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Effect of hexamethylpararosaniline chloride (crystal violet) on mild steel corrosion in acidic media

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ABSTRACT

The corrosion inhibition of mild steel in 0.5 M H_2SO_4 and 1 M HCl by hexamethylpararosaniline chloride (HMPC) was investigated using the gravimetric technique in the temperature range 303–333 K. The results indicate that HPMC inhibited the corrosion reaction in both acid media at all temperatures and inhibition efficiency increased with HMPC concentration. The inhibiting action is attributed to general adsorption of protonated and molecular HPMC species on the corroding metal surface. Adsorption followed a modified Langmuir isotherm and the Temkin isotherm, with very high negative values of the free energy of adsorption (ΔG^0_{ads}). An increase in temperature reduced the inhibition efficiency of HPMC in 0.5 M H_2SO_4 but increased efficiency in 1 M HCl. Activation parameters such as activation energy (E_a), activation enthalpy (ΔH^*) and activation entropy (ΔS^*) as well as the adsorption heat (Q_{ads}) were evaluated from the effect of temperature on corrosion and inhibition processes.

1. Introduction

The corrosion of iron and steel in acidic environments has been significantly suppressed by modification of the metal surface by adsorbed organic molecules [1–7]. The adsorbed inhibitor, depending on its molecular structure and the nature of the acid solution, may function by geometric blocking of active sites on the metal surface or may polarize the individual metal atoms to which they are attached, influencing the intrinsic reactivity of the metal. In any case, the active corrosion sites on the metal surface are occupied by the adsorbed inhibitor and the effectiveness of inhibition will be determined by the energy released on forming the metal-inhibitor bond compared to the corresponding changes when the pure acid reacts with the metal.

A series of reports from our laboratory [8–14] as well as those of other authors [15–17] provide considerable evidence that some organic dyes could function as effective corrosion inhibitors. The electronic structures of the dye chromophores, with extensive conjugation and presence of polar N, S or O atoms, play very important roles in this regard, facilitating their adsorption on the corroding metal surfaces according to some known adsorption isotherms. In continuation of our studies on the corrosion inhibiting efficacies of organic dyes, the present investigation was undertaken to elucidate the inhibiting effect of hexamethylpararosaniline chloride (HMPC), a basic triarylmethane dye known commercially as crystal violet (Fig. 1), on the corrosion of mild steel in hydrochloric and sulphuric acid solutions under varied experimental conditions. The effect of temperature on corrosion and inhibition processes was also assessed.

2. Experimental

Experiments were performed on mild steel sheets with weight percentage composition as follows; C-0.05, Mn-0.6, P-0.36, Si-0.03 and thickness 0.14 cm. The metal specimen were prepared, decreased and cleaned as previously described [8,9].

All chemicals and reagents were BDH grade and used as source without further purification. The blank corrodents were respectively 1 M HCl and 0.5 M H_2SO_4 solutions. The test inhibitor, HMPC was used in the concentration range 10^{-7}–10^{-3} M, while KI was used in 10^{-8}–10^{-6} M concentrations.

Gravimetric experiments were conducted on test coupons of dimension $3 \times 3 \times 0.14$ cm. Tests were conducted under total immersion conditions in 300 ml of test solutions at 303–333 K. The pre-cleaned and weighed coupons were suspended in beakers containing the test solutions using glass hooks and rods. All tests were made in aerated solutions and were run in triplicate. To determine weight loss with respect to time, the coupons were retrieved from test solutions at 2–5 h intervals appropriately cleaned, dried and re-weighed. The weight loss was taken to be

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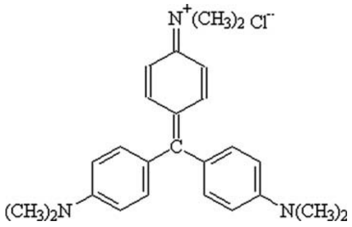

Fig. 1. Molecular structure of HMPC.

the difference between the weight of the coupons at a given time and its initial weight.

3. Results and discussion

3.1. Weight losses and corrosion rates

The corrosion rates of metals and alloys in aggressive solutions can be determined using different electrochemical and non-electrochemical techniques. The mechanism of anodic dissolution of iron in acidic solutions corresponds to [18]

$$\mathrm{Fe} + \mathrm{OH} \rightleftharpoons \mathrm{FeOH_{ads}} + \mathrm{H^{+}e} \qquad (1)(1a)$$

$$\mathrm{FeOH_{ads}} \rightarrow \mathrm{FeOH^{+}} + \mathrm{e} \qquad (2)(1b)$$

$$\mathrm{FeOH^{+}} + \mathrm{H^{+}} \rightleftharpoons \mathrm{Fe^{2+}} + \mathrm{H_2O} \qquad (3)(1c)$$

As a consequence of these reactions, including the high solubility of the corrosion products, the metal loses weight in the solution. Gravimetric techniques were thus employed in the present study. Fig. 2 compares the weight losses of the mild steel test coupons (Δw/g dm^{-2}) in 0.5 M H_2SO_4 and 1 M HCl solutions after 2 h and 5 h of immersion. The plots clearly illustrate higher corrosion susceptibility of the metal specimen in 0.5 M H_2SO_4, and corrosion rates in either aggressive solution were higher at longer exposure times. It has been suggested [19] that anions such as Cl^-, I^-, SO_4^{2-} and S^{2-} may also participate in forming reaction intermediates on the corroding metal surface, which either inhibit or stimulate corrosion. It is important to recognize that the suppression or stimulation of the dissolution process is initiated by the specific adsorption of the anion on the metal surface. Halide ions have been shown [8–11] to inhibit the corrosion of some metals in strong acids and this effect depends on the ion size, the electrostatic field set up by the charge of the ions on adsorption sites and the concentration of the halide ions. Indeed, this effect on mild steel is due to the ability of halide ions to replace hydroxyl ions adsorbed on the metal surface, thus reducing the catalytic effect of the hydroxyl ions.

The effects of different concentrations of HMPC on corrosion rates in both acid solutions after 5 h of exposure are shown in Fig. 3. HMPC is seen to reduce the weight loss at all studied concentrations, indicating inhibition of the corrosion reaction. This effect becomes more pronounced with increasing inhibitor concentration, which suggests that the inhibition process is sensitive to the amount of the additive present.

3.2. Inhibition efficiency and adsorption considerations

A quantitative characterization of the effect of HMPC on mild steel corrosion was achieved from assessment of the inhibition efficiency (η%) given by

$$\eta\% = \left(1 - \frac{\Delta w_{\mathrm{inh}}}{\Delta w_{\mathrm{blank}}}\right) \times 100 \qquad (4)(2)$$

where Δw_{inh} and $\Delta w_{\mathrm{blank}}$ are the corrosion rates in inhibited and uninhibited solution, respectively. Fig. 4 illustrates the variation of inhibition efficiency with logarithmic concentration of HPMC in 0.5 M H_2SO_4 and 1 M HCl. The plots show that η% increased progressively with inhibitor concentration. It is also obvious that the additive was more effective in 1 M HCl over a wide concentration range, suggesting that the nature of the acid anion influenced metal–inhibitor interactions.

Organic corrosion inhibitors are known to decrease metal dissolution via adsorption on the metal/corrodent interface to form a protective film which separates the metal surface from the corrosive medium. The adsorption route is usually regarded as a substitution process between the organic inhibitor in the aqueous solution [$\mathrm{Inh_{(sol)}}$] and water molecules adsorbed at the metal surface [$\mathrm{H_2O_{(ads)}}$] as follows [20,21]:

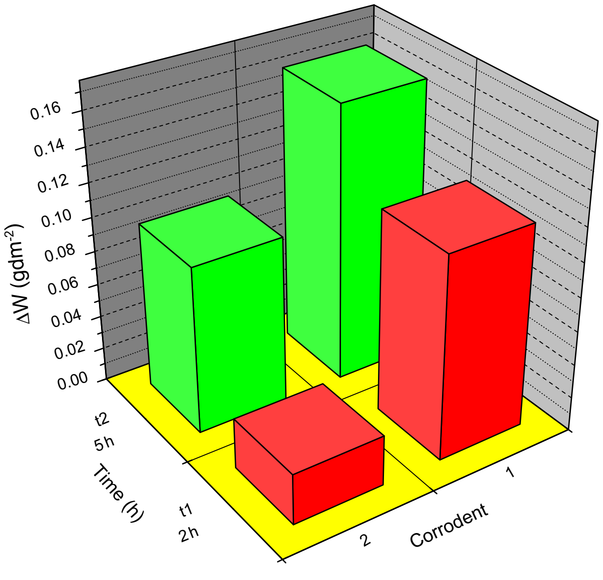

Fig. 2. Effect of exposure time on the weight loss of mild steel in (1) 0.5 M H_2SO_4 and (2) 1 M HCl.

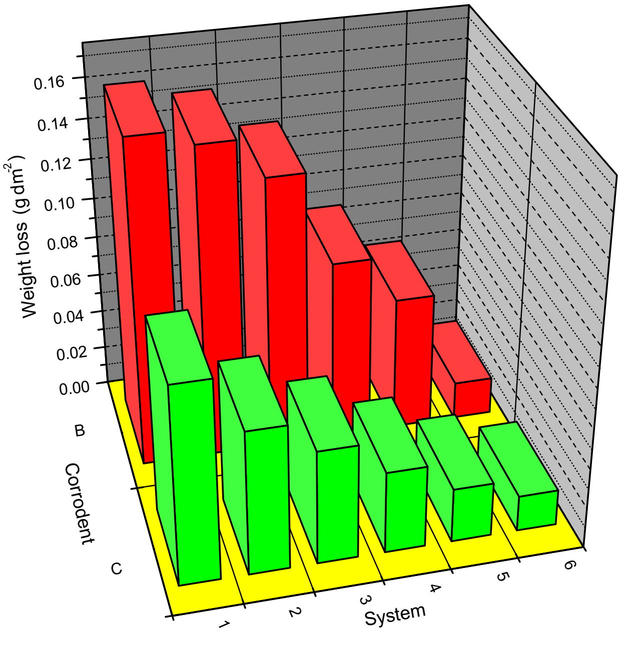

Fig. 3. Effect of different concentrations of HPMC on the corrosion of mild steel in (B) 0.5 M H_2SO_4 and (C) 1 M HCl; [1 = Blank; 2 = 0.0001, 3 = 0.001, 4 = 0.01, 5 = 0.1, 6 = 1.0 mM].

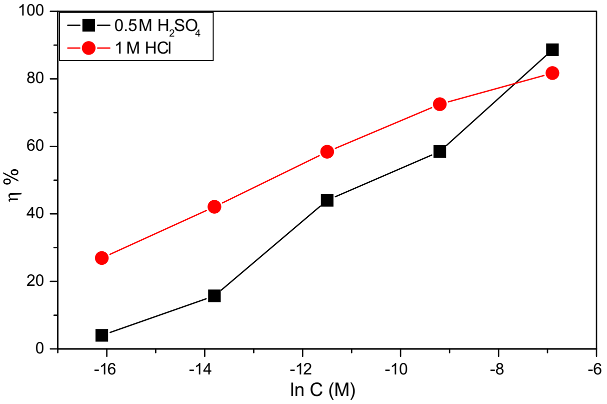

Fig. 4. Variation of inhibition efficiency with logarithmic concentration of HMPC.

$$Inh_{(sol)} + \chi H_2O_{(ads)} \leftrightharpoons Inh_{(ads)} \chi H_2O_{(sol)} \qquad (5)(3)$$

where χ represents the number of water molecules replaced by one molecule of adsorbed inhibitor. The adsorption bond strength is dependent on the composition of the metal and corrodent, inhibitor structure, concentration and orientation as well as temperature. In depth consideration of some of these variables will normally yield useful information regarding the adsorption mechanism. Since HMPC is a basic dye which can become protonated in the presence of strong acids, it is quite necessary to consider both cationic and molecular forms when discussing adsorption of the compound. The surface charge on iron in acidic solution is positive at the corrosion potential [5,20]. In hydrochloric acid, specific adsorption of chloride ions renders the metal surface more negative and susceptible to adsorption of protonated inhibitor species compared to sulphuric acid. Thus protonated HMPC species will be more readily adsorbed on the metal surface in 1 M HCl.

From Fig. 5 it can be seen that $\eta\%$ from HCl is lower at short exposure times, which means that formation of Fe–Inh complex is gradual and time dependent, a characteristic feature of the chemisorption process, which often results in improved inhibitor effectiveness. $\eta\%$ from H_2SO_4 on the other hand, did not show any notable time-dependence, implying that adsorption was rapid, probably physisorption.

The experimental data for HMPC were fitted to some frequently used adsorption isotherms of the general form [21,22]

$$f(\theta, \chi)\exp(-2a\theta) = KC \qquad (6)(4)$$

where $f(\theta, \chi)$ is the configurational factor which depends upon the physical model and underlying assumptions in the derivation of the isotherm, θ is the surface coverage related to the inhibition efficiency by [$\eta\% = 100 \times \theta$], C is the inhibitor concentration, χ is the size ratio representing the number of water molecules replaced by one molecule of adsorbate, a is the molecular interaction parameter and K is the equilibrium constant of adsorption. For the Langmuir isotherm, $\chi = 1$ and $a = 0$. Fig 6 shows plots of C/θ versus C, which was used to test for Langmuir behavior. The linear plots obtained suggest that HMPC adsorption from both acid solutions followed the Langmuir isotherm, though the isotherm parameters indicate some deviations from ideal Langmuir behavior. In either case, the slope deviates from unity (1.12 and 1.22 for 0.5

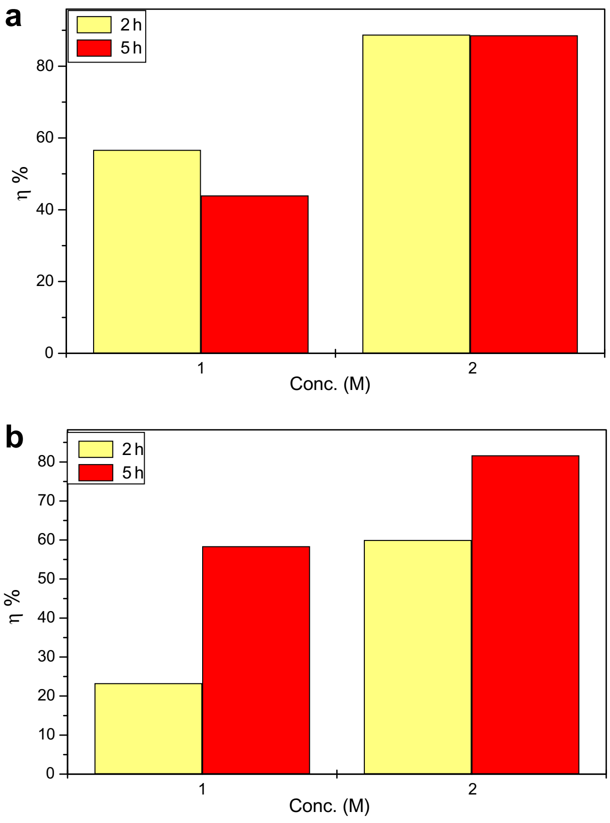

Fig. 5. Effect of exposure time on the inhibition efficiency of HMPC; [1 = 0.01 mM, 2 = 1.0 mM HPMC].

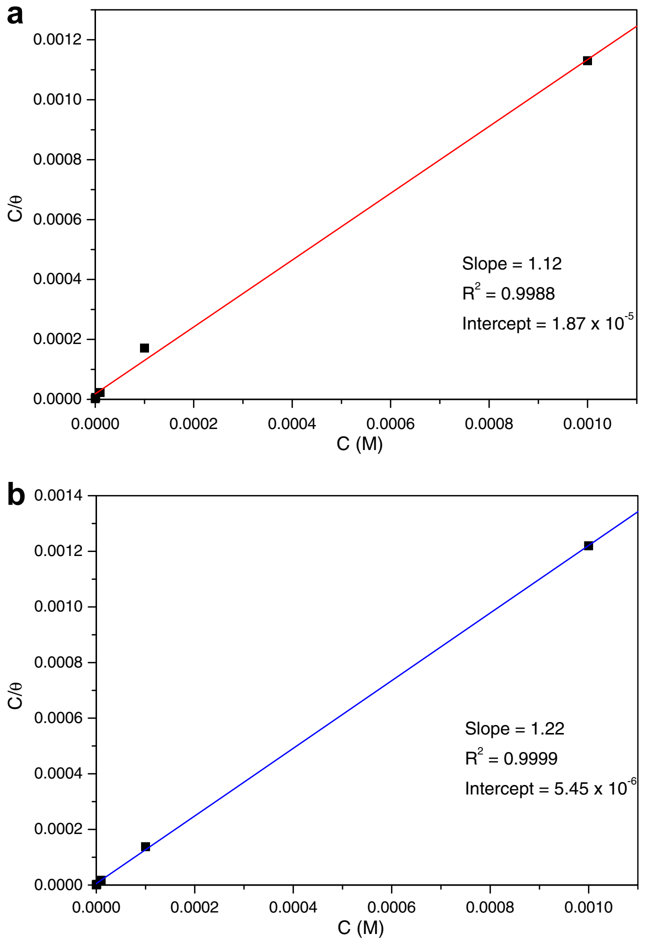

Fig. 6. Langmuir adsorption isotherm for HMPC on mild steel in 0.5 M H_2SO_4 and 1 M HCl.

M H_2SO_4 and 1 M HCl, respectively) with non zero intercept on the *y* axis, which could be traced to some limitations in the underlying assumptions. The results in fact imply that each HMPC molecule occupies 1.12 active corrosion sites on the mild steel surface in 0.5 M H_2SO_4 and 1.22 sites in 1 M HCl. For that reason the adsorption behavior is more appropriately represented by a modified Langmuir equation [23], taking into account the interactions between adsorbate species as well as changes in the heat of adsorption with changing surface coverage

$$\frac{C}{\theta} = \frac{n}{K_{ads}} + nC \qquad (5)$$

In the Temkin isotherm model, $\chi = 1$ and $a \neq 0$, where the values of *a* depend upon adsorbate–adsorbate interactions as well as adsorbate–adsorbent interactions. A negative *a* value corresponds to repulsion and a positive value to attraction. The plots of θ versus ln *C* give straight lines (Fig. 7) indicating that the adsorption of HMPC onto the mild steel surface from 0.5 M H_2SO_4 and 1 M HCl also follow the Temkin isotherm. Adherence to the Temkin isotherm is further evidence of the adsorption of neutral HMPC molecules on the metal surface from both acid solutions [5,24]. The free energies of adsorption (ΔG^0_{ads}) of HMPC from the different acid solutions were determined from K_{ads} by

$$K_{ads} = \frac{1}{55.5} \exp\left(-\frac{\Delta G^0_{ads}}{RT}\right) \qquad (7)(6)$$

ΔG^0_{ads} from 0.5 M H_2SO_4 was -50.6 kJ mol^{-1} and from 1 M HCl was -62.3 kJ mol^{-1}. The large negative ΔG^0_{ads} values imply that the adsorption of HMPC on the steel surface is favorable from thermodynamics point of view and indicates that the inhibitor is strongly adsorbed, covering both anodic and cathodic regions.

To further clarify the modes of inhibitor adsorption, experiments were conducted in the presence of iodide ions, which are strongly adsorbed on the surface of iron in acidic solution and facilitate adsorption of organic cation-type inhibitors by acting as intermediate bridges between the positive end of the organic cation and the positively charged metal surface. Specific adsorption of iodide ions on the metal surface leads to a recharging of the electrical double layer. The inhibitor is then drawn into the double layer by electrostatic interaction with the adsorbed I^- ions, forming ion pairs on the metal surface which increases the degree of surface coverage

$$I^-_{sol} \rightarrow I^-_{ads} \qquad (8)(7a)$$

$$I^-_{ads} + Inh^+_{sol} \rightarrow [I^- - Inh^+]ads \qquad (9)(7b)$$

Table 1
Effect of iodide ions on the corrosion of mild steel in 0.5 M H_2SO_4 and 1 M HCl in the absence and presence of HPMC

System	Inhibition efficiency (η%)	
	0.5 M H_2SO_4	1 M HCl
1×10^{-8} KI	60.8	31.7
1×10^{-6} KI	65.8	48.3
1×10^{-5} HPMC	56.7	23.3
1×10^{-8} KI + HPMC	84.7	70.0
1×10^{-6} KI + HPMC	80.0	58.3

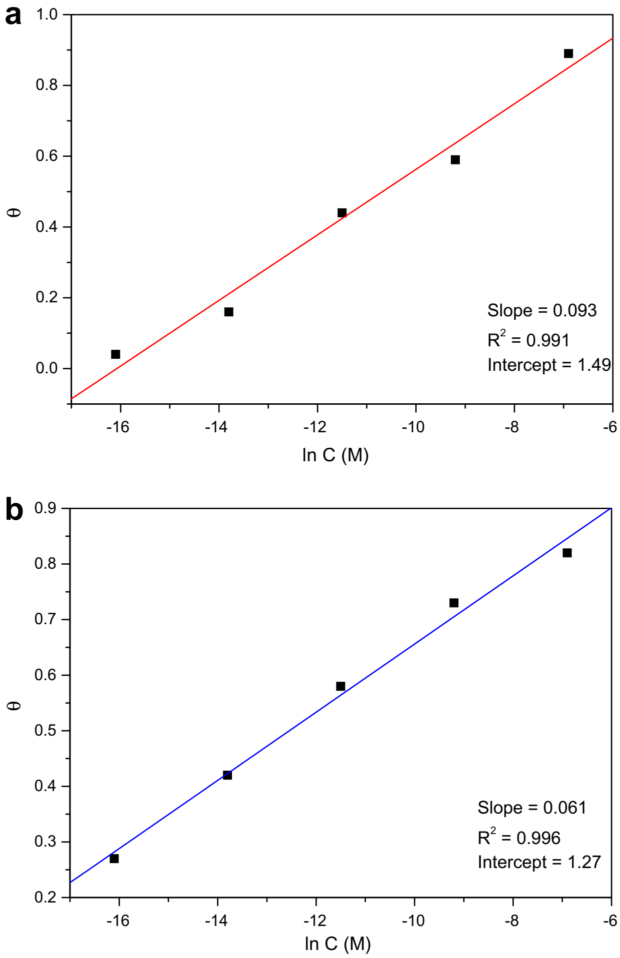

Fig. 7. Temkin isotherm for HMPC adsorption on mild steel in 0.5 M H_2SO_4 and 1 M HCl.

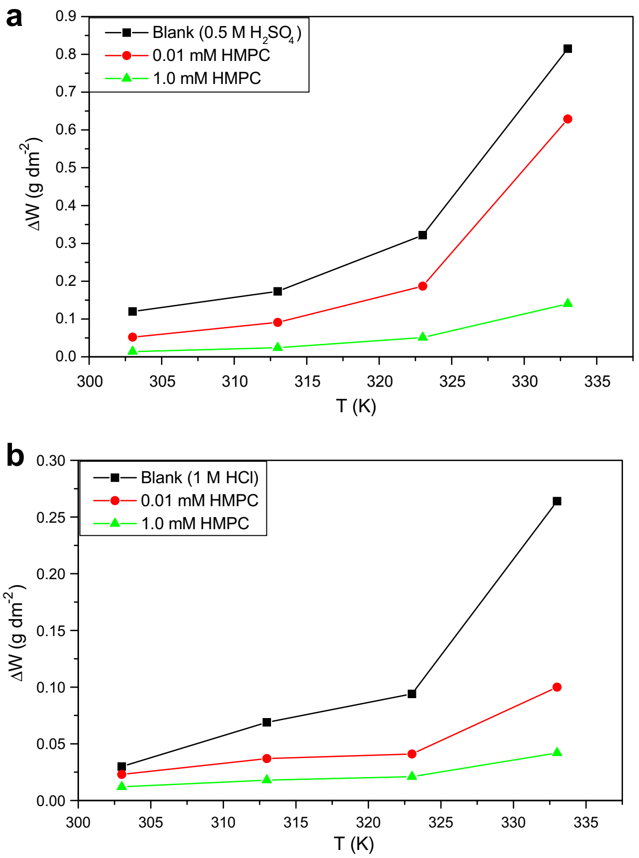

Fig. 8. Effect of temperature on the weight loss of mild steel in 0.5 M H_2SO_4 and 1 M HCl.

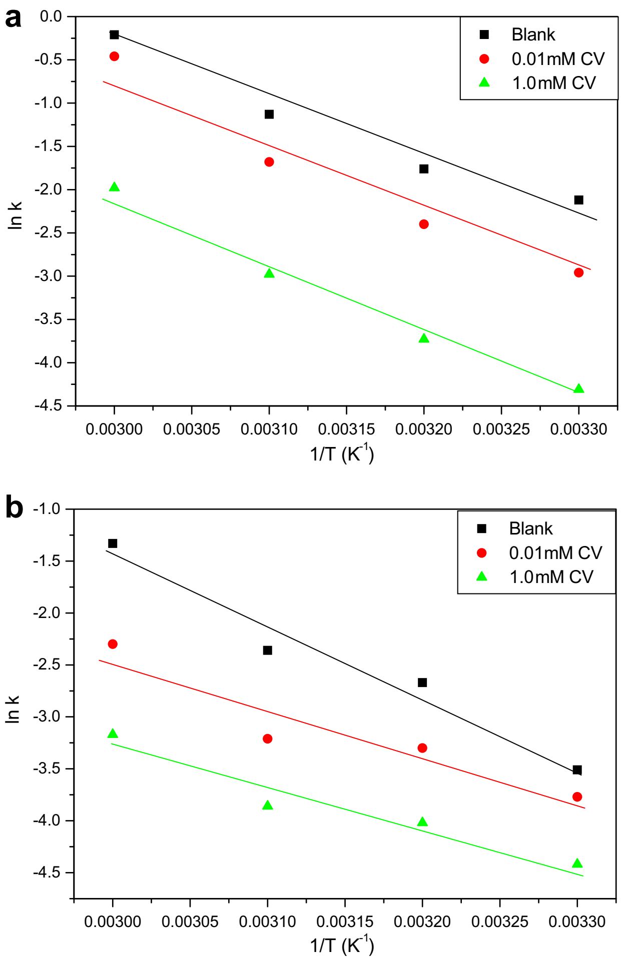

Fig. 9. Arrhenius plots for mild steel corrosion in 0.5 M H_2SO_4 and 1 M HCl without and with HPMC.

Table 2
Calculated values of activation energy (E_a), adsorption heat (Q_{ads}), activation enthalpy (ΔH^*) and activation entropy (ΔS^*) for mild steel corrosion in 0.5 M H_2SO_4 and 1 M HCl without and with HPMC

[HPMC] (mM)	E_a (kJ mol^{-1})	Q_{ads} (kJ mol^{-1})	ΔH^* (kJ mol^{-1})	ΔS^* (J mol^{-1}K^{-1})
0.5 M H_2SO_4				
0.00	52.9	–	50.3	−97.8
0.01	68.3	−39.0	65.6	−54.4
1.00	64.4	−11.3	61.4	−78.9
1 M HCl				
0.00	57.0	–	54.2	−95.2
0.01	37.4	46.1	34.9	−161.5
1.00	32.5	33.1	29.9	−183.3

Thus an improvement of η% on addition of KI is an indication of the participation of protonated inhibitor species in the adsorption process. Table 1 illustrates the effect of different concentrations of KI (10^{-8} M and 10^{-6} M) on the corrosion of mild steel in 0.5 M H_2SO_4 and 1 M HCl without and with 0.01 mM HPMC. More iodide ions are adsorbed on the metal surface from 0.5 M H_2SO_4 since the steel surface presents a more positive surface in this solution. In addition, electrostatic repulsions between Cl^- and I^- ions will impede iodide ion adsorption from HCl. Nevertheless, η% of HMPC in both corrodents is significantly improved in the presence of iodide ions, thus confirming the participation of protonated species in the corrosion inhibition and adsorption process. Consequently the overall adsorption process involves both physisorption of protonated species on cathodic sites and chemisorption of molecular species on anodic sites, though to varying extents.

3.3. Effect of temperature

To assess the effect of temperature on corrosion and corrosion inhibition processes, gravimetric experiments were performed at 10 K intervals in the temperature range 303–333 K in uninhibited acid (0.5 M H_2SO_4 and 1 M HCl) and in inhibited solutions containing 0.01 mM and 1.0 mM HMPC. The results obtained for a 2 h immersion period are given in Fig. 8 and show that corrosion rates in both uninhibited and inhibited acids increased with rise in temperature. The relationship between the corrosion rate (k) of mild steel in acidic media and temperature (T) is often expressed by the Arrhenius equation

$$k = A\ \exp(-E_a/RT) \qquad (10)(8)$$

where E_a is the effective activation energy, A the preexponential factor and R the universal gas constant. Corrosion rate increases exponentially with temperature because the hydrogen evolution overpotential decreases. The variation of logarithm of corrosion rate with reciprocal of absolute temperature is shown in Fig. 9 for 0.5 M H_2SO_4 and 1 M HCl, respectively in absence and presence of inhibitor. The calculated values of E_a are given in Table 2. Addition of CV is seen to increase the apparent activation energy for the corrosion reaction in 0.5 M H_2SO_4 but reduced that in 1 M HCl. Fig. 10 illustrates the change of inhibition efficiency with temperature variation. As temperature is raised, η% decreased steadily in 0.5 M H_2SO_4 and increased in 1 M HCl. The observed trend can be explained as follows; the increased E_a values obtained on addition of HMPC to 0.5 M H_2SO_4 including the decrease in η% with rise in

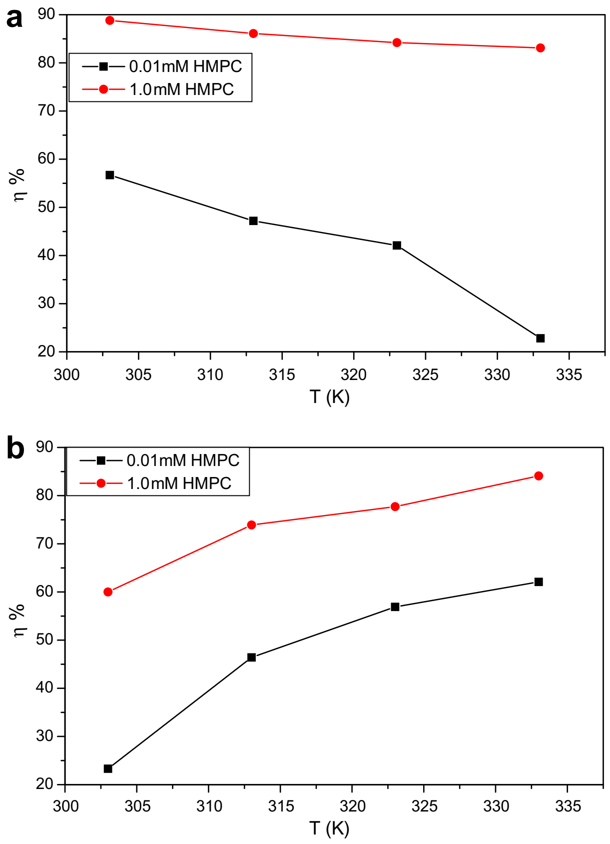

Fig. 10. Effect of temperature on the inhibition efficiency of HMPC.

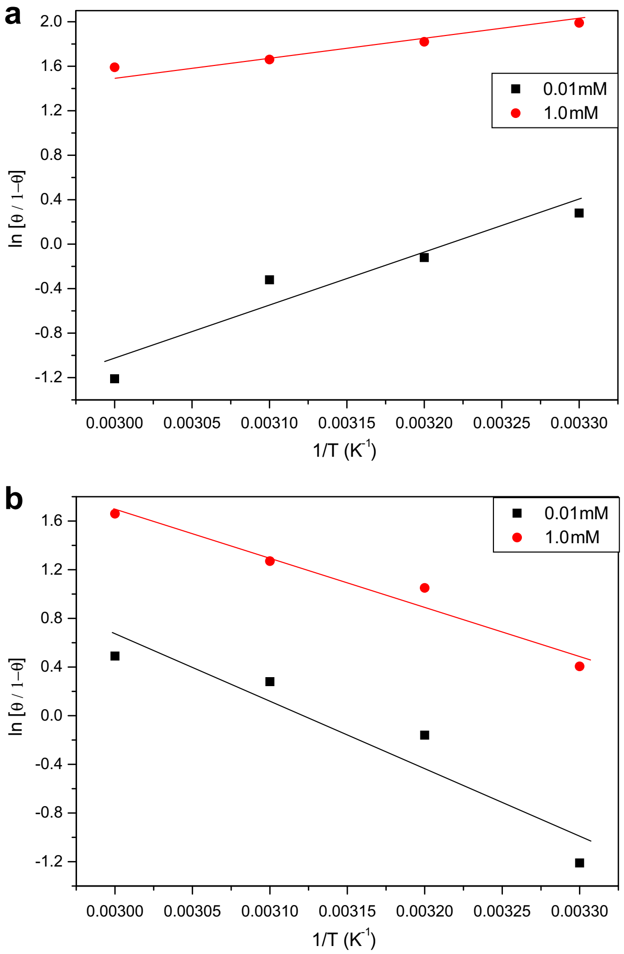

Fig. 11. Plots of ln [$\theta/1-\theta$] vs. $1/T$ at different concentrations of HMPC.

temperature suggests that the adsorption–desorption equilibrium is shifted towards desorption with increasing temperature, indicating a physical adsorption mechanism. At high concentration however, the amount of adsorbed inhibitor is somewhat high so a relatively high degree of surface coverage is maintained even at high temperature. In 1 M HCl η% increased with rise in temperature as well as lowered E_a in the presence of HMPC suggests likely specific interaction between CV and the mild steel surface. Such behavior has been attributed to a modification in the nature of adsorption, whereby the inhibitor is physically adsorbed at low temperature, whereas chemisorption is favored at higher temperatures. Similar interpretations can be found in the literature [5,6].

The values of the heat of adsorption were evaluated from the kinetic thermodynamic model [25]

$$\left[\frac{\theta}{1-\theta}\right] = A\cdot C\cdot \exp(-Q_{ads}/RT) \qquad (9)$$

where A is a constant, C is the inhibitor concentration, θ is the occupied and ($1-\theta$) is the vacant site not occupied by inhibitor. Plots of ln [$\theta/(1-\theta)$] against $1/T$ for two concentrations of HMPC (0.01 mM and 1.0 mM) are shown in Fig. 11. The plots also depict the different characters of adsorption of HMPC from 0.5 M H_2SO_4 (Fig. 11a) and 1 M HCl (Fig. 11b). The calculated values of Q_{ads} are given in Table 2. Negative values indicate that inhibitor adsorption and hence inhibition efficiency decreases with rise in temperature, while positive values mean the opposite effect.

Some other activation parameters such as the enthalpy change of activation (ΔH^*) and entropy change of activation (ΔS^*) were obtained from the Eyring transition state equation

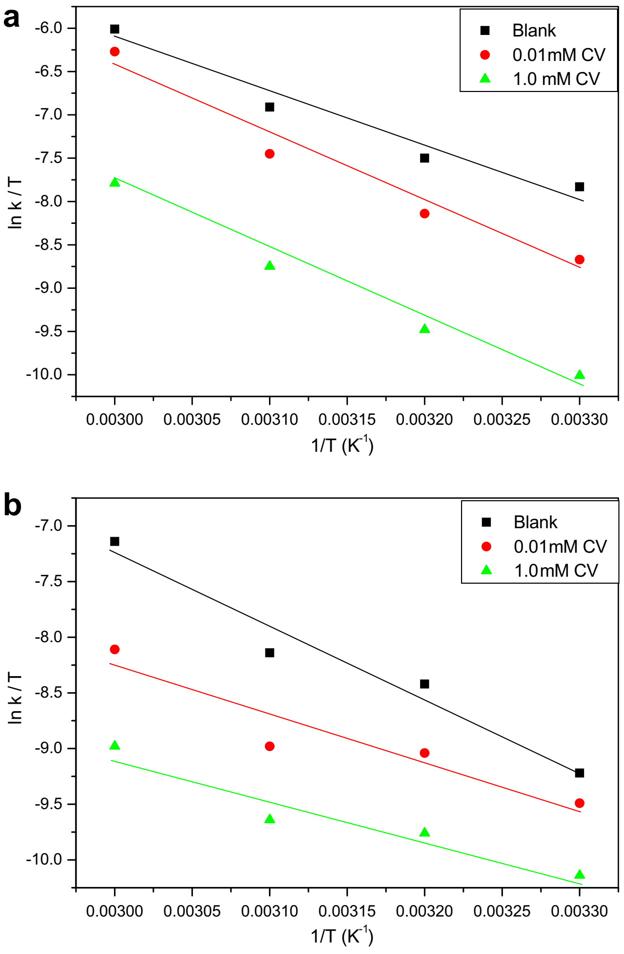

Fig. 12. Eyring plots for mild steel corrosion in 0.5 M H_2SO_4 and 1 M HCl without and with HPMC.

$$\ln\left(\frac{k}{T}\right) = -\left(\frac{\Delta H^*}{RT}\right) + \ln\left(\frac{R}{N_A h}\right) + \left(\frac{\Delta S^*}{R}\right) \qquad (10)$$

where N_A is the Avogadro number, h is Planck's constant and other terms retain their previous meanings. Fig. 12 shows plots of ln (k/T) versus 1/T obtained in 0.5 M H_2SO_4 (Fig. 12a) and 1 M HCl (Fig. 12b) without and with HMPC give straight lines with slope $-\Delta H^*/R$and intercept [$\ln(R/N_A h) + \Delta S^*/R$]. The calculated values of ΔH^* and ΔS^* obtained from these plots are given in Table 2. The positive values of ΔH^* both in absence and presence of inhibitor reflect the endothermic nature of the steel dissolution process. It is also clear that the activation enthalpies vary in the same manner as the activation energies, supporting the proposed inhibition mechanisms. Large and negative values of entropies imply that the activated complex in the rate determining step represents an association rather than a dissociation step, meaning that a decrease in disordering takes place on going from reactants to the activated complex.

4. Conclusions

HMPC was found to be an inhibitor for mild steel corrosion in 0.5 M H_2SO_4 and 1 M HCl solutions and its inhibition efficiency increased with increasing concentration. The corrosion process is inhibited by the adsorption of HMPC on the metal surface following the Temkin isotherm and a modified Langmuir isotherm. The inhibiting action is attributed to general adsorption of protonated and molecular species on cathodic and anodic sites on the corroding metal surface. Inhibition efficiency of HMPC in 1 M HCl increased with rise in temperature and addition of HMPC leads to a decrease in corrosion activation energy. Corrosion inhibition here is due to the formation of a chemisorbed film on the metal surface. However, inhibition efficiency of HMPC in 0.5 M H_2SO_4 decreased with rise in temperature and activation energy increased in the presence of HMPC. The activation parameters such as activation enthalpy (ΔH^*) and activation entropy (ΔS^*) as well as the adsorption heat (Q_{ads}) were calculated and corroborate the proposed inhibition mechanisms.

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