Effect of temperature on the corrosion inhibition on mild steel in 2.0M H$_2$SO$_4$ by some organic compounds containing *S* and *N* atoms in absence and presence of halides

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ABSTRACT

The corrosion rate of mild steel in 2.0M H$_2$SO$_4$ containing 10% EtOH in absence and presence 0.1M halides was studied by both hydrogen evolution and mass loss methods. The obtained results showed that the rate of corrosion was increased with the increase of temperature . The activation parameters for mild steel corrosion in 2.0M H$_2$SO$_4$ in absence and presence 0.1 M Cl$^-$ & Br$^-$ show that the presence of Cl$^-$ or Br$^-$ in the corrosive medium leads to a decrease in the $\Delta E_{app,}$ $\Delta H^{\pm}$ and $\Delta S^{\pm}$ values than that of acid solutions which means that these ions becomes more effective as the temperature increases and corrosion inhibition is found . The studied compounds are found to accelerate the corrosion of mild steel at 20^{0}C then at 30^{0}C, the inhibition of corrosion is increased then a decrease in the inhibition of corrosion or acceleration at 50 to 60^{0}C in compounds A, B and C is found, which indicates that these compounds are physically adsorbed on the mild steel surface and it also indicated that the inhibited film formed on the metal surface most probably destroyed with faster rates at high temperatures. Corrosion inhibition by studied compounds in presence 0.1M Cl$^-$ or Br$^-$ at different temperatures shows that corrosion of mild steel largely decreases with rising temperature this was explained to be that the halides are chemically adsorbed on steel surface and a co-adsorption of the studied compounds (A-C) is occurred.

Key words: Corrosion inhibition, Mild steel, Sulphric acid, Action of halides, Effect of temperature.

INTRODUCTION

The corrosion process is usually accelerated when temperature is raised especially in media in which evolution of hydrogen accompanied corrosion. If oxygen takes part in a cathodic reaction during corrosion. The relationship between corrosion rate and temperature becomes more complicated owing to the lower solubility of oxygen at elevated temperatures[1].

Temperatures effects on acidic corrosion, most often in HCl and H$_2$SO$_4$ acid have been the object of a number of investigations and the activation energies is recorded. In general the higher value of activation energy in inhibitors absence often interpreted as an indication of the formation of adsorptive film of a physical (electrostatic) character. The opposite dependence demonstrates the chemisorptive between the inhibitor molecules and the metal surface is probable[2-10].

The effect of temperature in the range (20-60°) C on the performance of four studied compounds at coverage surface $\theta \approx 24\%$ of each of them on mild steel corrosion in 2.0 M H_2SO_4 containing 10% EtOH in absence and presence of halides (Cl^-, Br^-, I^-) will be carried out using chemical measurements(HEM and MLM).

EXPERIMENTAL

The four studied compounds employed in this investigation are shown in (table 1).They were friendly inhibitors and were used as medicine. The structure ,molecular weight of the studied compounds are presented in (table1).

Table 1: Symbol, source, trade name, chemical structure

Symbol	Trade name	Source	Chemical Structure
A	Diamox	Trademark	
B	Minid glypizide	Phrmaci & Upjohn	
C	Bactrim	Roche	
D	Lidaprim	Hasflund nycomed pharma	

Table 2 : Chemical composition of steel specimen

Fe	C	Si	Mn	Ni	P	S	V	Cu	Cr	Mo
98.52	0.31	0.21	0.81	0.02	0.01	0.01	0.002	0.06	0.02	0.02

The following chemical composition of the studied mild steel specimen is given in table(2).

Solutions of sulphuric acid (2.0M H_2SO_4) were prepared using bi-distilled water as the corrosive medium. Sulphuric acid and ethanol are used form analytical grade quality (BDH). Stock solutions of the studied compounds were prepared in ethanol .All test solutions were contained 10% ethanol to maintained the compounds completely soluble.

Before all measurements the specimens were polished first with series of emery papers of type (231 Qwetordry Imperical Paple aesoc), starting with a course one followed with the fine one as described elsewhere [6-9]. In each new study the measurements of the sample area was choked. The study was carried out using sample which has the same from as that described by Mylius[11].

The study is carried out by chemical methods at different temperatures (20-60) C°,using an ultra thermostat (JulaboU3No.8311), in absence

and presence of the studied compounds at fixed coverage (surface coverage $\theta \approx 24\%$) of the four studied compounds (A-D).

The study also was done in absence and in the presence at the fixed value of surface coverage ($\theta \approx 24\%$) of the compounds in 2.0M H_2SO_4 acid solutions in the absence and in the presence of (0.1M) of halides ions (Cl^-, Br^-, I^-).

RESULTS AND DISCUSSION

The effect of temperature on corrosion and corrosion inhibition on mild steel in 2.0 M H_2SO_4 by halides

The study of corrosion and corrosion inhibition of mild steel in 2.0M H_2SO_4 in absence and presence of halides using chemical measurements were carried out by hydrogen evolution and mass-loss methods at 20,30,40,50 and 60°C.

Figure (1a,b,c) shows the effect of temperature on the volume of hydrogen evolved in 2.0M H_2SO_4 in absence and presence 0.1M Cl^- and Br^- respectively.

It is clear that the corrosion rate of mild steel increases with rising temperature in both uninhibited and inhibited acid ,i.e. , the slope of the resulting straight lines mostly increases .

The inhibition percentages were calculated from hydrogen evolution (Inh.$_{HEM}$ %) and mass – loss (Inh . $_{MLM}$%) measurements using the following two equations[12-16]:

$$Inh._H\% = 100 (1 - R / Ro) \qquad ...(1)$$
$$Inh._M\% = 100 (1 - R' / Ro') \qquad ...(2)$$

Where R_o and R are the rates of hydrogen evolution in absence and presence of the halides respectively, and it will be given in the term (ml . cm^{-2}. min^{-1}), while R_o^- and R^- are the rates from mass- loss in absence and presence of the halides, respectively and it will be given in the term (gm. cm^{-2}.min^{-1}). Iodide ions gave inhibition about 100% at all temperatures, because of the strong adsorption of I^- on mild steel so it is excluded[17].

The variation of the Inh. % with temperature for the Cl^- & Br^- in 2.0M H_2SO_4 are

Table 3: Activation parameters for mild steel corrosion in
2.0M H_2SO_4 in absence and presence 0.1 M Cl^-& Br^-

halide	$\Delta E(kJ.mol^{-1})$	$\Delta H(kJ.mol^{-1})$	$-\Delta S(J.mol^{-1}K^{-1})$
Blank	50.47	48.38	340.322
Cl^-	32.13	33.05	204.49
Br^-	26.94	25.08	235.29

Table 4: The inhibition percentage for mild steel corrosion in 2.0M H_2SO_4 in
presence of studied compounds (A-D) at different temperatures

C° t	A		B		C		D	
	Inh.$_H$%	Inh.$_M$%	Inh.$_H$%	Inh.$_M$%	Inh.$_H$%	Inh.$_M$%	Inh.$_H$%	Inh.$_M$%
20	-3.22	-5.11	-47.09	-46.50	-12.90	-10.80	34.56	33.25
30	23.11	19.99	24.56	23.22	25.45	24.93	24.93	23.08
40	12.17	10.18	1.84	1.06	6.95	5.84	6.89	5.22
50	-1.47	-2.65	-7.15	-15.27	4.90	3.21	12.88	10.78
60	-5.29	-6.70	-30.64	-28.11	1.05	1.10	27.15	25.18

illustrated in Fig (2). As it can be seen that the inhibition percentage almost increases with the increase of temperature, this may be occurred by chemical adsorption of Cl^- or Br^- on mild steel. The adsorption of these halides on the metal surface reduces the surface area available for corrosion. The degree of protection increases with an increase in temperature due to higher degree of surface coverage resulting from enhanced halides adsorption[18,19]. The results calculated from two methods chemical are in good agreements.

In fig. (3) the logarithm of the corrosion rate of mild steel in absence and presence of 0.1M Cl^- & Br^- are plotted as a function of (1/T). Values of apparent activation energy, ΔE_{app} were calculated from the slopes of the straight lines and are given in table (3). In absence of halides, ΔE_{app} is equal to 50.47 kJ. M^{-1} for 2.0M H_2SO_4. The results are in good agreements with that obtained by a number of authors [19—23]. Values of ΔE_{app} in presence of Cl^-or Br^- are lower than 80 kJ mol $^{-1}$.

Table 5: Activation parameters for mild steel corrosion in 2.0M H_2SO_4 in presence of studied compounds(A-D) at different temperatures

halide	$\Delta E(kJ.mol^{-1})$	$\Delta H(kJ.mol^{-1})$	$-\Delta S(J.mol^{-1}K^{-1})$
A	65.52	61,34	595.144
B	69.51	9.37	38.168
C	97,58.	13.47	59.186
D	04.53	38.63	17.141

Table 6: The inhibition percentage for mild steel corrosion in 2.0M H_2SO_4 in presence of studied compounds (A-D) in (0.1)M Cl- at different temperatures

C° t	A		B		C		D	
	$Inh._H\%$	$Inh._M\%$	$Inh._H\%$	$Inh._M\%$	$Inh._H\%$	$Inh._M\%$	$Inh._H\%$	$Inh._M\%$
20	28.97	27.00	40.50	38.22	21.29	20.11	65.16	63.26
30	48.81	46.53	59.75	58.25	65.15	63.51	76.16	74.21
40	53.41	50.25	64.17	63.80	90.43	88.21	60.00	59.30
50	59.95	57.91	65.62	64.21	91.47	89.30	63.77	62.11
60	65.41	63.25	67.17	66.26	91.63	90.11	25.81	24.18

Table 7: The inhibition percentage for mild steel corrosion in 2.0M H_2SO_4 in presence of studied compounds (A-D) in(0.1)M Br-at different temperatures

C° t	A		B		C		D	
	$Inh._H\%$	$Inh._M\%$	$Inh._H\%$	$Inh._M\%$	$Inh._H\%$	$Inh._M\%$	$Inh._H\%$	$Inh._M\%$
20	86.39	65.31	69.39	65.11	85.87	83.25	67.74	65.27
30	83.25	80.11	89.01	87.12	86.80	84.67	88.99	84.50
40	84.99	82.53	90.11	89.81	93.70	92.11	69.83	64.80
50	87.08	85.70	91.18	90.23	94.51	93.27	72.74	70.55
60	87.12	86.45	92.45	91.17	96.14	94.57	56.89	54.44

Table 8: Activation parameters for mild steel corrosion in 2.0M H$_2$SO$_4$ in presence of studied compounds(A-D)

Comp.	ΔE (kJ.mol^{-1})	ΔH (kJ.mol^{-1})	-ΔS (kJ.mol^{-1}K^{-1})
A	38.68	61,34	196.69
B	40.41	9.37	187.59
C	49.68	13.47	170.63
D	66.82	38.63	106.60

Table 8: Activation parameters for mild steel corrosion in 2.0 M H$_2$SO$_4$ in presence of studied compounds(A-D) in (0.1) M Br- at different temperatures

Comp.	ΔE (kJ.mol^{-1})	ΔH (kJ.mol^{-1})	-ΔS (kJ.mol^{-1}K^{-1})
A	49.91	46.43	232.57
B	40.24	41.83	115.08
C	31.07	28.20	241.69
D	59.74	69.12	94.44
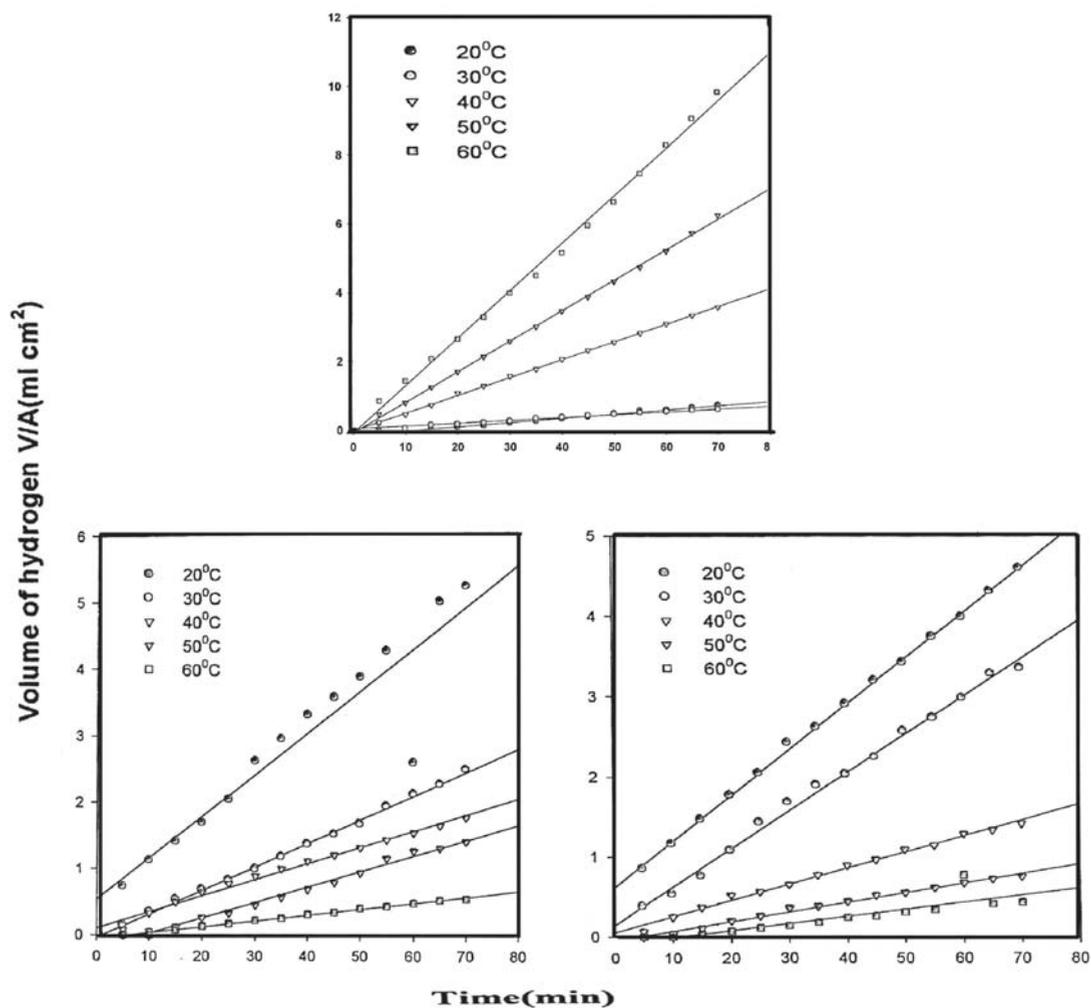
Fig.1(a,b,c):Volume of hydrogen/time curves of mild steel in 2.0 M H$_2$SO$_4$ in absence and presence 0.1M (Cl$^-$,Br$^-$) at different temperatures

In table (3) the observed activation energy for the reaction in the inhibited solutions is lower than that for the reaction in the acid solution . Machu [24] concluded from his studies on the influence of temperature on corrosion inhibition that with powerful Cl⁻ or Br⁻ ,the activation energy is lower for the inhibited than for the uninhibited reaction , which means that an inhibitor becomes more effective as the temperature increases[25,26]. The lower activation energy was also explained by Putilova etal.[27] to be due to an increase in surface area of the metal covered by inhibitor molecules as the temperature rises.

Hoar and Holliday[27] also observed a lowering of activation energy upon addition of effective inhibitors, a result which they attributed to a slow rate of adsorption as a resultant of closer
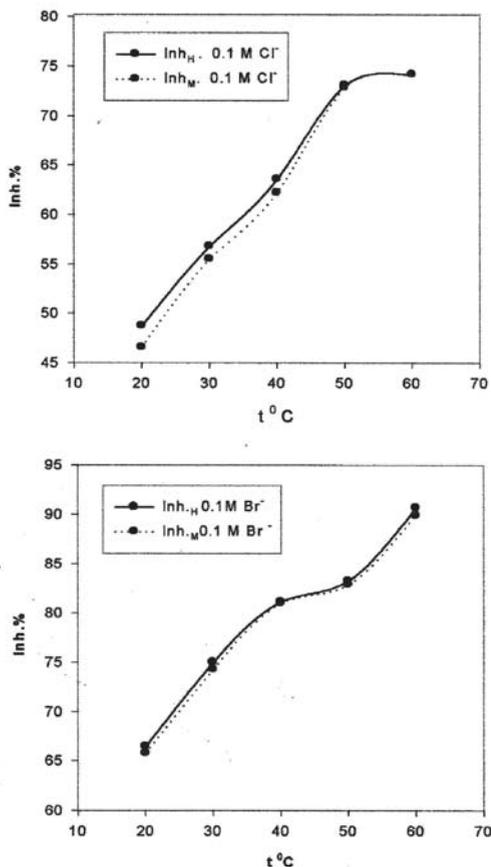
Fig. 2: Variation of inhibition efficiency with temperature for the corrosion of mild steel in 2.0 M H₂SO₄ in presence 0.1M (Cl⁻,Br⁻)
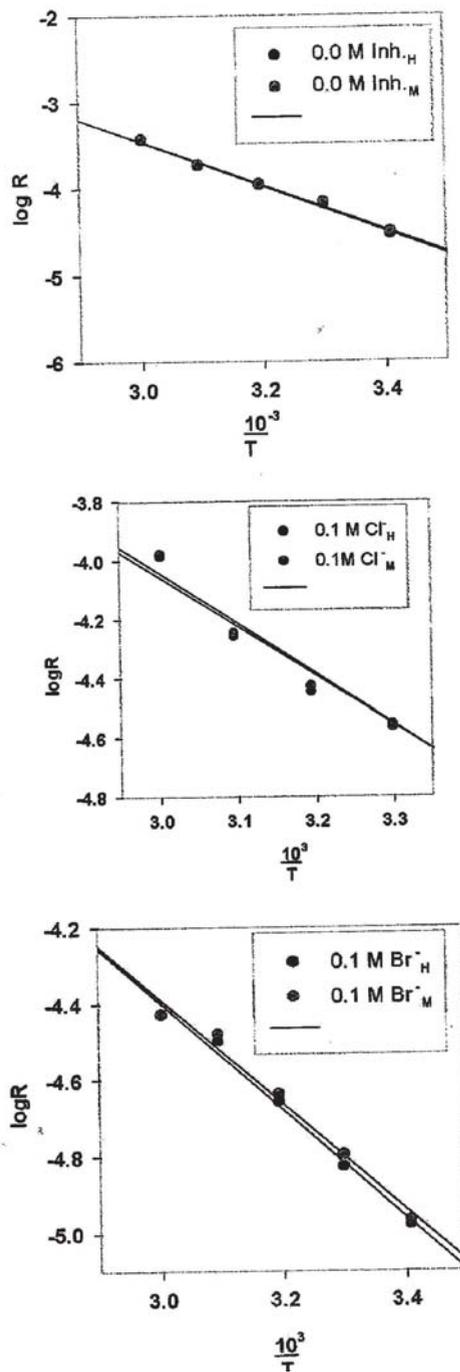
Fig. 3: Arrhenius plots for the corrosion of mild steel in2.0MH₂SO₄ in absence and presence 0.1M (Cl⁻,Br⁻)

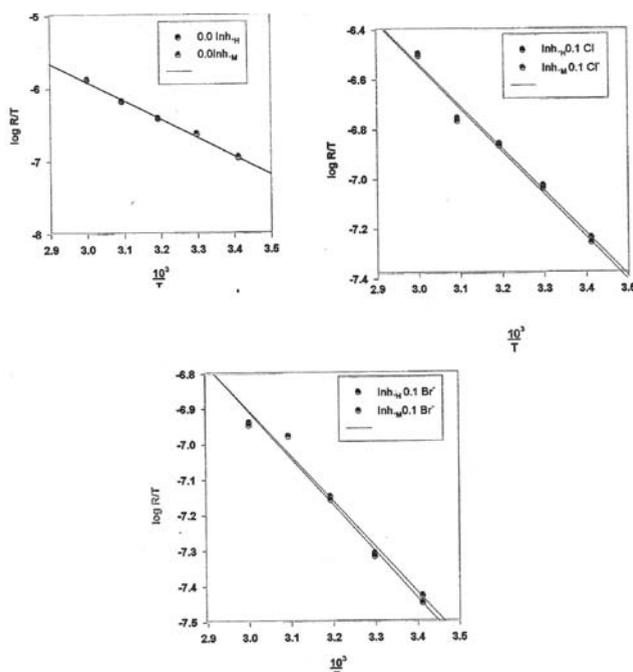**Fig. 4: Plots of log(R/T) vs (1/T) for the corrosion of mild steel in 2.0M H$_2$SO$_4$ in absence and presence 0.1M(Cl$^-$,Br$^-$)**
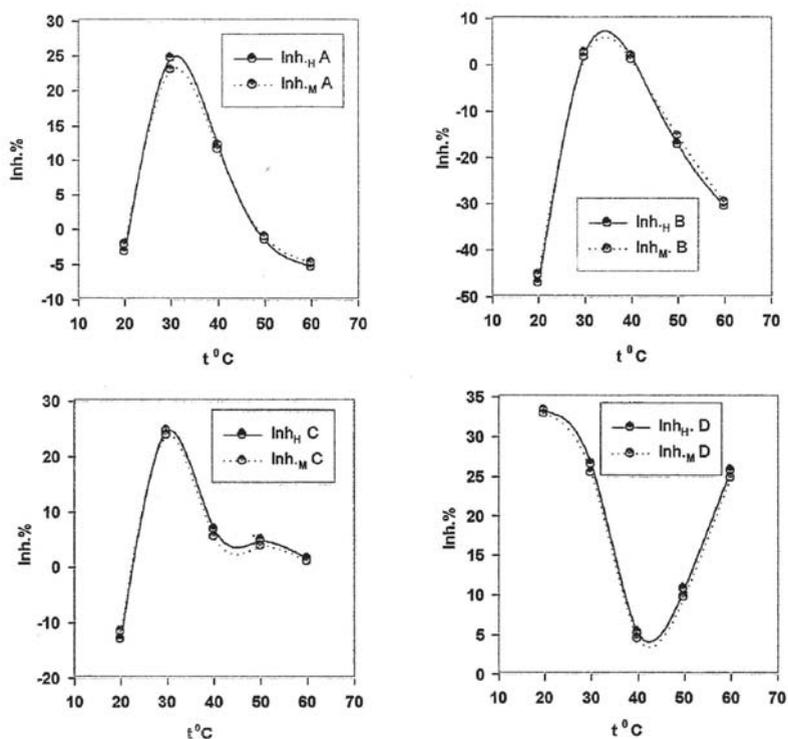

Fig. 5: Variation of inhibition efficiency with temperature for the corrosion of mild steel in 2.0M H$_2$SO$_4$ in presence of studied compounds

approach to equilibrium during the experiments at the higher temperatures.

An alternative form of Arrhenius equation is the transition state equation [21 ,27-31] :

$$R = RT / Nh \ exp \ (\Delta S^{\pm}/ R) \ exp \ (-\Delta H^{\pm}/RT) \quad ...(3)$$

where h is plank's constant, N Avogadro's number, $\Delta S^{\pm}$ the entropy of activation and $\Delta H^{\pm}$ the enthalpy of activation . The plots of log (R/T) vs .1/T in absence and presence of halides gave straight lines (Fig. 4), with a slope of $\Delta H^{\pm}$ /2.303R and an intercept of (log R / Nh + $\Delta S^{\pm}$ /2.303*R*) from which the values of $\Delta H^{\pm}$ and $\Delta S^{\pm}$ are calculated table (3).

The table shows that the presence of Cl⁻ or Br⁻ in the corrosive medium leads to a slight decrease in the $\Delta H^{\pm}$,$\Delta S^{\pm}$ values than that of acid solutions .
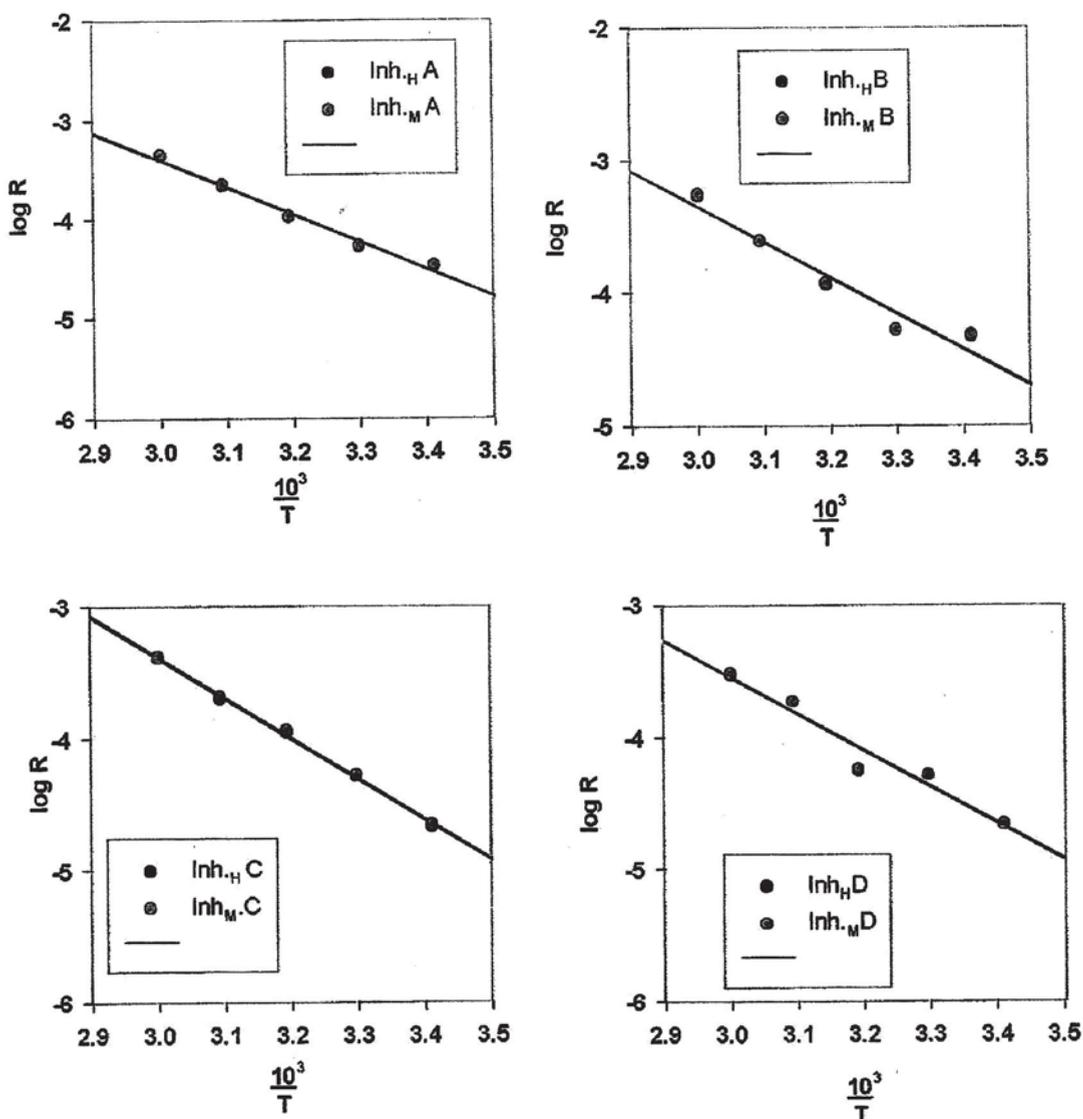

Fig. 6: Arrhenius plots for the corrosion of mild steel in
2.0 M H$_2$SO$_4$ in presence of studied compounds

The effect of temperature on the inhibition of the acid corrosion of mild steel in 2.0M H$_2$SO$_4$ by studied compounds

The effect of temperature in the range (20-60) C° on the performance of the studied compounds at coverage surface θ≈24% of them on mild steel corrosion in 2.0M H$_2$SO$_4$ was studied. It was found that the corrosion rate of mild steel increases with rising temperature.

Figure (5) shows the variation of Inh% with temperature for mild steel in studied compounds. The recorded (Inh. %) values at different temperatures in studied solution in table(4), showed that compounds (A,B,C) acted as inhibitors for corrosion at 20°C but at 30°C, the Inh.% of corrosion decreases with the increase in temperature in compounds (A,B,C) and acceleration of corrosion is observed. In compound (D) the Inh. % decreases
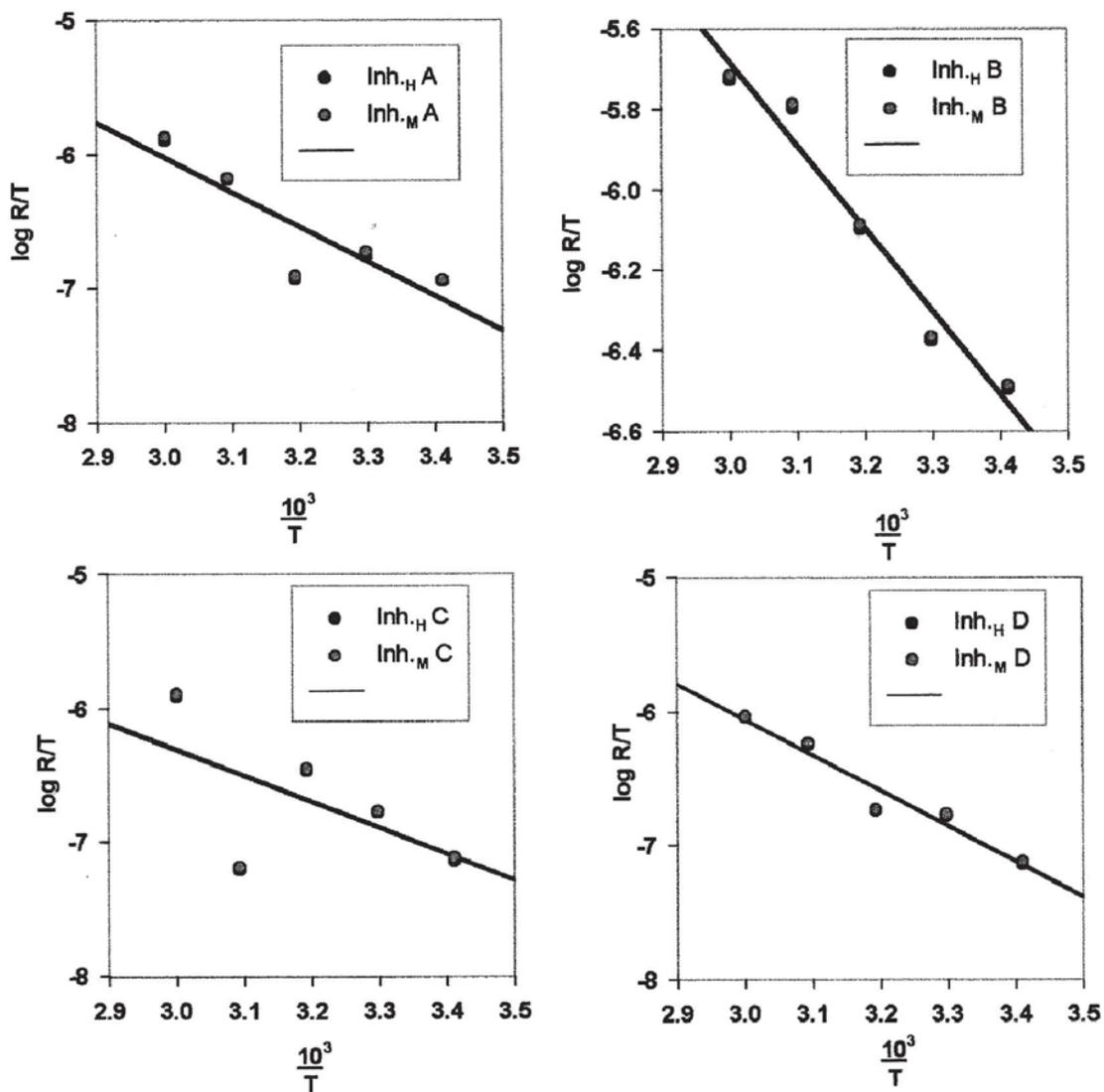

Fig 7: Plots of log (R/T) vs (1/T) for the corrosion of mild steel in 2.0M H$_2$SO$_4$ in presence of studied compounds

with increasing temperature ,until reach 50°C then a decrease in the corrosion rate or an increase in Inh. % with increasing temperature is occurred.

In general , the decrease in inhibition efficiency or in acceleration of corrosion on the mild steel surface indicated that the inhibited film formed on the metal surface is less protective in nature at higher temperatures(31) . Most probably desorption of the inhibitor molecules from the metal surface occurs with faster rate at high temperatures[32].

On the other side, the decrease in inhibition action with increase in temperature may by explained as that: the time lag between the process of adsorption and dsorption of inhibitor molecules over metal surface remains exposed to the acid environment for a longer period thereby increasing the rate of corrosion with increase in temperature and therefore inhibition efficiency falls for these compounds. In compound D it can be seen a slow decreasing of inhibition was obtained followed by increasing at the higher temperatures $\geq$40°C, the increasing of inhibition in compound D may
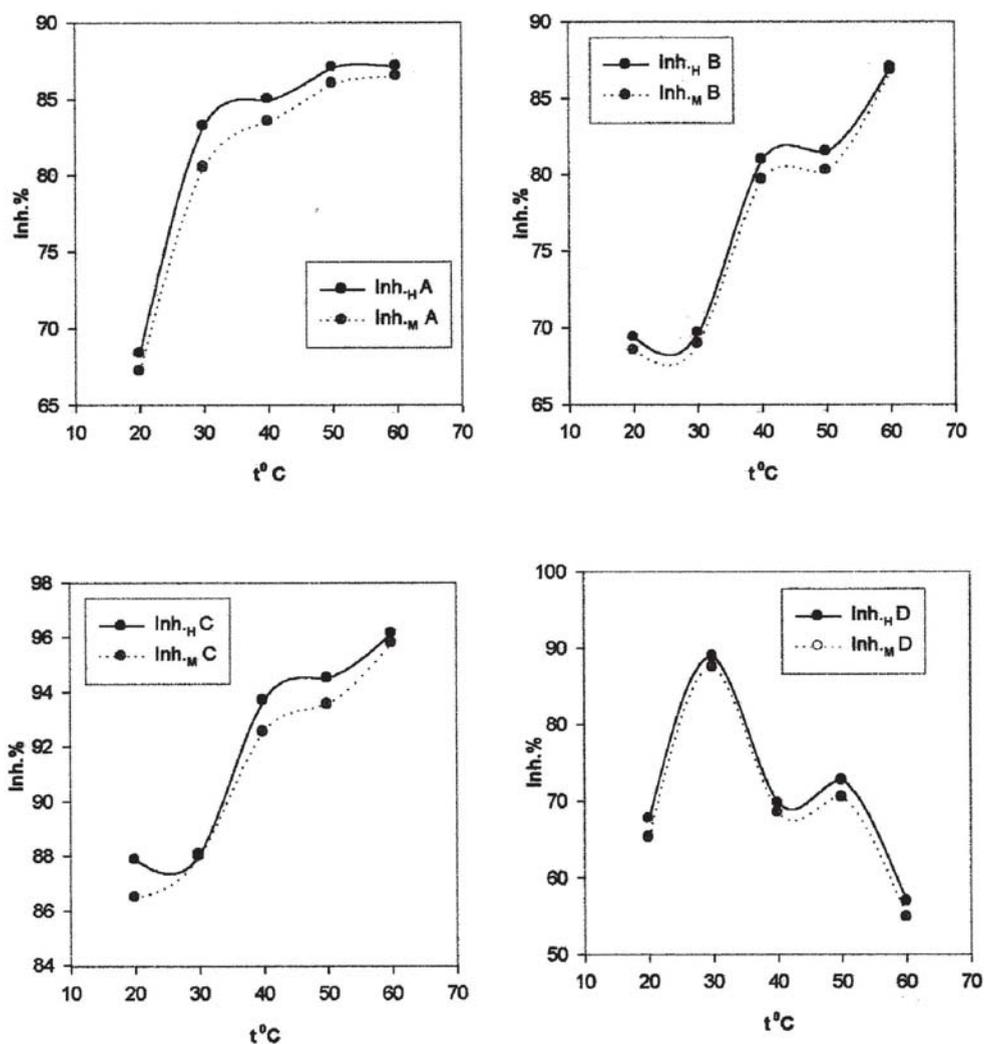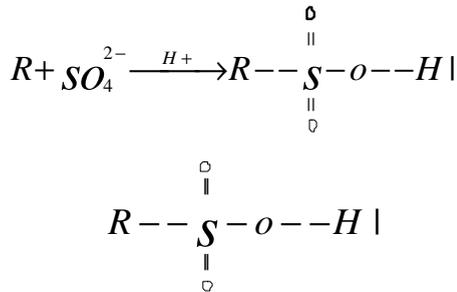

Fig. (8a):Variation of inhibition efficiency with temperature for the corrosion of mild steel in 2.0 M H$_2$SO$_4$ in presence of studied compounds in 0.1 M (Cl⁻)

explained by a chemical interaction between SO_4^{--} and methexy group at high temperature on mild steel dissolution. The increasing of inhibition with the increase of temperature as result of chemical reaction was reported before[33,34].

$$R + SO_4^{2-} \xrightarrow{H+} R--\overset{\overset{O}{\|}}{\underset{\underset{O}{\|}}{S}}-o--H|$$

$$R--\overset{\overset{O}{\|}}{\underset{\underset{O}{\|}}{S}}-o--H|$$

So the previous reaction will proceeds if R is an electron donating groups; in this study R is OCH_3 , electron donation groups . Also this reaction will proceed more rapidly when methyl group is presented as methoxy (compound D) than when it is as methyl (compound C) .

The formation of the complex will lead to increase of the covered mild steel surface area and to increase of inhibition percentage of compound (D) .

In figure (6) log R with 1/T for mild steel in presence of studied compounds at ($\theta \approx 24\%$)are shown and the obtained results are recorded in
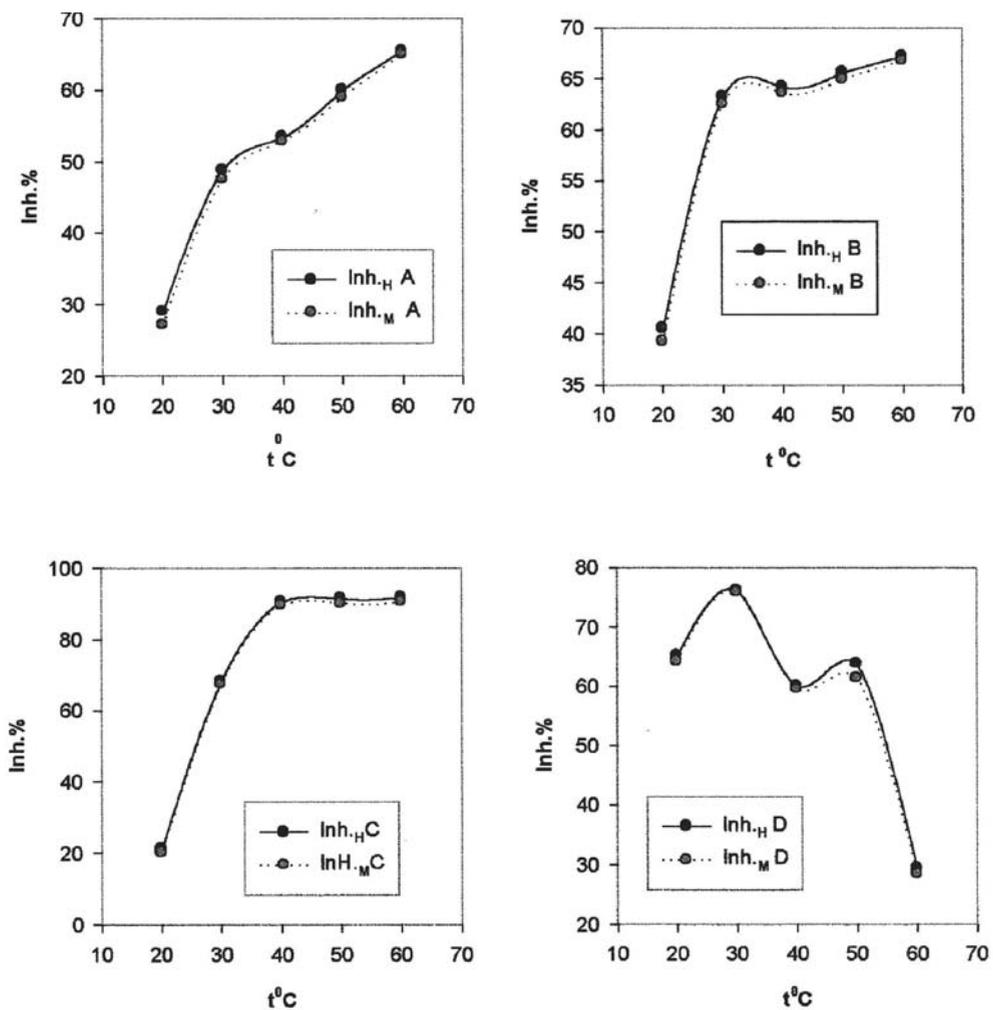

Fig. 8(b):Variation of inhibition efficiency with temperature for the corrosion of mild steel in 2.0 M H_2SO_4 in presence of studied compounds in 0.1 M (Br)
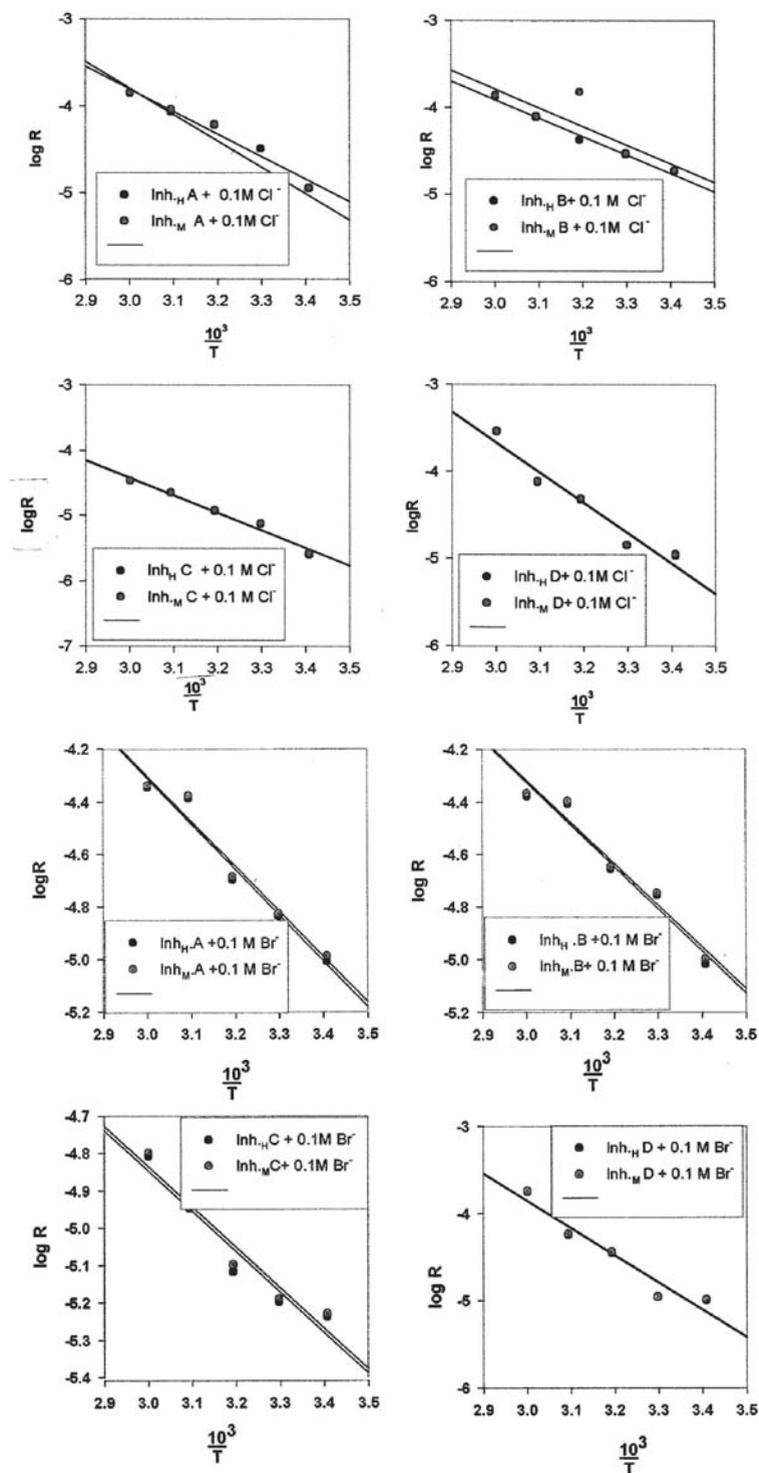
Fig. 9: Arrhenius plots for the corrosion of mild steel in 2.0 M H$_2$SO$_4$ in presence studied compounds in 0.1 M (Cl$^-$,Br$^-$).

table 5. Inspection of the data in table (5) shows that the apparent activation energy, ΔE_{app}. of the dissolution of mild steel in 2.0M H_2SO_4 is less than ΔE_{app}. for inhibited solution .The activity of the inhibitor which retards corrosion at low temperatures is considerably deduced at higher temperatures. The behavior of such inhibitors which include thiourea in H2SO4,can be compared with the behavior of unstable catalyst poisons. So adsorption falls appreciably with increasing, temperature, leading to considerable rise in the corrosion rate

due to the greater area of metal exposed to acid .

Hoar and Holliday[19] also observed a lowering of activation energy upon addition of effective inhibitors, a result which they attributed to a slow rate of adsorption with a resultant closer approach to equilibrium during the experiments at the higher temperatures.

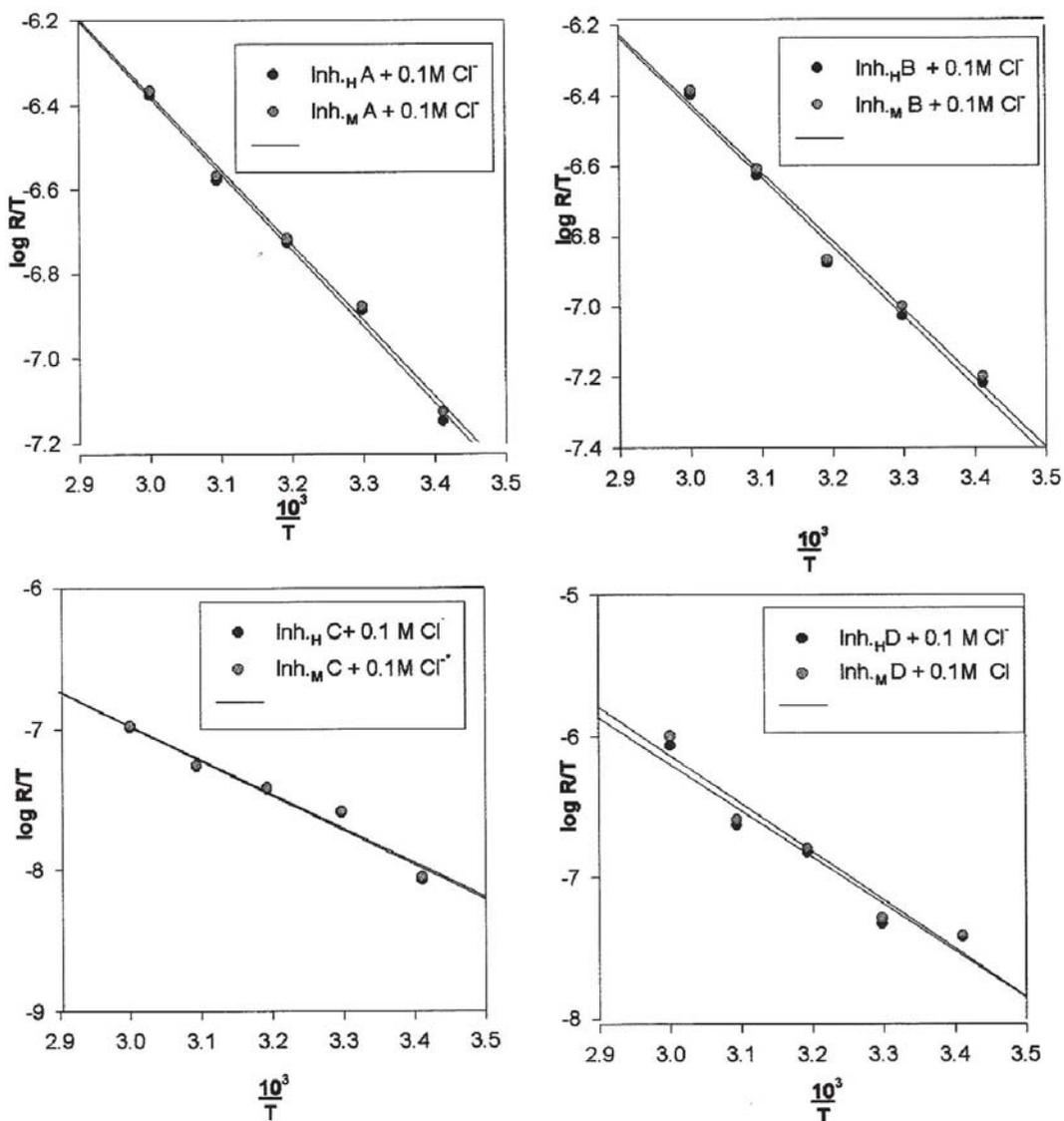Inspection of the data in table (5) also shows that the apparent activation energy , ΔE_{app}.

Fig. 10: Plots of log (R/T) vs (1/T) for the corrosion of mild steel in 2.0 M H_2SO_4 in presence studied compounds in 0.1M (Cl^-,Br^-)

,of the dissolution of mild steel in 2.0M H_2SO_4 in presence of studied compounds (A -D) are higher than that of the acid solution. This means that the presence of inhibitors tend to increase the value of activation energy and consequently decrease the rate of dissolution of the mild steel electrode. This indicates that the studied compounds act as inhibitors through the increasing the activation energy of the dissolution reaction by the adsorption at the metal solution interface making a mass barrier for charge transfer.

The plot of log (R/T) vs 1/T in presence of the studied compounds gave straight lines, Fig.(7), with a slope of - $\Delta H\pm$ /2.303 R and an intercept of (log R / Nh+ $\Delta S\pm$ /2.303R).

The effect of temperature on the inhibition of the acid corrosion of mild steel in 2.0M H_2SO_4 by the studied compounds in presence of 0.1M (Cl⁻ or Br⁻)

The effect of temperature in the range (20-60 °)C on the performance of the studied compounds in presence of 0.1M (Cl⁻ or Br⁻) on mild steel corrosion in 2.0M H_2SO_4 was carried out using chemical measurements. The results shows that the corrosion rate of mild steel increases with rising temperature.

Figures (8 a,b) show the variation of Inh.% with temperature for the studied compounds with 0.1 M Cl⁻ or Br⁻ in 2.0M H_2SO_4. These results are recorded in tables (6&7).As can be seen that the inhibition percentage almost increases with the increase of temperature probably by chemical adsorption of these compounds . In compound (D) it can be seen that the inh% increases, until reaches 40°C then a decrease at 60°C which may explained by a chemical interaction between compound and mild steel surface or between methoxy group presence in compound (D) with sulphat ions presented in H_2SO_4 then by the increase in temperature the bonding breaking and a decrease of inhibition is occurred.

The plots of log R vs I/T can be represented as straight line function (Arhenius equation) Fig. (9) where T is the temperature in Kelvin's in presence studied compounds with 0.1M Cl⁻ or Br⁻.

Values of $\Delta H\pm$ and $\Delta S\pm$ are calculated from both HEM and MLM and recorded in tables (8&9). The tables show that the presence of Cl⁻ or Br⁻ in the corrosive medium leads to a slight increase in the $\Delta E\pm_{app}$. than that of the acid solution . While in compound (D) $\Delta E\pm_{app}$ for the acid solution was lower than that for the inhibited one. This means that the presence of inhibitors with 0.1 Cl⁻ or Br⁻ tend to increase the rate of dissolution of the mild steel electrode.

The plots of log (R/T) vs. 1/T in presence studied compounds with 0.1M Cl⁻ or Br⁻ will gave straight lines with a slope of - $\Delta H\pm$ /2.303 *R* and an intercept of (log *R* / Nh + $\Delta S\pm$ / 2.303 *R*) , from which the values of $\Delta H\pm$ and $\Delta S\pm$ are calculated in tables (8&9) Figs.(10) .

CONCLUSION

It can be concluded that

´ The corrosion rate of mild steel in 2.0M H_2SO_4 containing 10% EtOH in absence and presence 0.1M halides is directly proportional to the increase in the temperature.

´ The ΔE_{app} in the presence of halides solutions were smaller than that of pure acid solution and it was explained to retarded metal dissolution in presence ions.

´ Studied compounds accelerated the corrosion of mild steel at 20°C then at 30°C the inhibition of corrosion is increased then a decrease in the inhibition of corrosion or acceleration at 50 to 60°C in compounds A, B and C is found, which indicates that these compounds are physically adsorbed on the mild steel surface and it also indicated that the inhibited film formed on the metal surface most probably destroyed with faster rates at high temperatures.

´ In compound D the corrosion inhibition continue to decrease then at 60°C the corrosion inhibition increases this was explained to be due to chemical interaction between the methoxy group of compounds D and SO_4^{2-} of the acid. The values of ΔE_{app} of the dissolution of mild steel in 2.0 M H_2SO_4 containing 10% EtOH in absence and presence of studied compounds ($\theta \approx 24\%$) show that inhibited solution are higher than that of the uninhibited.

Corrosion inhibition by studied compounds in presence 0.1 M Cl⁻ or Br⁻ at different temperatures shows that corrosion of mild steel decreases with rising temperature this was explained to be that the halides are chemically adsorbed on steel surface and a co-adsorption of the studied compounds (A-C) is occurred.

In compound(D) the inhibition efficiency increases until 40^0C then decreases the increases with increase of temperature this was explained on the basis that at low temperature the formation of chemical interaction between methoxy group in the compound (D) and SO_4^{2-} then at high temperatures this interaction become less.

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