

Role of Citrate Ions in the Phosphonate-based Inhibitor System for Mild Steel in Aqueous Chloride Media

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ABSTRACT

The corrosion inhibition efficiency of phosphonic acid and its derivatives for the inhibition of corrosion of mild steel in neutral chloride media is decided by its ability to form protective film over the surface. In this context, the effect of addition of metal cations and certain organic compounds, such as citrate in conjugation with phosphonic acid to impart synergistic corrosion inhibition has been explored. The experiments were carried out using various concentrations of trisodium citrate and zinc ions in an aqueous solution of 2-carboxyethyl phosphonic acid (2CEPA) at 25 ppm. The corrosion characteristics have been determined using electrochemical impedance spectroscopy together with determination of corrosion rate by weight-loss method. It has been observed that a combination of inhibitive ions, namely citrate, 2CEPA, and zinc ions at 25 ppm gives 96 per cent inhibition efficiency and this corrosion inhibition is due to the formation of a protective film. By increasing the concentration of citrate beyond 25 ppm, the corrosion inhibition efficiency decreases. This paper discusses the role of citrate and zinc ions in imparting added corrosion inhibition ability using 2CEPA on the basis of experimental results.

Keywords: Corrosion inhibition, phosphonic acid, surface coating, corrosion inhibitors, trisodium citrate, electrochemical inhibition spectroscopy, EIS, 2CEPA, weight-loss method, 2-carboxyethyl phosphonic acid

1. INTRODUCTION

The phosphonic acid and its derivatives are effective corrosion inhibitors for mild steel in neutral media. These derivatives form sparingly soluble compounds with the metal ions existing in the solution, which precipitate on the surface to form protective layers. Examples of organic phosphonic acids are 1-hydroxythane-1,1-diphosphonic acid (HEDP), aminotrimethylenephosphonic acid (ATMP), hydroxyphosphonoacetic acid^{1,2} (HPA), etc. The phosphonates are introduced alone or in combination with metal cations. Research in this area has been stimulated by the need to find corrosion inhibitor

formulations free of toxic chromates, at the same time, with efficiency similar to those of chromates.

The phosphonates, when blended with certain metal cations and polymers, have been reported to cause synergistic effects favourable for corrosion inhibition at the decreased inhibitor concentrations³⁻⁷. Addition of small amounts of environment-friendly organic additives to the phosphonic acid and metal cation blend has been reported to increase the corrosion inhibition of mild steels^{8,9}. Various analytical methods, such as Auger spectroscopy, radioisotope techniques¹⁰, x-ray diffraction, ESCA, etc were reported for studying the corrosion inhibitive effect

on the mild steel through adsorption studies involving various metal cations and phosphonic acids.

Understanding the inhibitor film formation and its deterioration process is important for the development of new corrosion inhibitors, having better film persistency. In this context, electrochemical impedance spectroscopy (EIS) has been successfully exploited, particularly to gain insight into the corrosion and protection mechanism involving adsorbed species^{11,12}. Information on the use of electrochemical impedance spectroscopy spectra, interpretation of the Nyquist and Bode plots in relation to corrosion inhibition/inhibitor films has been well-defined. The interpretation of present results on electrochemical impedance spectroscopy spectra are based on generally agreed explanations cited in the literature¹¹⁻¹³. The time constants derived from electrochemical impedance spectroscopy depend on the numbers of reactions taking place on the surface or various reaction rates of the same type of reactions. For example, two-time constants give rise to two types of reactions or two types of reaction rates. In this paper, an attempt has been made to bring out mechanistic features of corrosion inhibitive film formation giving special attention to the role of zinc cation and citrate anion in the formulation based on 2-carboxyethyl phosphonic acid (2CEPA).

2. EXPERIMENTAL METHODS

2.1 Materials & Surface Preparation

Solutions in triple-distilled water of 2-carboxyethyl phosphonic acid, sodium chloride, sodium citrate, and zinc sulphate were prepared using analytical-grade reagents (Aldrich). The pH of above solutions were adjusted to 7 by adding appropriate quantity of dilute sodium hydroxide. The normalised mild steel sheets of percentage composition of manganese (0.54), silicon (0.05), sulphur (0.01), phosphorous (0.01), and carbon (0.01), the remainder iron was used. Rectangular specimens of 1.0 cm*4.0 cm*0.2 cm, circular specimens of 1 cm diameter and square test specimens of 1cm*1cm were used for weight change measurements, electrochemical measurements, and surface film examination, respectively. The surfaces were polished successively using 1/0 to 6/0 emery papers and degreased with trichloroethylene and washed with triple-distilled water.

3. WEIGHT CHANGE MEASUREMENTS

The mild steel specimens in triplicate were immersed in the test solution (1000 ml) for 7 days. The loss-in-weight was determined after withdrawal of the specimens from the test solutions, washing with soap solutions and distilled water to remove surface corrosion products.

The percentage inhibition efficiency was calculated as

Percentage inhibition efficiency

$$= \frac{W_1 - W_2}{W_1} * 100$$

where W_1 and W_2 are the weight losses of mild steel in uninhibited and inhibited solutions.

4. ELECTROCHEMICAL MEASUREMENTS

A three-electrode cell assembly consisting of a mild steel as working electrode, a large area Pt mesh as counter electrode, and a saturated calomel electrode as reference electrode was used. The electrochemical impedance spectroscopy measurements at frequencies in the range 0.002–50,000 Hz were carried out using the Gamry electrochemical system. Peak-to-peak amplitude (10 mV ac) signal was used for electrochemical impedance spectroscopy measurements at $25 \pm 2^\circ\text{C}$. The mild steel electrode was placed in chloride solution (60 ppm) containing 2CEPA and zinc ions (25 ppm each) and of citrate ions (0-50 ppm) [test solution], electrochemical impedance spectroscopy measurements were taken for monitoring the inhibitor film formation and deterioration processes. The electrochemical impedance spectroscopy plots were analysed using the Gamry EIS300 electrochemical software. All the plots were analysed using two-, three- and four-time constant models.

5. RESULTS & DISCUSSIONS

The corrosion inhibition efficiencies of mild steel in chloride solution (60 ppm) containing 2CEPA, zinc ions, and citrate ions are presented in Table 1. It shows that immersion of mild steel in citrate, 2CEPA, and zinc ions separately decreases the

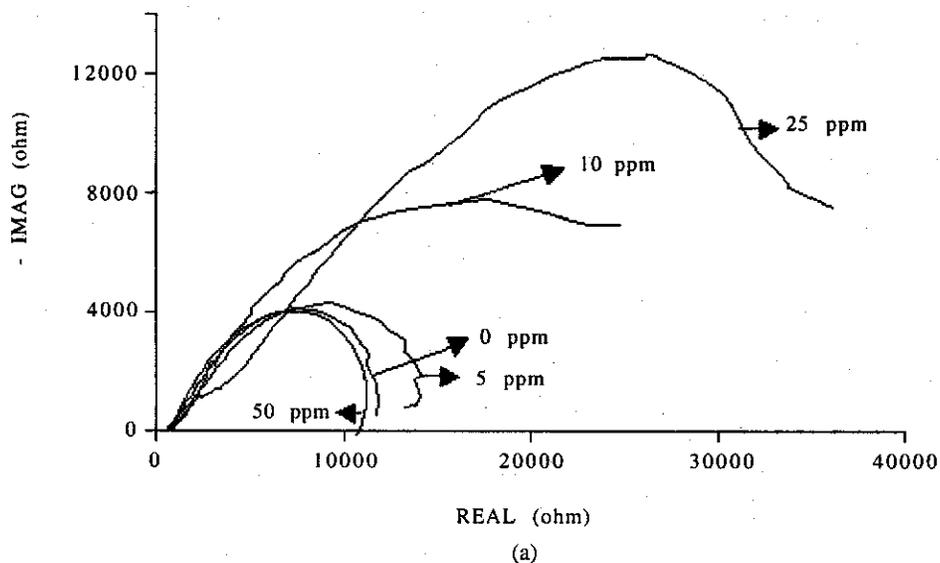
Table 1. Weight-loss data of mild steel immersed in various inhibitor concentrations for 192 h

Citrate concentration (ppm)	Corrosion inhibition efficiency (%)			
	Chloride (60 ppm)	Chloride (60 ppm) + 2CEPA (25 ppm)	Chloride (60 ppm) + zinc ions (25 ppm)	Chloride (60 ppm) + 2CEPA and zinc ions (25 ppm each).
0	---	- 33.3	- 22.2	33.3
5	- 5.6	- 22.2	- 33.3	66.7
10	- 16.7	- 22.2	- 27.8	77.8
25	- 22.2	- 33.3	- 16.7	97.8
50	- 16.7	- 50.0	8.3	93.9

corrosion inhibition efficiency. However, addition of citrate to 2CEPA and zinc ions mixture increases the corrosion inhibition efficiency of mild steel. The maximum corrosion inhibition efficiency is achieved for mild steel immersed in citrate, 2CEPA, and zinc ions (25 ppm each).

Figures 1(a) and 1(b) show electrochemical impedance spectroscopy plots (Nyquist and Bode q vs $\log f$ formats) recorded on the electrode after immersing in test solution for 0.5 h. As shown in Fig. 1(a), the diameter of the Nyquist semicircle increases with increase in the citrate concentration (except at 50 ppm). It also suggests that the presence of higher concentrations of citrate changes the corrosion kinetics on the electrode surface. The respective Bode q vs $\log f$ plots

[Fig. 1(b)] of the same data showed two clear peaks (one at the higher frequency and the other at the lower frequency) for electrode immersed in 5 ppm and 25 ppm citrate concentrations. At other concentrations, only one peak (at lower frequency) was observed. The peak at higher frequency range indicated changes at the electrode interfacial structure, and consequently, resulted in an extra time constant. The two peaks in the q vs $\log f$ plots mean that there are two major electrochemical kinetic processes on the electrode surface. In the case of Nyquist plots, two unresolved semicircles (broad semicircles) were noticed. The semicircles at the lower frequency and the other at higher frequency are due to electrochemical corrosion process and the inhibitor film formation¹³. The inhibitor film has a small time constant and has a phase shift in the higher frequency range.

**Figure 1(a). Electrochemical impedance spectroscopy plots: Nyquist and Bode**

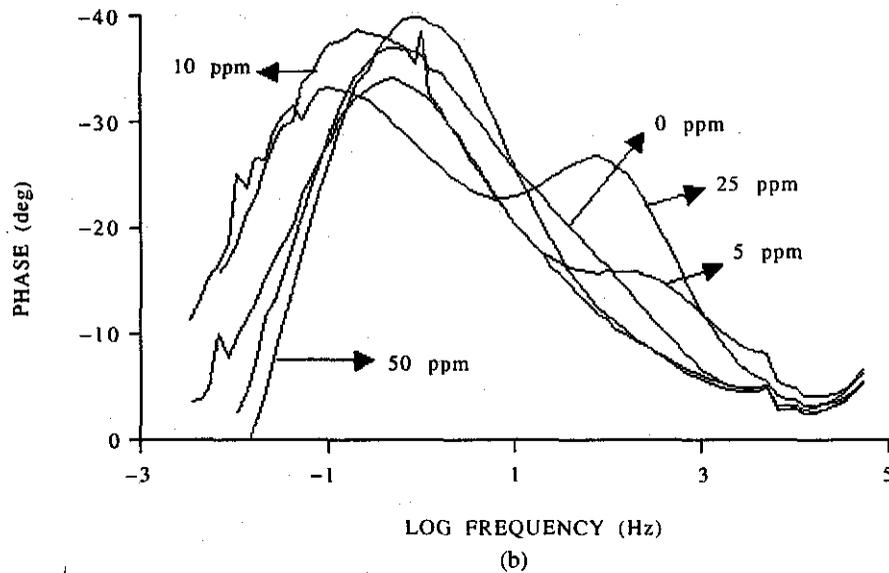


Figure 1(b). EIS plots: Nyquist of mild steel immersed in 2CEPA and zinc ions (25 ppm each) and various concentrations of citrate at pH 7, 30 °C for 0.5 h.

Figure 2(a) shows well-separated semicircles of the Nyquist plots of the electrode immersed in the test solutions for 24 h. The diameters of the Nyquist plots of electrode immersed in the test solutions increased on increasing the immersion period from 0.5 h to 24.0 h. Except the Nyquist plot of electrode immersed in 25 ppm citrate (diffusion-type), all other plots were of charge-transfer type. The respective q vs $\log f$ plots show one peak at the higher frequency and the other at the lower frequency. The height of the high-frequency peak increases on increasing the immersion period from

0.5 h to 24.0 h. The increase in the higher frequency peak correlates with the inhibitor film growth¹³.

The two-time constant model is widely used by a number of researchers¹³⁻¹⁵ for simulating the properties of an electrode covered with a single-layer inhibitor paint film [Fig. 3(a)]. The electrochemical impedance spectroscopy plots of mild steel immersed in the above inhibitor were analysed using the two-time constant model. The deviation was observed between the experimental data and the simulated data. This shows that the inhibitor film may contain

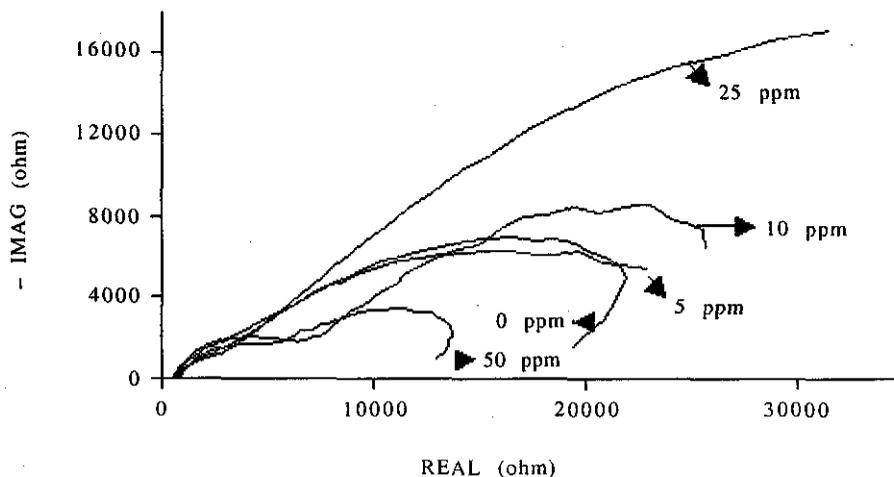


Figure 2(a). Nyquist and Bode plots

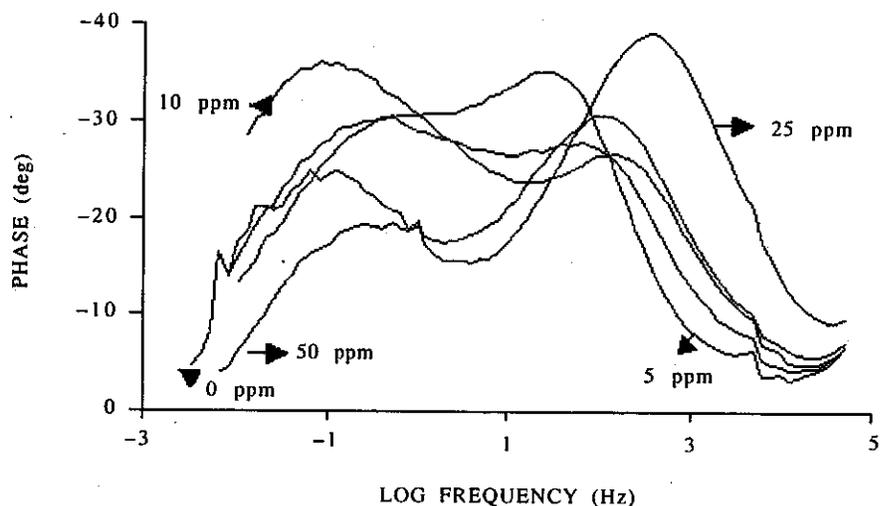


Figure 2(b). Nyquist plots of mild steel immersed in 2CEPA and zinc ions (25 ppm each) and various concentrations of citrate at pH 7, 30 °C for 24 h.

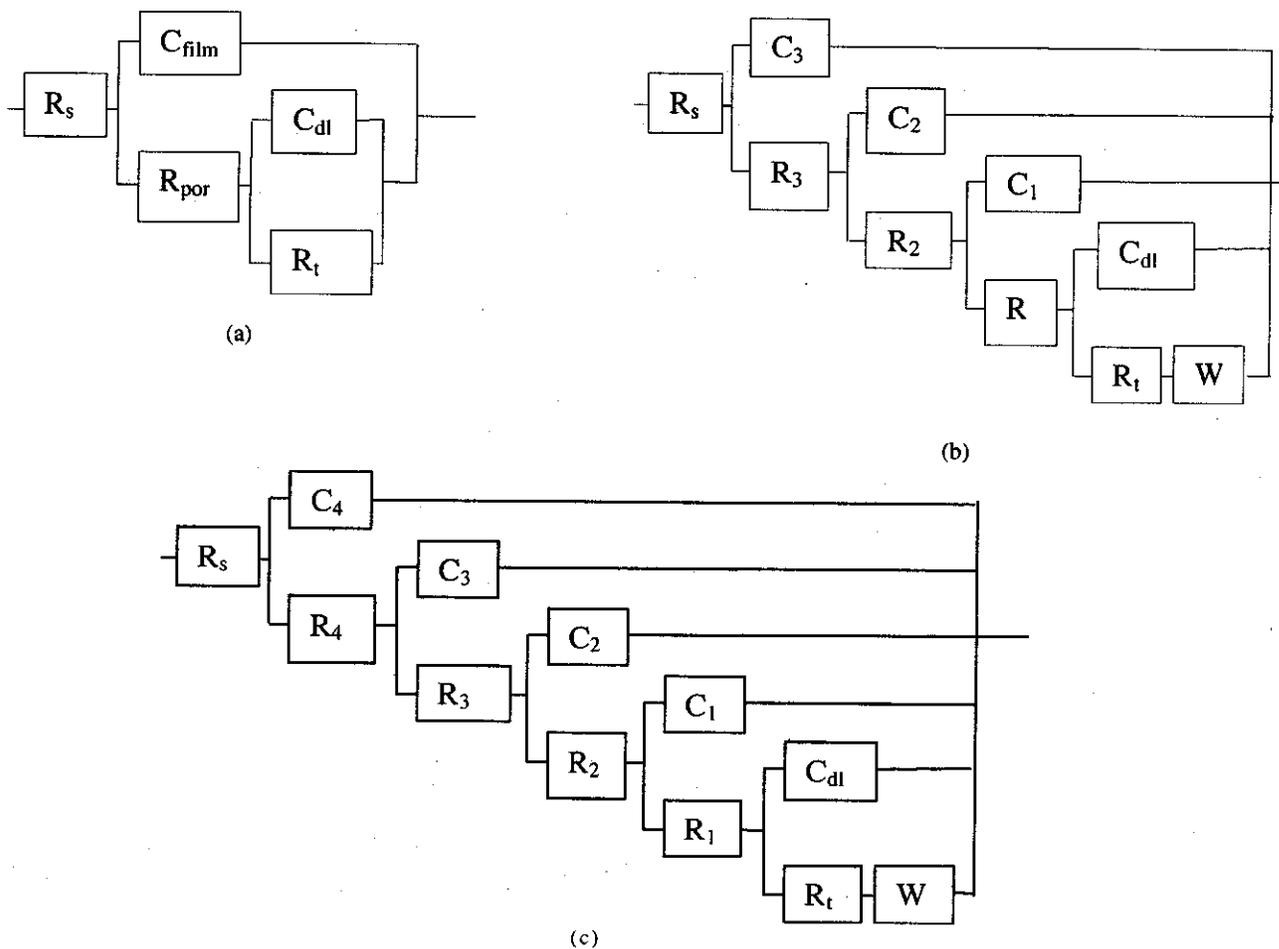


Figure 3. Equivalent circuits (models) for an electrode filmed with a nonconducting inhibitor film: (a) a single-layer inhibitor model, (b) a three-layer model, and (c) a four-layer model with Warburg impedance using a computer-controlled electrochemical software.

more than one layer. Tan¹³, *et al.* observed that the inhibitor film formed on the electrode contained multilayers, preferably three and four layers.

The electrochemical impedance spectroscopy spectra were simulated using a three-layer model [Fig. 3(b)] and a four-layer model [Fig. 3(c)] with Warburg impedance using computer-controlled electrochemical software (Gamry EIS300). The electrode surface electrochemical kinetic parameters of mild steel surface immersed in inhibitor derived by analysing the respective electrochemical impedance spectroscopy plots using the above models are given in Table 2. The R_1 - R_4 and C_1 - C_4 are the resistance and the capacitance of each layer of the inhibitor film. R_i and C_{dl} are the charge-transfer resistance of mild steel surface and capacitance of electrical double layer formed on the interface between the metal and the electrolyte. The satisfactory

simulation of the impedance and characteristic of the inhibitor filmed electrode surface by the 3-layer and the 4-layer models (Fig. 4) indicate that the inhibitor film has a multilayer structure.

Table 2 shows that the charge-transfer resistance (R_i) values increased on increasing the immersion period from 0.5 h to 24 h. The formation of high frequency peak was noticed after almost 24 h immersion. It suggests that a slow process might have been involved because adsorption equilibrium would be achieved much faster. The above evidences show that the main structure of the inhibitor film was built up in the first 24 h.

Figures 5(a) and 5(b) show the Nyquist and q vs $\log f$ formats recorded on the electrode after immersing in test solution for 96 h. Only the diameters of the Nyquist plots of electrode immersed in 10 ppm

Table 2. Electrochemical impedance spectroscopy parameters of mild steel immersed in chloride solution (60 ppm), 2CEPA and zinc ions (25 ppm each) and various concentrations of citrate

Immersion period (h)	Citrate (ppm)	R_i KΩ	C_{dl} μF	R_1 KΩ	C_1 μF	R_2 KΩ	C_2 μF	R_3 KΩ	C_3 μF	R_4 KΩ	C_4 μF	W
0.5	0	5.9	210	3.9	50	1.1	12	0.4	2.4	--	--	--
	5	6.4	277	4.4	50	1.1	13	0.5	1.2	0.2	0.3	--
	10	11.2	217	5.4	54	1.2	14	0.4	1.8	0.2	0.1	936
	25	16.2	519	13.1	89	4.3	22	2.0	2.5	1.1	0.7	--
	50	5.5	135	3.6	32	0.8	12	0.2	1.9	--	--	--
24.0	0	9.2	127	4.6	28	2.8	2	2.7	0.2	0.7	0.1	--
	5	9.4	414	7.4	60	3.0	12	1.5	1.6	0.5	0.5	--
	10	10.7	328	7.6	47	3.1	8	1.7	2.1	0.1	0.4	--
	25	25.7	230	11.6	54	3.0	6	3.0	0.5	0.9	0.2	249
	50	4.9	318	3.7	42	2.4	3	1.9	0.5	0.4	0.2	--
96.0	0	5.9	698	4.5	27	2.3	1	1.1	0.2	--	--	216
	5	5.3	912	4.2	72	1.7	10	1.0	0.4	0.8	0.1	203
	10	23.1	979	18.3	110	7.9	12	2.8	5.2	0.4	1.2	360
	25	58.5	293	33.5	65	7.0	16	3.3	0.9	2.0	0.3	108
	50	4.7	998	4.0	169	1.8	27	0.9	1.3	0.6	0.2	117
192.0	0	5.3	897	4.9	10	1.4	13	0.8	0.6	0.5	0.1	140
	5	4.7	991	4.2	56	0.8	1	0.5	0.1	--	--	150
	10	6.6	990	6.1	78	1.3	4	1.0	0.1	--	--	140
	25	10.0	306	5.9	43	1.4	5	1.0	0.7	0.2	0.2	896
	50	3.3	995	2.7	111	0.6	14	0.5	0.7	0.3	0.1	192

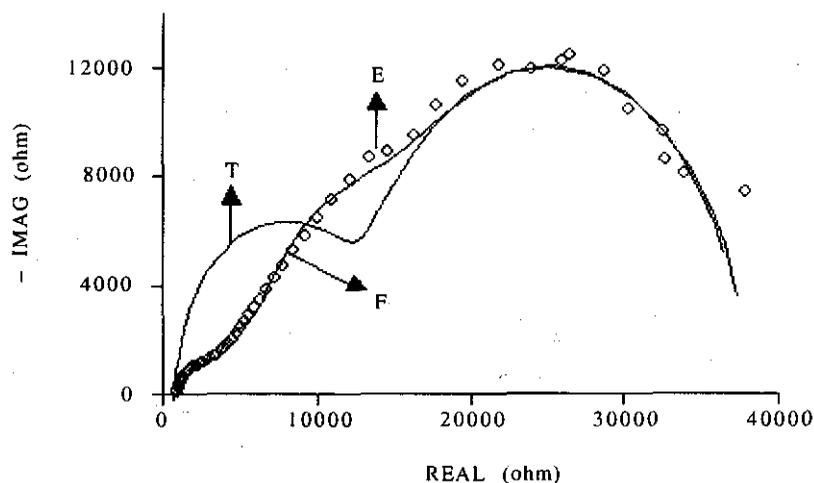


Figure 4. Experimental and simulated plots of mild steel immersed in inhibitor solution. T is the two-time constant model, F is the four-time constant model, and E is the experimental data.

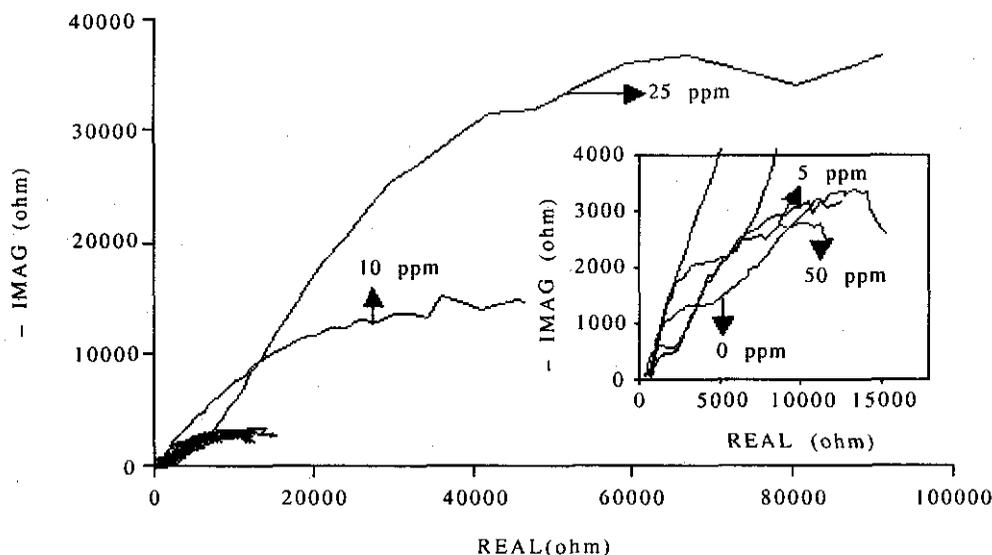


Figure 5(a). Nyquist and Bode plots

and 25 ppm increased. The diameters of the remaining Nyquist plots decreased. All the Nyquist plots of Fig. 4(a) are diffusive-types. The q vs $\log f$ formats of Fig. 5(b) show that the high-frequency peak completely dominates over the low-frequency peak. It shows that the inhibitor film buildup reaches almost the maximum level. The high charge-transfer resistance values (R_t) and Warburg constants for electrode immersed in 10 ppm and 25 ppm citrate concentrations may be due to the continuous diffusion of the inhibitor molecules. For the other concentrations

of citrate, the low R_t values for the low concentration of citrate indicate that the inhibitor is not adequate to protect the mild steel surface by forming inhibitor film. Since citrate alone is corrosive in nature (Table 1), addition of high concentrations of citrate (50 ppm) to 2CEPA and zinc ions accelerates corrosion of the mild steel due to the presence of excess citrate.

The Nyquist and q vs $\log f$ formats recorded on the electrode after immersing in test solutions

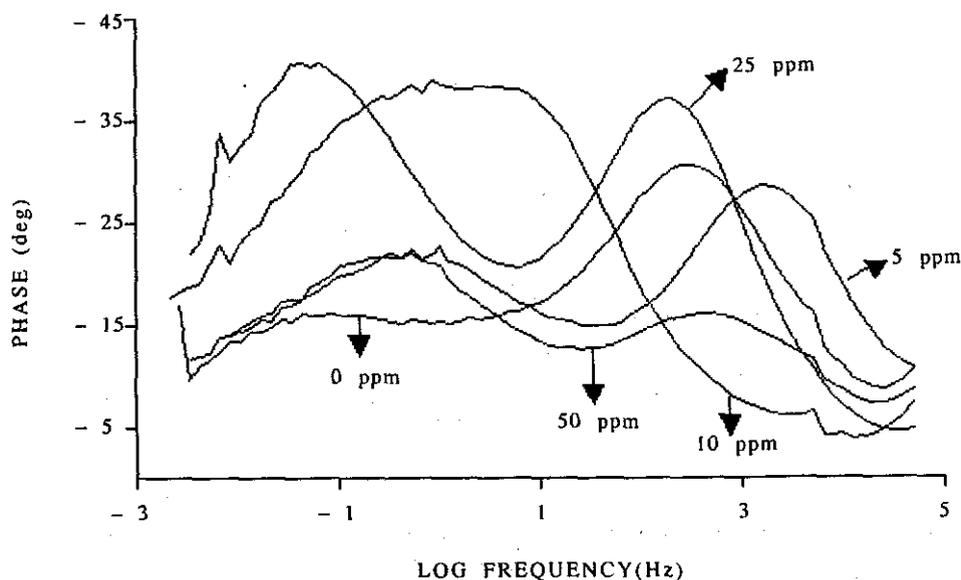


Figure 5(b). Nyquist plots of mild steel immersed in of 2CEPA and zinc ions (25 ppm each), and various concentrations of citrate at pH 7, 30 °C for 96 h.

for 192 h are shown in Figs 6(a) and 6(b). On comparing Fig. 5(a) with Fig. 6(a), a decrease in the diameters of the Nyquist plots of Fig. 6(a) is noticed. It shows that the resistance of inhibitor film formed on the mild steel immersed in inhibitor decreases on increasing the exposure time from 96 h to 192 h. All the Nyquist plots of Fig. 6(a) are diffusive-types. The q vs $\log f$ formats of Fig. 6(b) show the presence of high-frequency peaks and low-frequency peaks. The low R_i values may be due to the diffusion of corroding ions in

the inhibitor film, which weakened the resistance of the inhibitor film.

Figure 7 shows the relationship between the charge-transfer resistance and the immersion period. In case of 10 ppm and 25 ppm concentrations of citrate, the R_i values increase on increasing the immersion time up to 96 h and thereafter, these decrease on increasing the immersion time. It shows that till 96 h, building up of the inhibitor film takes place and after that, the film starts deteriorating.

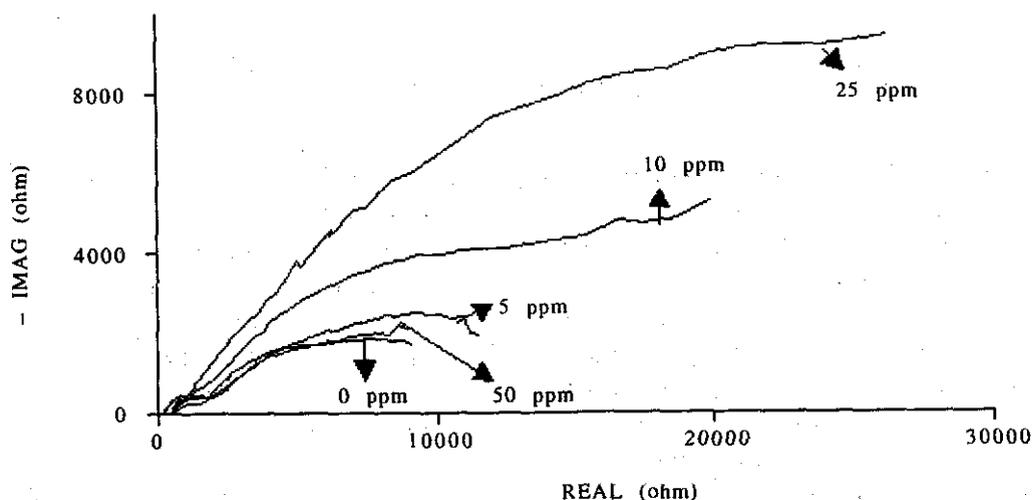


Figure 6(a). Nyquist and Bode plots

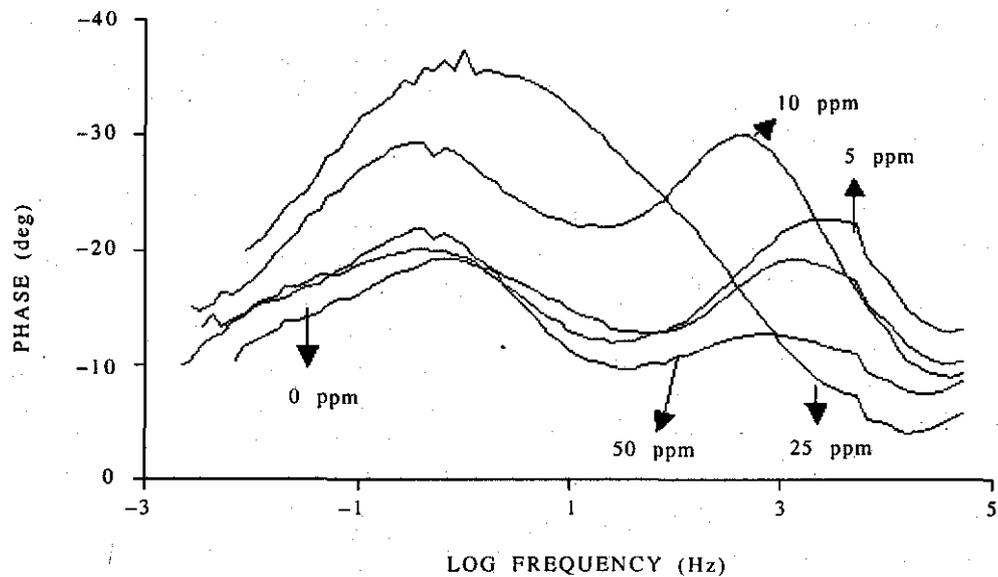


Figure 6(b). Nyquist plots of mild steel immersed in 2CEPA and zinc ions (25 ppm each) and various concentrations of citrate at pH 7, 30 °C for 196 h.

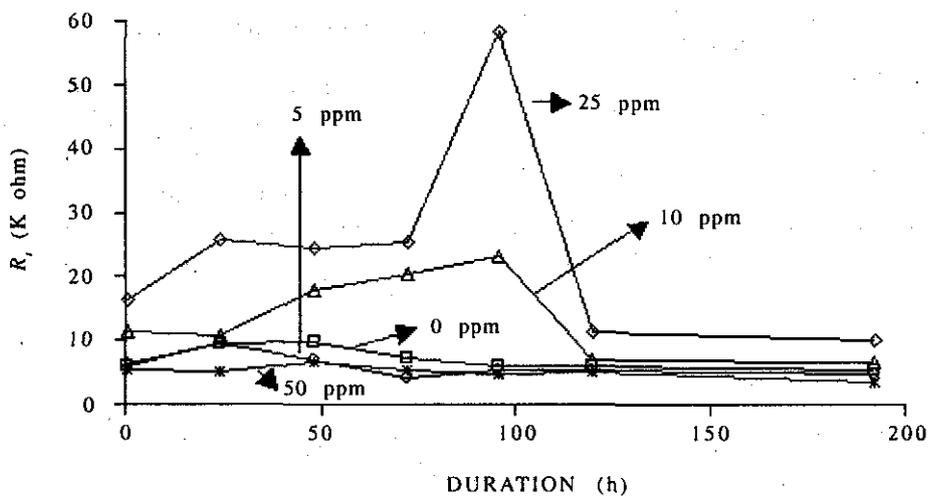


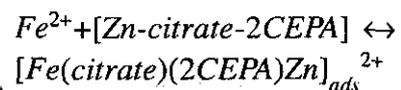
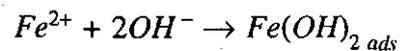
Figure 7. Relationship between immersion time and polarisation resistance of mild steel immersed in 2CEPA and zinc ions (25 ppm each) and various concentrations of citrate at pH 7, 30 °C.

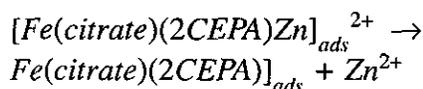
In case of other concentrations, deterioration starts after 48 h of immersion.

For support of the above evidences, weight-loss experiments were conducted using mild steel as test coupon. The results have been presented in Table 2. It shows that the minimum corrosion rate is achieved for the mild steel immersed in citrate, 2CEPA and zinc ions (25 ppm each).

5.1 Mechanism of Action

On the surface of iron, water soluble zinc complex adsorbs and reacts with the iron ions available on the surface¹⁶:





The iron-inhibitor complex is more stable than zinc-inhibitor complex (stability constants for Zn-pyrophosphate and Fe-pyrophosphate complexes* are 12.54 and 6.45). The anodic current becomes very small when the iron-inhibitor complex replaces the iron hydroxides, which implies that the surface coverage of iron hydroxide should be zero. These results were reflected by the weight-loss experiment [maximum inhibitor efficiency of 96 per cent is noticed for inhibitor system containing 2CEPA, citrate, and zinc ions (25 ppm each)].

For the inhibition process, the zinc-inhibitor complex diffuses on the steel surface and forms iron-inhibitor complex. During this process, there is more accumulation of zinc-inhibitor complex and iron-inhibitor complex on the surface, which forms multilayer structure on the surface. Depending upon the composition of the inhibitor layers, the resistance changes. The thickness of each layer may roughly be evaluated by applying the following relationship:

$$D = \epsilon_0 \epsilon A/C$$

where ϵ_0 , ϵ , A , and C are the vacuum permittivity, dielectric constant of inhibitor film^{17,18} (Approx. 2),

area of the specimen, and the capacitance, respectively. The results show that the thickness of the first, second, third, and the fourth layer is 20-30 Å, 200-300 Å, 2000-3000 Å, and 8000-10000 Å, respectively.

A physical model can be suggested as shown in Fig. 8. The resistance of the first layer is maximum indicating higher molecular cross-linked inhibitor film containing mostly iron-inhibitor complex. The second layer may contain iron-inhibitor and zinc-inhibitor complexes. The zinc-inhibitor complex may penetrate the first layer and form iron-inhibitor complex. The third layer may contain dense water-soluble zinc-inhibitor complex because the resistance of the film is only slightly higher than the solution resistance. The resistance of the fourth layer is very close to that of the solution resistance, indicating that this layer may contain slightly more concentrated water-soluble zinc-inhibitor complex with corroding ions.

6. CONCLUSIONS

The electrochemical impedance spectroscopy results reveal that the corrosion inhibitive film formed on the mild steel surface by the citrate-based inhibitor system contains multilayers. Each layer has different resistance value. The first layer has higher resistance value than the second layer. The corrosion inhibitive

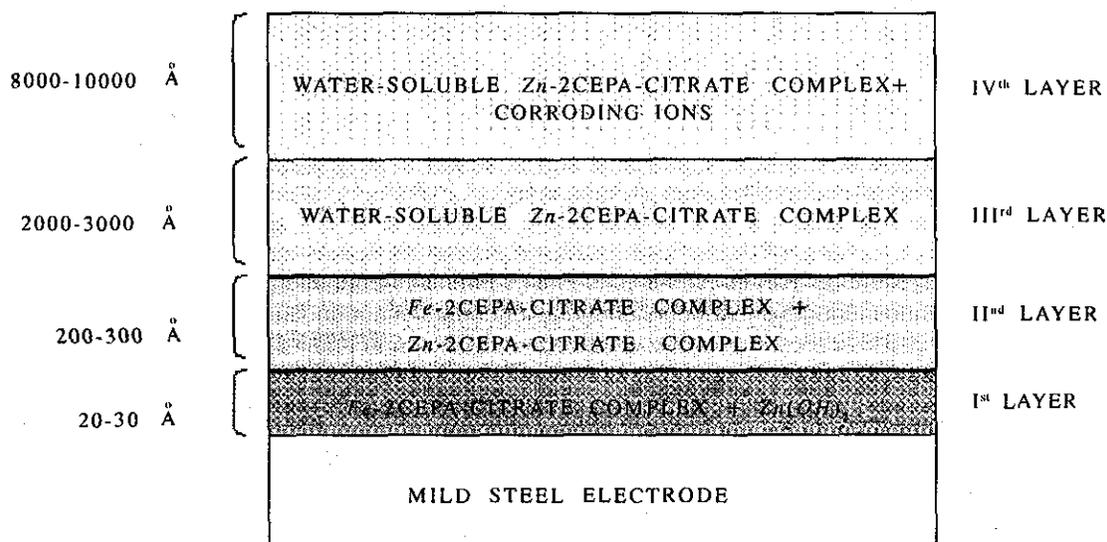


Figure 8. Physical model of inhibitor layers formed on electrode (mild steel)

*Saraiya, S.C. & Sundaram, A.K. A review on the study of complexes by polarography submitted to Bhaba Atomic Research Centre, Bombay, India (1969).

film buildup starts within 24 h of immersion period, and till 96 h of immersion, strengthening of the corrosion inhibitive film takes place. After 96 h of immersion, the corrosion inhibitive film starts deteriorating because of the penetration of corroding ions due to the poor availability of water-soluble zinc complex for inhibiting iron-inhibitor complex formation. The first layer's thickness is less than the second layer's thickness. The higher resistance of the first layer may be due to cross-linked iron-corrosion inhibitive film formation.

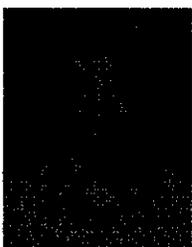
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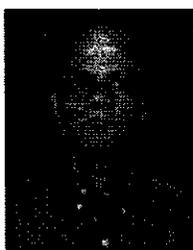
Contributors



Dr G. Gunasekaran obtained his MSc (Chemistry) from the Pondicherry University (Gold medallist), in 1991. He was awarded PhD by the Gandhigram Rural University in 1998 for his work 'Synergistic inhibition of corrosion of mild steel using 2-carboxyethylphosphonic acid, zinc ions, and organic additives'. He joined Naval Materials Research Laboratory (NMRL), Ambarnath, as Scientist B to take-up work on cathodic protection of marine structures. His areas of research are: Bio-fouling control, corrosion inhibitors, development of sacrificial and platinised titanium anodes, and galvanic corrosion. He has 20 publications, 2 patents, and several reports to his credit.



Mr B.I. Dubey obtained his BSc (Chemistry) from the Gorakhpur University in 1975. He joined NMRL, Ambarnath, in the Corrosion and Cathodic Protection Division. Since then, he is actively engaged in the development of aluminium alloy anodes of different compositions for the cathodic protection of underwater hull and appendages in marine environment. He was one of the member of the group, which received group award from DRDO Hqrs for the development of ICCP system along with the fieldwork, ie, service to the Navy. He has carried out R & D work on cathodic protection of marine structures and was engaged with the fitment of ICCP system on different classes of I.N. ships and has acquired high degree of expertise in the area. He has got one patent, three papers and several reports to his credit. Presently, he is engaged on studies on galvanic corrosion and cathodic protection of aluminium hull by sacrificial and ICCP systems.



Dr J. Rangarajan obtained his PhD from the Bombay University in 1982 for his work on Electrochemistry of zinc. He was awarded the DAE research fellowship for carrying out work at the Bhabha Atomic Research Centre (BARC), Mumbai, during 1978-83. He joined as Senior Scientific Officer/Lecturer at the Inorganic and Physical Chemistry Dept of the Indian Institute of Science, Bangalore, where he worked on electro-catalyst, photo-electrochemistry, and preparative inorganic chemistry. He later joined NMRL, Ambarnath, in 1986 to take-up the work on electrochemistry and fuel cells. He has 2 theses and 14 papers. He has worked in the areas of corrosion, cathodic protection and was instrumental in initiating this programme on fuel cells in NMRL. He now leads the Corrosion Protection and Electro Chemistry Group of NMRL.