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The Counter Ion Influence of Cationic Surfactant and Role of Chloride Ion Synergism on Corrosion Inhibition of Mild Steel in Acidic Media

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The corrosion inhibition of mild steel in 0.5 M H_2SO_4 solution by cationic surfactants, cetylpyridinium chloride (CPC) and cetylpyridinium bromide (CPBr) was studied by potentiodynamic polarization curves. The results showed that the counter ions of these surfactants, i.e., chloride and bromide, and the addition of chloride ion to any of the surfactants significantly influenced the protection efficiency (P_{icor}) and mode of inhibition. Surfactants alone and in combination with chloride ions were found to obey Temkin adsorption isotherm. Chemical adsorption was proposed from the obtained thermodynamic parameters such as the free energy of adsorption, ΔG^{o}_{ads}. The synergism parameter (S_{Θ}), as a quantitative estimation of the interaction of surfactants and chloride, was found to be greater than unity indicating that the enhanced protection efficiency caused by the addition of chloride ions to the surfactants is due to a co-operative adsorption of both species. The experimental data and the extracted thermodynamic parameters confirmed the role of the counter ions (Cl^- and Br^-) which has been confirmed by the addition of Cl^- ions as additive for synergism effects.

Keywords: Cationic, surfactant, corrosion, inhibition, synergism.

1. INTRODUCTION

Corrosion of metals plays a crucial role due to its dual impacts both for economy and safety. Various types of steels are a major construction materials and extensively used throughout industry (chemical and electrochemical industries, medical, nuclear, petroleum, power, and food production), and almost in everyday life. However, it suffers from a certain type of corrosion within some environments [1]. Most commercial acid-inhibitors are organic compounds containing heteroatoms such as nitrogen, oxygen, sulphur, and phosphorous atoms. Inhibitors adsorb on the surface of the metal, thus resulting in an adsorption-film acting as a barrier between the metal surface and the corrosive medium, and blocking the active sites [1]. Surfactants either ionic or non-ionic [2-12] have been reported as corrosion inhibitors for various metals, such as copper, aluminium, and mild-steel [5-12].

Several mechanisms have been proposed for the inhibition of metal corrosion by organic inhibitors. Nowadays, researches in this field focus on the relationship between inhibition efficiency and molecular structures of organic inhibitors, especially the adsorption orientation of the inhibitors on the metal surface [13–15]. Generally, it has been assumed that the first stage in the mechanism of corrosion inhibition in aggressive acid media is based on its adsorption of an inhibitor on the metal surface. The process of adsorption of inhibitors are influenced by the nature of the metal surface of the metal, the chemical structure of the organic inhibitor, the distribution of charge in the molecule, the type of aggressive electrolyte and the type of interaction between organic molecules and the metallic surface [14, 16, 17].

It has been reported that quaternary ammonium inhibitors are effective in hydrochloric and sulfuric acids. The effect of concentration, functional groups and halide ions (counter ions) of quaternary ammonium inhibitors on the corrosion of iron and steel have been studied extensively [16,18-21]. On the other hand, the inhibitive effect can be enlarged by adding halide ions to the solution where it is pre-adsorbed and thus enhances the adsorption of positively charged cationic surfactants. Saleh et al [22,23] have reported that the cetylpyridinium chloride and hexadecylpyridinium bromide showed high inhibition efficiency for the corrosion of low carbon steel. In the present work, the comparison effect of the counter ion of surfactant on its protection efficiency of mild steel in 0.5 M sulfuric acid and synergism with chloride ions is investigated by electrochemical methods and the plausible synergistic mechanism has been proposed.

2. EXPERIMENTAL

Mild steel samples have the following composition (wt. %); 0.07% C, 0.29% Mn, 0.07% Si, 0.012% S, 0.021 % P and the remainder iron. Three studied surfactants cetylpyridinium chloride (CPC) and cetylpyridinium bromide (CPBr), (structures shown in Fig. 1) and sodium chloride were obtained from Aldrich and used as received. The solution of 0.5 M H_2SO_4 was prepared by dilution of AR grade 96% H_2SO_4. Stock solutions of surfactants and halides were prepared in 0.5 M H_2SO_4 and the desired concentrations were obtained by appropriate dilution.

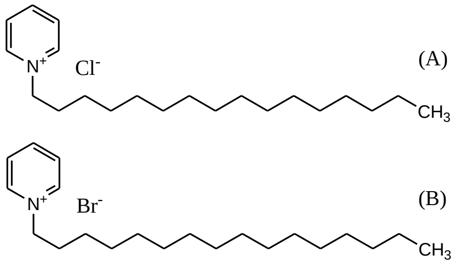


Figure 1. Molecular structures of (A) CPC and (B) CPBr.

Electrochemical measurements were carried out in a conventional three-electrode cell with a platinum counter electrode (CE) and a Hg/Hg_2SO_4/ (1.0 M) SO_4^{2-} coupled to a fine Luggin capillary as the reference electrode (RE). In order to minimize the ohmic contribution, the Luggin capillary was kept close enough to the working electrode (WE). The latter was fitted into a glass tube of proper internal diameter by using epoxy resins. The WE surface area of 0.5 cm^2 was abraded with emery paper (grade 320–500–800-1000-1200) on test face, rinsed with distilled water, degreased with acetone, and dried with a cold air stream. Before measurements the electrode was immersed in the test solution at open circuit potential (OCP) for 15 min at 25 ^oC or until the steady state is obtained. All electrochemical measurements were carried out using an EG&G Princeton Applied Research (model 273A) potentiostat/galvanostat controlled by m352 electrochemical analysis software. The potential of potentiodynamic polarization curves was done starting from a cathodic potential to anodic potential with scan rate of 2 mV/s. Current densities were calculated on the basis of the apparent geometrical surface area of the electrode. The measurements were repeated at least three times to test the reproducibility of the results.

3. RESULTS AND DISCUSSION

3.1. Open circuit potential measurements

The variation of the open circuit potential (OCP) of mild steel was followed as a function of time in (curve 1) 0.5 M H_2SO_4 solution containing 2x10^{-5} M of (curve 2) CPBr or (curve 3) CPC and the data are shown in Fig. 2**.**

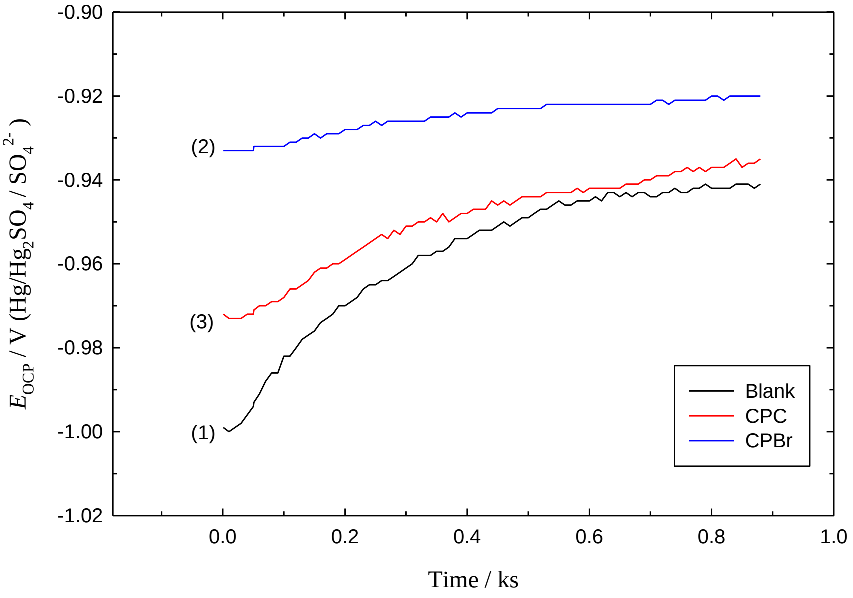
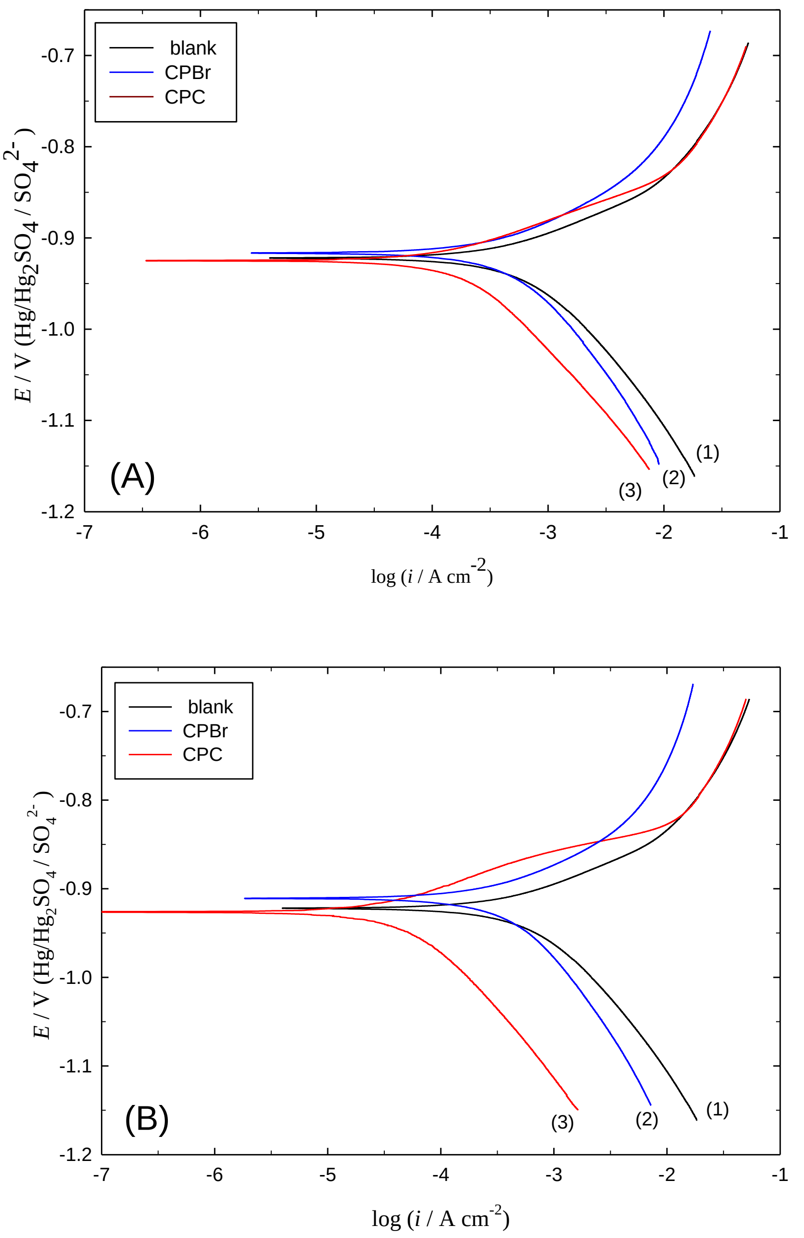


Figure 2. OCP-time curves for mild steel immersed in 0.5 M H_2SO_4 in the presence and absence of 2x10^{-5} M of (2) CPBr or (3) CPC.

In the absence of surfactant (curve 1), the OCP changed quickly towards more positive values, indicating the initial dissolution process of the air formed oxide film and the attack on the bare metal [24,25]. A steady potential corresponding to the free corrosion potential E_{cor} of the metal was readily attained in the case of CPBr than in the case of CPC [26]. This indicates that the counter ion of the two surfactants plays an important role in the adsorption process. Addition of both surfactants (curves 2 and 3) drifts the steady state E_{cor} to more positive values without changing the general shape of the OCP - time plots.

The classification of a compound as an anodic or cathodic type inhibitor is feasible when the OCP displacement is at least ± 85 mV in relation to that one measured for the blank solution [27]. However from Fig. 2 the positive shift in E_{cor}, in the presence of any of the two surfactants is less than 30 mV revealing that the tested inhibitors affect both the anodic dissolution of iron and the hydrogen evolution reaction.

3.2. Polarization studies of CPC and CPBr.



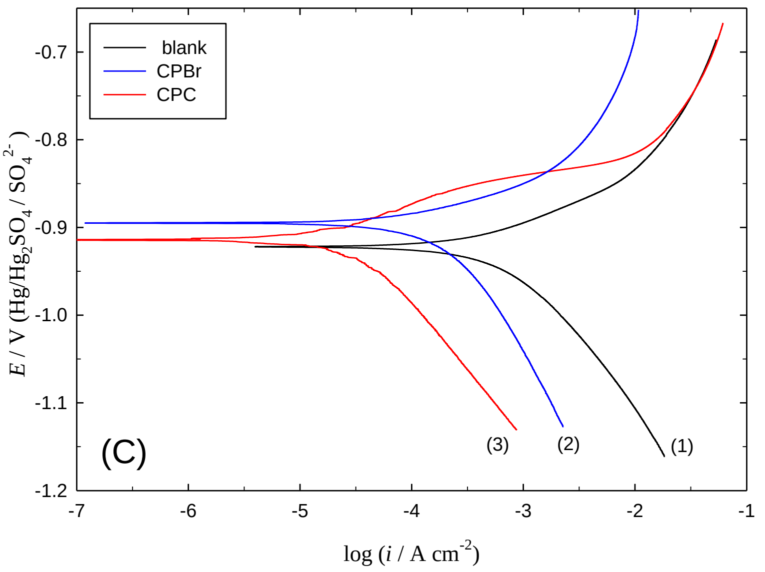


Figure 3. Polarization curves obtained at mild steel electrode in (1) 0.5 M H_2SO_4 solution containing (A) 10^{-5}, (B) $2x10^{-5}$, (C) $7x10^{-5}$ M of (2) CPBr and (3) CPC.

Different parameters such as the free corrosion potential (E_{cor}), corrosion current density (i_{cor}), slope of the cathodic branch (β_c) and slope of the anodic branch (β_a) were obtained and listed in Table 1**.** The cathodic Tafel slope (β_c) for mild steel in the absence and presence of surfactants does not change significantly indicating that the two surfactants do not change the mechanism of the HER and the corrosion is rather inhibited by blocking of the iron surface by simple adsorption process.

Table 1. Electrochemical parameters for the corrosion of mild steel obtained in 0.5 M H_2SO_4 in the absence and presence of $2x10^{-5}$ M CPC or CPBr.

System	E_{cor} / V	i_{cor} / mA cm^{-2}	βa / V $(decade)^{-1}$	βc / V $(decade)^{-1}$
Blank	-0.923	0.969	0.219	0.188
CPC	-0.926	0.330	0.224	0.192
CPBr	-0.911	0.459	0.294	0.198

As shown in Table 1 the maximum shift in E_{cor} in the presence of inhibitors is less than 85.0 mV. According to [27] it is possible to classify certain inhibitor as cathodic or anodic type if the displacement in E_{cor} (inhibitor) is at least 85 mV with respect to E_{cor} (blank). It is evident from the present results that two surfactants could be classified as a mixed type inhibitor but in the case of CPC with a predominant effect on the cathodic reaction, consistently with OCP-time curves shown in Fig. 2.

3.3 Polarization studies in presence of chloride ions.

Figure 4 shows the polarization curves for mild steel in blank, 0.5 M H_2SO_4 (curve 1), blank containing 7x10^{-6} M of surfactant (curves 2), 0.10 M NaCl (curves 3) and 7x10^{-6} M of surfactant + 0.10 M NaCl (curves 4) where panel (A) and (B) for CPC and CPBr, respectively.

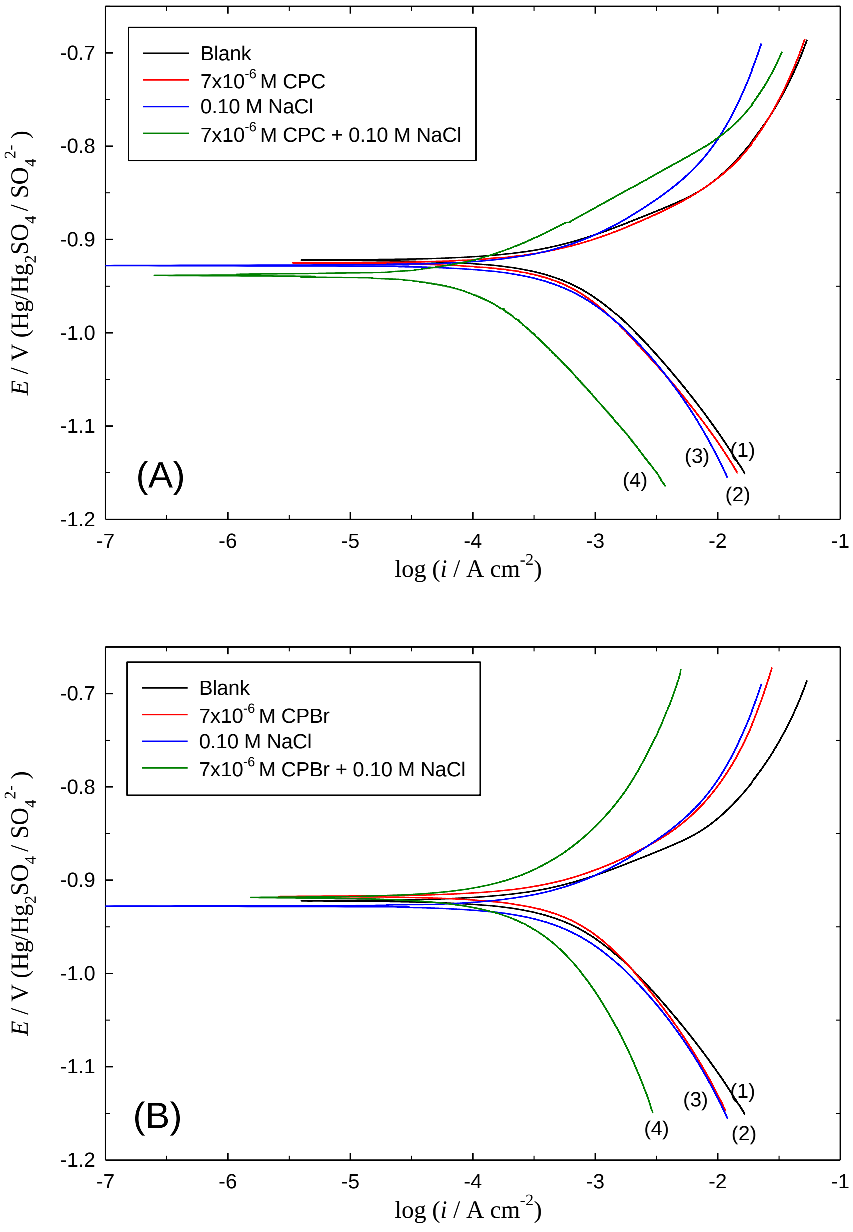


Figure 4. Polarization curves obtained at mild steel electrode in (1) 0.5 M H_2SO_4 containing (2) 7x10^{-6} M of (A) CPC or (B) CPBr (3) 0.10 M NaCl, and (4) 0.1 M NaCl and 7x10^{-6} M of (A) CPC or (B) CPBr.

It is clear that the mild steel corrosion is slightly inhibited in the presence of either 7x10^{-6} M surfactant (curves 2) or 0.10 M NaCl (curves 3). In other words, both cathodic and anodic reactions of mild steel electrode are drastically retarded by chlorides. This could be ascribed to adsorption of halide over the corroded surface [28]. In the presence of a combination of surfactants and chloride ions (curves 4), however, both anodic and cathodic branches are dramatically shifted to lower currents. Interestingly both branches are inhibited in contrary to the use of any species alone. This shows that the chloride ions improve the protective action of studied surfactants, especially at higher anodic potentials, by retarding anodic dissolution of iron, and the mixture of halide and any of studied surfactants acts as a mixed type inhibitor.

3.4. Protection efficiency

Polarization curves of mild steel were measured in 0.5 M H_2SO_4 in the presence of different concentrations of the two surfactants (data are not shown). The protection efficiencies extracted from these results are shown in Fig. 5 in which the dependence of the protection efficiency on the concentration of the two surfactants are represented.

The protection efficiency (P_{icor}) is given by;

$$P_{\mathrm{icor}} = \left(1 - \frac{i_{\mathrm{cor2}}}{i_{\mathrm{cor1}}}\right) x\ 100 \qquad (1)$$

where i_{cor1} and i_{cor2} are corrosion current densities in the absence and presence of inhibitor, respectively. As the concentration increases the protection efficiency increases until it reaches a constant value at a certain concentration which corresponds to the critical micelle concentration (CMC) (about >5x10^{-4} M along two surfactants) [29, 30].

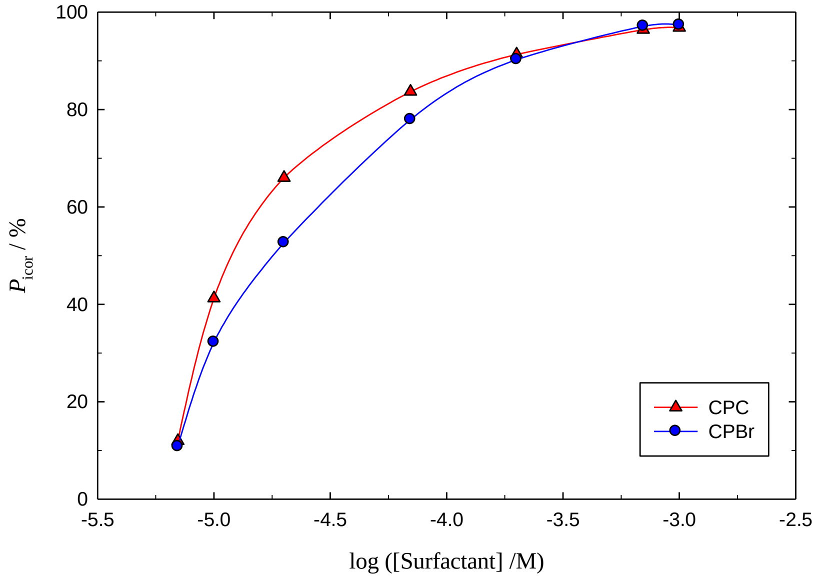


Figure 5. Effects of surfactant concentration on P_{icor} obtained at mild steel in 0.5 M H_2SO_4.

The inhibition is attributed to the adsorption of the surfactant on the iron surface. At [surfactant] about10^{-5} M, the molecules of surfactant adsorb at the surface individually with a low percent coverage. As the concentration increases (in the range $7x10^{-5}$ M ≈ CMC) the amount adsorbed increases leading to a higher degree of coverage and consequently higher corrosion inhibition. Adsorption is enhanced due to the inter hydrophobic chain interaction. Such interaction assists the formation of a thin film of surfactant molecules at the iron surface. This film is of hydrophobic nature due to anchoring of the hydrophobic chains into the solution. At higher inhibitor concentration near the CMC a plateau is obtained. This may be attributed to the formation of a bimolecular layer of surfactant through the interaction of the hydrocarbon chains by tail-tail orientation at the electrode–electrolyte interface [31, 32].

Similar polarization curves in the presence of constant concentration of the two surfactants, i.e., $7x10^{-6}$ M, coexisting with different concentrations of the chloride ions, i.e., in the range 10^{-6} to 1.0 M were measured and the protection efficiency extracted for these results are shown in Fig. 6.

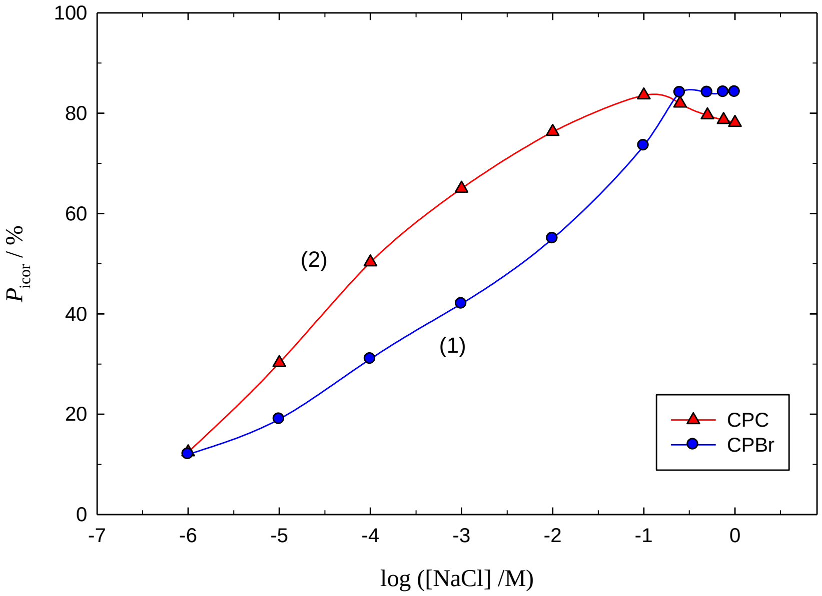


Figure 6. Effects of NaCl concentrations on P_{icor} obtained at mild steel in in 0.5 M H_2SO_4 containing $7x10^{-6}$ M (1) CPBr and (2) CPC.

Inspection of this figure reveals several interesting features;

1- The protection efficiency increases with the increase in the chloride concentration reaching a constant value around 84 % in the presence of 0.10 M NaCl.

2- When chloride ions concentration increases above 0.10 M the protection efficiency decreases in the case of CPC, but stays constant in the case of CPBr. Cl^- could be specifically adsorbed on free anodic sites in the case of CPC as represented by the following equations:

$$\mathrm{Fe} + \mathrm{Cl}^- \rightleftharpoons \mathrm{FeCl}^-_{ads} \qquad (2)$$

$$\mathrm{FeCl}^-_{ads} + \mathrm{Cl}^- \longrightarrow \mathrm{FeCl}_2^+ + 3e^- \qquad (3)$$

This leads to the formation of a soluble complex (FeCl_2^+) at high concentrations of the chloride so protection efficiency decreases.

3.5. Synergism

Inhibition action of organic inhibitors depends mainly on their adsorption behavior on the metal surface, which depends on the counter ions of the organic compounds, surface charge density and potential of zero charge of metals. It is likely that the adsorption of a cationic surfactant is enhanced by increasing the negative charge density on the metal surface. Thus the pre-adsorption of a halide could enhance the adsorption of the cationic surfactant due to ion–pair interactions between the CPC and/or CPBr molecules and the halide ions, resulting in what is the so-called inhibition synergism. In this part the effect of chloride concentration on the inhibition efficiency of CPC and CPBr is studied. This interaction can be quantized by a parameter called synergism parameter (S_Θ) [33, 34] which is defined as:

$$S_\theta = \frac{1-\theta_{1+2}}{1-\theta'_{1+2}} \qquad (4)$$

where $\theta_{1+2} = (\theta_1 + \theta_2) - (\theta_1\, \theta_2)$, θ_1 and θ_2 are the degrees of surface coverage in the presence of chloride and CPC, respectively, and θ'_{1+2} is the degree of surface coverage in presence of both species. Note that the degree of surface coverage θ values calculated from the polarization data ($\theta = P_{icor}\ \% /100$)). S_θ approaches unity when no interaction between the inhibitor molecules exists, while $S_\theta > 1$ indicates a synergistic effect as a result of a co-operative adsorption. In case of $S_\theta < 1$, antagonistic behavior prevails due to a competitive adsorption [35].

Figure 7 shows the S_Θ values estimated in the presence of 0.5 M H_2SO_4 containing 7×10^{-6} M of the two surfactants each in the presence of different concentrations of chloride ions. S_θ increases with the Cl^- concentration up to a maximum values before it decreases at some value of the Cl^- concentration depending on the surfactant. The values are found to be more than unity, suggesting the synergistic action of chloride anions and cationic surfactants. The above results reveal that small concentration of both CPC and CPBr can act as an effective inhibitor in 0.5 M H_2SO_4 solution in the presence of chloride ions. In the present case E_{cor} equals -512 mV vs. SCE of iron and in view of the fact that the potential of zero charge for iron in H_2SO_4 -550 mV vs. SCE [36], it is expected that the steel surface is positively charged. Since the anions of Cl^- and SO_4^{2-} could be specifically adsorbed, they create an excess negative charge exposed to the solution and favor more adsorption of the cations [37,38] and the electrostatic influence on the inhibitor adsorption may be the reason for an increased protective effect in halide-containing solution [39]. As the halide ions are chemisorbed on anodically polarized metal surface (positively charged surface) it favors the consecutive adsorption of surfactant cations.

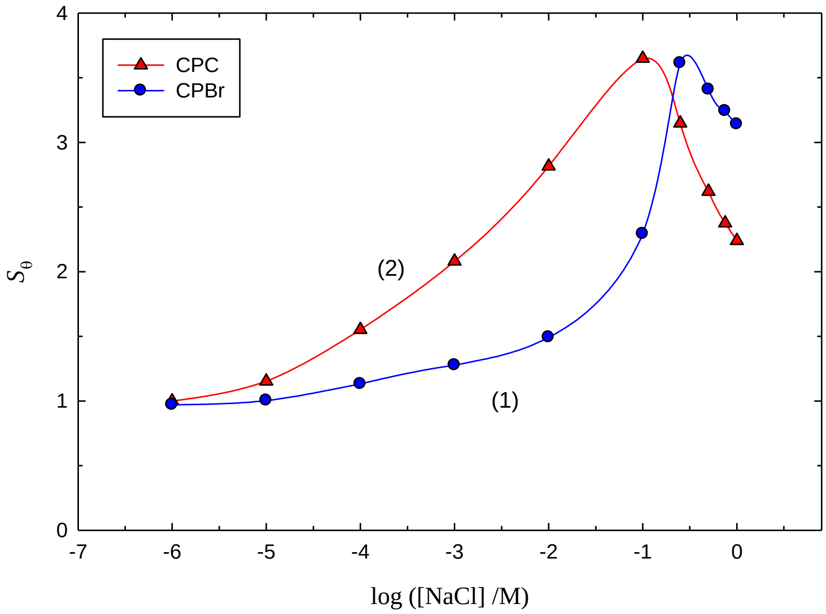


Figure 7. Effects of NaCl concentrations on the synergism parameter S_Θ obtained at mild steel in the presence of 7x10^{-6} M of (1) CPBr or (2) CPC in 0.5 M H_2SO_4.

The following points can be extracted from the above figure:

(a) In the case of CPC, high concentrations of chloride might give rise to weak combination of anions and CPC cations due to the formation of a soluble product, and thus S_Θ and P_{icor} decreases with the increase in the concentration of the Cl^- [40].

(b) The synergism parameter S_Θ in the case of CPBr is generally smaller than that in case of the CPC, this may be due to adsorption of CPBr molecules via nitrogen atom on the anodic sites of the steel surface might compete with halide ions for active sites on the metal surface leading to lesser synergistic effect [41].

(c) Generally, at low concentrations of NaCl S_Θ in the case of CPC is greater than in the case of CPBr, consistently with the results of the protection efficiency (see Fig. 6).

3.6. Adsorption isotherms

Adsorption isotherms can provide basic information on the interaction between the inhibitor and mild steel surface. Attempts were made to fit experimental data to various isotherms including Frumkin, Langmuir, Temkin, Freundlich, Bockris- Swinkels, and Flory–Huggins isotherms. It has been found that the experimental results in this study for both CPC and CPBr alone and with chloride systems accord with Temkin isotherm (Eq. 5) [42-45] and the plots are presented in Fig. 8 for the surfactant alone (A) and the surfactant/NaCl (B) systems:

$$exp(-2a\Theta) = KC \tag{5}$$

where Θ is the degree of surface coverage (determined from the polarization data as shown above), C the inhibitor concentration, a the molecular interaction parameter and K the equilibrium constant of the adsorption process.

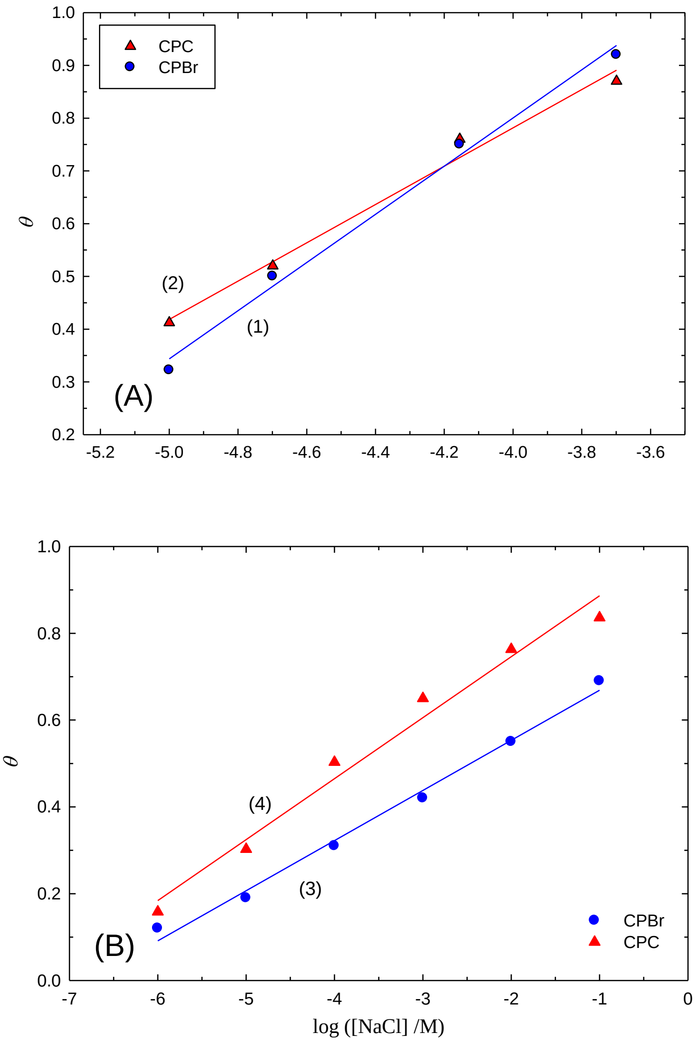


Figure 8. Temkin adsorption isotherm obtained for (1) CPBr, (2) CPC, (3) CPBr/NaCl system and (4) CPC/NaCl system on mild steel in 0.5 M H_2SO_4.

The validity of Temkin's isotherm favors the assumption of a chemisorptive bond between the metal and the inhibitor molecules [46], i.e., the unshared electron pairs in nitrogen atom and/or pi electron of pyridinum ring of both CPC and CPBr could interact with d-orbitals of iron to provide a protective chemisorbed film. The adsorption parameters deduced form Temkin adsorption isotherms are listed in Table 2. It is seen that the values of *a* in the case of the surfactant only and with their combination with chloride ions are negative indicating that repulsion exists in the adsorption layer. It is generally known that *K* denotes the strength between the adsorbate and adsorbent. Large values of *K* imply more efficient adsorption and hence better protection efficiency.

The standard adsorption free energy (ΔG^{o}_{ads}) was estimated using the following well known equation [47, 48]:

$$K = \frac{1}{55.5} exp\left(\frac{-\Delta G^{0}}{RT}\right) \tag{6}$$

The ΔG^{o}_{ads} values obtained are more negative than 40 kJ/mol in both surfactants, which means that both physical adsorption and chemical adsorption would take place. Generally, values of ΔG^{o}_{ads} up to 20 kJ/mol are consistent with the electrostatic interaction between the charged molecules and the charged metal (physical adsorption) while those more negative than 40 kJ/mol involve sharing or transfer of electrons from the inhibitor molecules to the metal surface to form a co-ordinate type of bond (chemisorptions) [49,50], consistently with the results of protection efficiency.

Table 2. Thermodynamic parameters and other parameters estimated from fitting Temkin isotherm as given in Fig. 8.

System	*a*	*K*	R^2	ΔG^{0}_{ads} / kJ mol^{-1}
CPC	-4.10	3.56E+06	0.9942	-47.34
CPBr	-2.5	5.67E+05	0.9967	-42.96
CPC/NaCl	-7.92	1.40E+07	0.9994	-50.75
CPBr/NaCl	-9.97	6.21E+06	0.9946	-48.72

The values of *K* and ΔG^{o}_{ads} are consistent with the above results of P_{icor} and S_{Θ}. For instance, ΔG^{o}_{ads} in case of CPC is more –ve than that of CPBr and ΔG^{o}_{ads} of (CPC+Cl^-) is more –ve than that of CPC alone. That is to say CPC has better inhibition action than CPBr. This might be due to the specific adsorption of the Cl- which facilitates the subsequent adsorption of the surfactant cation (CP^+) and thus this step offers the required environment for further chemical adsorption.

3.7. Adsorption Mechanism

The adsorption of inhibitor on steel/solution interface is affected by the chemical structure of inhibitor, the nature and charged surface of the metal. In the present systems, based on the chemical structure of CPC and CPBr, both have several possible active sites for the adsorption process. CPC and CPBr can be classified as an electrolyte, namely, the organic part (pyridinium) which is the cation, and the inorganic part (Cl^- and Br^- for CPC and CPBr, respectively), which is the anion. Accordingly, both surfactants exerts its inhibition action by adsorption of the cation part on the steel surface through that hydrophilic part, i.e., pyridinium ring (the polar or ionic group) attacks the metal surface while the hydrophobic part $C_{16}H_{33}$– extends to solution face to form a hydrophobic barrier to decrease the corrosion rate.

In addition to the above, CPC and CPBr may adsorb on the metal surface via the chemisorption mechanism, a coordinate bond may be formed between the lone electrons pairs of N atom and the empty d-orbitals of Fe atoms. Also pyridinium ring has plentiful pi-electrons owing to aromatic ring; they can be also adsorbed on the metal surface on the basis of donor–acceptor interactions between pi-electrons of the pyridinium ring and vacant d-orbitals of Fe. Thus a co-operative adsorption exists between halides and CPC or CPBr dominates over competitive adsorption. According to co-operative mechanism, chloride ions are initially adsorbed on anode of metal surface and then surfactant cation is adsorbed on the layer of halide ions by coulombic attraction forming ion pairs on mild steel surface [51]. This last step is followed by chemical adsorption as evident from the relatively high –*ve* values of ΔG^o_{ads}.

4. CONCLUSIONS

CPC and CPBr inhibitors with the same head and alkyl chain but with different counter ion were compared for their inhibition action on the corrosion of steel in 0.5 M H_2SO_4. CPC mainly acts as a cathodic-type inhibitor while CPBr acts as a mixed type inhibitor. The addition of chlorides enhances the protection action of the two surfactants and both of them act as a mixed-type inhibitor. The values of S_Θ (synergistic parameter) are greater than unity showing that the corrosion inhibition brought about by surfactants in combination with chloride ions is synergistic in nature and co-operative adsorption between the halides and surfactants prevails over competitive adsorption. Adsorption of the used cationic surfactants on the mild steel surface, either in the presence or absence of halides, obeys Temkin adsorption isotherm. The extracted thermodynamic parameters confirmed the synergism and the above obtained conclusions.

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