\left(\dfrac{Icorr}{T}\right)$ versus (1/T) straight lines are obtained with a slope of $\dfrac{-\Delta H*}{2.303\ R}$ and an intercept of Log $\dfrac{R}{Nh} + \dfrac{\Delta S*}{2.303\ R}$ from which the values of ΔH* and ΔS* were calculated and listed in Table 3.

TABLE 3. Activation parameters of the dissolution reaction of steel electrode in 0.1 M HCl solution in absence and presence of 1000 ppm of polymers (7-9).

Concentration	Ea kJ mol^{-1}	$\Delta H *$ kJ mol^{-1}	$-\Delta S *$ kJ mol-1k-1	$\Delta G *$ kJ mol^{-1}
0.1 M HCl	6.1	7.4	0.223	73.8
0.1 M HCl + 1000 ppm comp (7)	22.8	21	0.214	75.8
0.1 M HCl + 1000 ppm comp (8)	24.3	29	0.165	84.2
0.1 M HCl + 1000 ppm comp (9)	23.2	42	0.129	80.3

Conclusion

The inhibition effect of the polymer compounds on mild steel in hydrochloric acid was examined by potentiodynamic polarization. The results of inhibition efficiency value increases with the increasing of the inhibitor concentration. Polarization curves indicated that the polymer acts as inhibitor in 0.1 MHCl solutions. The inhibition efficiently increases by decreasing the temperature. The inhibitions is accomplished by adsorption of the polymer compounds on the mild steel surface and the adsorption in spontaneous and obeys the Langmuir isotherm.

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تثبيط تآكل الصلب فى المحاليل الحامضية
باستخدام بعض البلمرات المحتوية على حلقات
غير متجانسة

- تضمن البحث تحضير بعض المونومرات 4، 5، 6 عند تفاعل حمض الميثاكريلك فى وجود كلوريد الميثيلين وداى سيكلوهكسيل كربوداى إيميد كعامل مساعد نازع للماء عند درجة حرارة صفر درجة مئوية مع المركبات المحضرة 1، 2، 3.

- كما تم تحضير البلمرات 7، 8، 9 من بلمرة المونومرات 4، 5، 6 فى وجود أزوبس بيوترونيتريل كبادئ وثنائى ميثيل فورم أميد كمذيب عند درجة حرارة 70 درجة مئوية.

- تم دراسة تأثير البلمرات المشيدة كمثبطات ذات كفاءة لتآكل الصلب الكربونى فى محلول حمض الهيدروكلوريك ووجد أن تزيد كفاءة تثبيط البلمرات بزيادة تركيزها وأيضاً تقل كفاءة تثبيط جميع البلمرات بزيادة درجة الحرارة.