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## **Inhibitive action of phosphonic acid -Ni<sup>2+</sup> system on the corrosion of carbon steel in 60 ppm chloride Environment**

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### **ABSTRACT**

The corrosion inhibition effect of carbon steel in 60 ppm of chloride ion environment by 2-chloroethylphosphonic acid (2-CIEPA)- Ni<sup>2+</sup> system has been investigated using classical weight loss method. The results show that 32% inhibition efficiency (IE) achieved with binary system consisting of 100 ppm of 2-CIEPA and 50 ppm of Ni<sup>2+</sup>. The addition of 50 ppm of sodium gluconate (SG) on 2-CIEPA- Ni<sup>2+</sup> system increased the IE from 32% to 98%. FTIR investigate the nature of the protective film formed on the metal surface. The protective film consists of Fe<sup>2+</sup>-2-CIEPA complex, Fe<sup>2+</sup>-SG complex and Ni(OH)<sub>2</sub>. SEM and AFM used to prove the formation of protective film. EDS study determines the elements of inhibitor present on the metal surface. The mechanistic aspects of corrosion inhibition

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based on the results obtained from the classical weight-loss method and the electrochemical method such as AC impedance and polarization study.

**KeyWords:** Carbon steel, 2-chloroethylphosphonic acid, sodium gluconate, Inhibition efficiency, protective film;

## INTRODUCTION

Carbon steel is the most important engineering material. Corrosion problem occurs in these industries and can cause disastrous damage to metal and alloy structures causing economic consequences in terms of repair, replacement, and product losses. The protection against corrosion is important for metals containing intermetallic particles, which are widely used in industry but susceptible to pitting corrosion in the media containing chloride ions. Corrosion inhibitors are widely used in industry to prevent or to reduce the corrosion rates of surface of the metal from the corroding medium. The organic compounds and several carboxylates such as sodium molybdate[1,2] sodium salicylate, sodium cinnamate, adipate and phosphonic acid have been used as inhibitors[3-5]. Synergistic effect of succinic acid and  $Zn^{2+}$  in controlling corrosion of carbon steel in well water has been reported [6]. Several phosphonic acids have used as corrosion inhibitor [7-11]. Phosphonic acids are organic compounds containing which are effective chelating agents that used in cooling water and desalination systems to inhibit scale formation and corrosion. The approach of inhibitors with nickel is recent trend in corrosion inhibition [12-14].

The aim of the present study is

- 1 To evaluate the inhibition efficiency of 2-chloroethylphosphonic acid (2-CIEPA) in controlling the corrosion of carbon steel of the 60 ppm of chloride environment in the absence and presence of  $Ni^{2+}$ .
- 2.To enhance the inhibition efficiency of 2-CIEPA, the additive sodium gluconate (SG) are added.
- 3.To study the mechanistic aspects by potentiodynamic polarization study and AC impedance study.
- 4.To study the nature of protective film formed on the carbon steel by SEM, AFM, EDS and FTIR spectrophotometer.
- 5.To propose a suitable mechanism for corrosion inhibition based on the results from the above study.

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## MATERIALS AND METHODS

### Preparation of the specimen

Carbon steel (0.026%S, 0.06%P, 0.4%Mn, 0.1% C, and the rest Fe) specimen of dimension 1 cm x 4 cm x 0.2 cm were used for weight loss study. Carbon steel specimens polished to mirror finish with help of emery paper of different grade and degreased with trichloroethylene.

### Weight loss method

Carbon steel specimens in triplicate were immersed in 100 ml of water in 60 ppm of chloride environment containing various concentrations of the inhibitors, in the absence, presence of Ni<sup>2+</sup> ions, for a period of seven days.

The weight of the specimens before and after immersion was determined using Shimadzu balance, AY62 model. The corrosion products were cleansed with Clarke's solution[15]. The corrosion rates (CR) of the metal specimens were calculated with the help of the following relationship:

where

$$CR = \frac{\Delta m}{A * t} \quad (1)$$

CR - corrosion rate

$\Delta m$  - loss in weight (mg)

A - surface area of the specimen (dm<sup>2</sup>)

t - Period of immersion (days)

The inhibition efficiency (IE) was then calculated using the equation

$$IE = 100 \left( 1 - \frac{W_2}{W_1} \right) \quad (2)$$

Where, W<sub>1</sub> and W<sub>2</sub> are the corrosion rates in the absence and presence of the inhibitor respectively.

### Polarization study

Polarization studies were carried out by using CHI electrochemical impedance analyzer, model 660A. A three electrode cell assembly was employed. The working electrode used was rectangular mild steel with one face of the electrode exposed and the rest shielded with red lacquer. A saturated calomel electrode (SCE) was used as the reference electrode and a rectangular platinum foil was used as the counter electrode. According to the Stern-Geary equation, the steps of the linear polarization plot are substituted to get corrosion current

$$I_{\text{corr}} = b_a \times b_c / 2.303 (b_a + b_c) R_p$$

Where,  $R_p$  is polarization resistance.

The results, such as Tafel slopes ( $b_a$  and  $b_c$ ), and corrosion current ( $I_{\text{corr}}$ ) and corrosion potential ( $E_{\text{corr}}$ ) values were calculated.

### AC impedance study

The instrument used for polarization was also used for AC impedance study. The cell set up was the same as that used for polarization measurements. The real part and imaginary part of the cell impedance were measured in ohms at various frequencies. The values of charge transfer resistance ( $R_t$ ) and the double layer capacitance ( $C_{dl}$ ) were calculated.

### Surface analysis

The carbon steel specimens were immersed in various test solutions for a period of seven days; the specimens were taken out, washed with distilled water and then dried. The nature of the film formed on the surface of metal specimens was analysed by the following techniques.

### FTIR Spectra

The film formed on the carbon steel carefully removed with sharp edged glass rod, mixed thoroughly with KBr and made into pellets, and the FTIR spectra recorded on a Perkin-Elmer 1600 spectrophotometer.

### SEM / EDS study

The Surface morphology measurements of the carbon steel examined by using JEOL JSM 6390 model. All SEM micrographs of carbon steel are taken at a magnification of  $X=500$ .

### Atomic Force Microscopy

When carbon steel specimen immersed in the inhibitors solution for a period of seven days, After seven days the metal specimens was removed, rinsed with double distilled water, dried and subjected to the surface examination. The protective films were examined with atomic force microscopy (AFM) using A100 model, A.P.E research, Italy. The topography of the entire samples from a scanned of  $10\ \mu\text{m} \times 10\ \mu\text{m}$  is evaluated for a set point of 20nm and a scan speed of 10mm/ second.

## RESULTS AND DISCUSSION

### Analysis of results of mass loss method

The corrosion inhibition efficiency of carbon steel immersed in 60 ppm of chloride ion environment in the absence and presence of inhibitor systems are given in Table 1 & Table 2. It is seen from Table 1 that the 2-CIEPA alone is poor inhibitor. But in presence of  $\text{Ni}^{2+}$ , it shows low IE. As concentration 2-CIEPA increases, IE increases. For example, 50 ppm of  $\text{Ni}^{2+}$  and 100 ppm 2-CIEPA of shows 32% IE. However, at higher concentration offer good IE. This suggests that a synergistic effect exists between 2-CIEPA –  $\text{Ni}^{2+}$  system.

**Table 1.** Corrosion inhibition efficiency (IE) of carbon steels in 60 ppm of chloride environment in the presence of inhibitor obtained by weight loss method.

S. No	$\text{Ni}^{2+}$ (ppm)	IE %
1	0	0
2	10	6
3	20	13
4	30	14
5	40	16
6	50	16
7	60	18

**Table 2.** Corrosion inhibition efficiency (IE) of carbon steels in 60 ppm of chloride environment in the presence of inhibitor obtained by weight loss method.

S.No	Ni <sup>2+</sup> (ppm)	2-CIEPA (ppm)	IE %
1	0	0	0
2	50	100	32
3	50	200	86
4	50	300	98
5	50	400	98
6	50	500	98
7	50	600	98

### **Influence of sodium gluconate on 2-CIEPA – Ni<sup>2+</sup> system**

The influence of sodium gluconate on 2-CIEPA – Ni<sup>2+</sup> system in 60 ppm of chloride ion environment are given in Table 3. It is seen from Table 3 that the formulation consisting of 100 ppm of 2-CIEPA and 50 ppm of Ni<sup>2+</sup> has 32% IE. When various concentrations of SG (25, 50, 75 .....150 ppm) are added, excellent inhibition efficiencies are obtained at low concentration itself. For example, when 50 ppm of SG is added to the formulations consisting of 100 ppm of 2-CIEPA and 50 ppm of Ni<sup>2+</sup>, the inhibition efficiency increases from 32% to 98%. This suggests a synergistic effect existing between SG and Ni<sup>2+</sup> - 2-CIEPA system.

**Table 3.** Corrosion inhibition efficiency (IE) of carbon steels in 60 ppm of chloride environment in the presence of inhibitor obtained by mass loss method.

S. No	Ni <sup>2+</sup> (ppm)	2-CIEPA (ppm)	SG (ppm)	IE %
1	0	0	0	--
2	50	100	25	92
3	50	100	50	98
4	50	100	75	98
5	50	100	100	98
6	50	100	125	98
7	50	100	150	98

### Analysis of polarization study

The polarization curves of carbon steel immersed in 60 ppm of chloride environment in the presence and absence of inhibitors are shown in Figure 1. The corrosion parameters are given in Table 4.

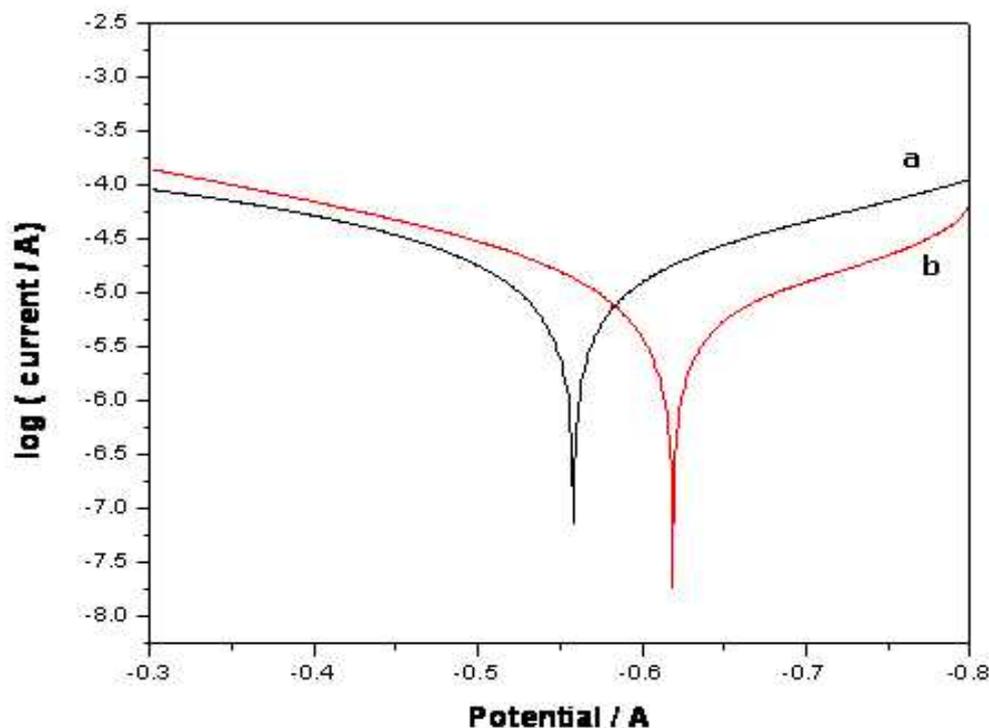
**Table 4** : Corrosion parameters of carbon steel immersed in various test solution obtained by polarization method.

Description	$E_{\text{corr}}$ mV/decade	$b_a$ mV/decade	$b_c$ mV/decade	$I_{\text{corr}}$ A/cm <sup>2</sup>
60 ppm Cl <sup>-</sup>	-598	192	198	12.9x 10 <sup>-6</sup>
60 ppm Cl <sup>-</sup> + 50 ppm Ni <sup>2+</sup> + 100 ppm 2- CIEPA + 50 ppm SG	-619	137	198	1.50 x 10 <sup>-6</sup>

When the carbon steel immerse in 60 ppm of chloride environment, the corrosion potential ( $E_{\text{corr}}$ ) is -598 mV Vs SCE. The formulation consisting of 100 ppm of 2-CIEPA, 50ppm of Ni<sup>2+</sup> and 50 ppm of SG shifts the corrosion potential from -598 to -619 mV Vs SCE. That is, corrosion potential shifts to cathodic direction. This suggests that the cathodic reaction is controlled predominantly.

Now the shifts in the anodic and cathodic slopes can be compared. Tafel values for the chloride ion environment are almost same. The Tafel values for the formulation are not equal ( $b_a = 137$  mV/decade  $b_c = 198$  mV/decade)

When the carbon steel immerse in chloride ion environment, the corrosion current ( $I_{\text{corr}}$ ) is 12.09x10<sup>-6</sup> A/cm<sup>2</sup>. It is decreased to 1.50 x 10<sup>-6</sup> A/cm<sup>2</sup> for the best formulation. The current of the iron dissolution is decreased significantly indicating that the metal surface became passive by forming inhibitor layer. The passivity ion is probably due to the formation of 2-CIEPA – Fe<sup>2+</sup> and SG – Fe<sup>2+</sup> surface layer.



**Fig 1: Polarization curves of carbon steel immersed in various test solution**

a. 60 ppm  $\text{Cl}^-$

b. 60 ppm  $\text{Cl}^-$  + 50 ppm of  $\text{Ni}^{2+}$  + 100 ppm of 2- CIEPA + 50 ppm of SG

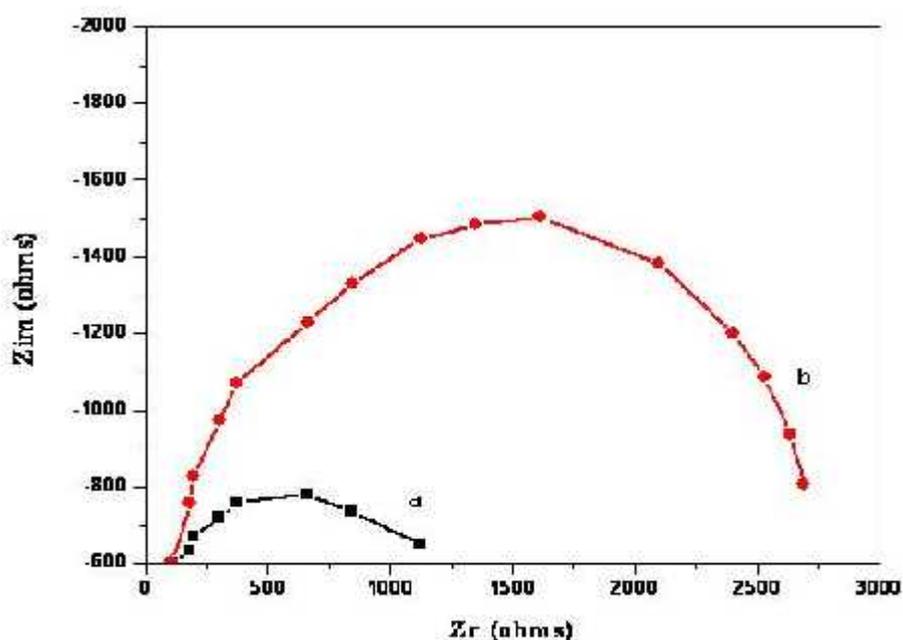
The significant reduction in corrosion current for inhibitor formulation may indicate more adsorption of the inhibitors and better inhibitions performance. This result suggests that a protective film is formed on the metal surface. This protects the metal from corrosion.

### **Analysis of AC impedance Study**

The Nyquist plots for the corrosion behavior of carbon steel immersed in various solutions are shown in Figure 2. The AC impedance parameters, namely, charge transfer resistance ( $R_t$ ) and double layer capacitance ( $C_{dl}$ ) are given in Table 5.

**Table 5 :** Corrosion parameters of carbon steel immersed in various test solution obtained by AC impedance study.

System	$R_t$ ( $\Omega \text{ cm}^2$ )	$C_{dl}$ ( $\text{F/cm}^2$ )
60 ppm $\text{Cl}^-$	342.17	$27.00 \times 10^{-6}$
60 ppm $\text{Cl}^-$ + 50 ppm of $\text{Ni}^{2+}$ + 100 ppm of 2- CIEPA + 50 ppm of SG	2600.80	$1.85 \times 10^{-6}$



**Fig 2:** AC impedance spectra of carbon steel immersed in various test solution

a. 60 ppm  $\text{Cl}^-$

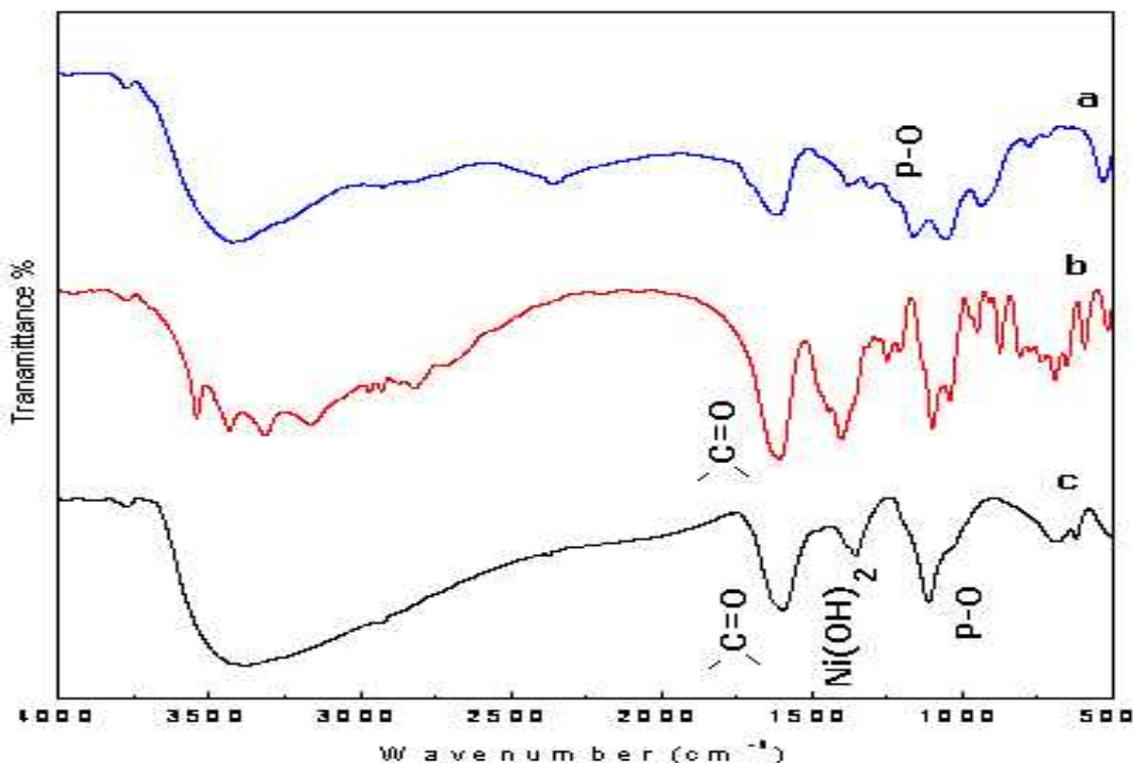
b. 60 ppm  $\text{Cl}^-$  + 50 ppm of  $\text{Ni}^{2+}$ + 100 ppm of 2- CIEPA + 50 ppm of SG

The carbon steel immersed in 60 ppm chloride ion environment,  $R_t$  value of  $342.17 \Omega \text{ cm}^2$  and  $C_{dl}$  value of  $27.00 \times 10^{-6} \text{ F cm}^{-2}$  observed. When the formulation consisting of 50 ppm  $\text{Ni}^{2+}$ + 100 ppm of 2- CIEPA + 50 ppm of SG added to chloride environment,  $R_t$  value increases tremendously from  $342.17 \Omega \text{ cm}^2$  to  $2600.80 \Omega \text{ cm}^2$ . The  $C_{dl}$  decreases from

$27.00 \times 10^{-6} \text{ F cm}^{-2}$  to  $1.85 \times 10^{-6} \text{ F cm}^{-2}$ . This suggests that a protective film formed on the surface of the metal. This accounts for the very high IE of 2-CIEPA -  $\text{Ni}^{2+}$  - SG system.

### Analysis of FTIR spectra

The FTIR spectrum of pure 2-CIEPA is shown in Figure 3a. The P-O stretching frequency of phosphonic acid group appears at  $1159 \text{ cm}^{-1}$ . The FTIR spectrum of pure sodium gluconate is shown in figure 3b. The C=O stretching frequency of carboxyl group appears at  $1652 \text{ cm}^{-1}$ .



**Fig 3. FTIR spectra of:**

- (a) Pure 2-CIEPA,**
- (b) Pure Sodium gluconate (SG)**
- (c)  $\text{Ni}^{2+}$  (50 ppm), 2-CIEPA (100 ppm) and SG (50 ppm)**

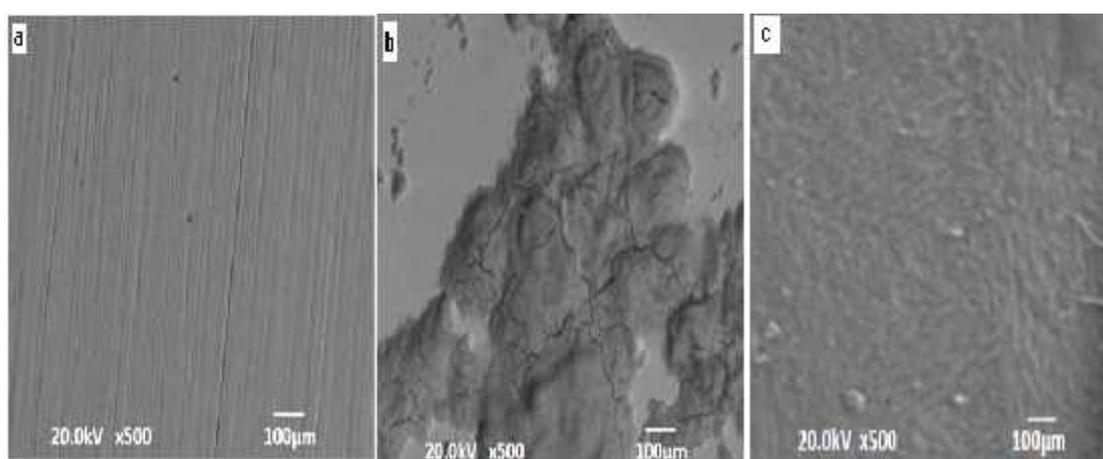
The FTIR spectrum (KBr pellet) of the film formed on the carbon steel surface after immersion in the water containing 50 ppm of SG, 50 ppm of  $\text{Ni}^{2+}$  and 100 ppm of 2-CIEPA in the chloride ion environment is shown in figure 3c. The P-O stretching frequency has decreased from  $1159 \text{ cm}^{-1}$  to  $1099 \text{ cm}^{-1}$ . This shift is caused by the decreased of the electron cloud density from the oxygen atom to  $\text{Fe}^{2+}$ , it is suggested that oxygen atom of the

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phosphonic acid is coordinated to  $\text{Fe}^{2+}$ , resulting in the formation of  $\text{Fe}^{2+}$ -2-CIEPA complex on the metal surface. In the case of SG, the  $\text{C}=\text{O}$  stretching frequency has decreased from  $1652\text{ cm}^{-1}$  to  $1597\text{ cm}^{-1}$ . This indicates that carboxyl group of SG has coordinated with  $\text{Fe}^{2+}$  on the anodic sites of the metal surface, resulting in the formation of  $\text{Fe}^{2+}$  - SG complex. The peak at  $1382\text{ cm}^{-1}$  is due to  $\text{Ni}(\text{OH})_2$  formed on the cathodic sites of the metal surface [16,17]. The FTIR spectra led to conclusion that the protective film consists of  $\text{Fe}^{2+}$ - SG complex,  $\text{Fe}^{2+}$  - 2-CIEPA complex and  $\text{Ni}(\text{OH})_2$ .

### Analysis of SEM

The scanning electron microscopy image of magnification (X500) of carbon steel specimen immersed in 60 ppm of chloride environment for seven days in absence and presence of inhibitor are shown in figure 4. The SEM micrograph of polished carbon steel (control) in figure 4a shows the smooth surface of the metal [18]. This shows the absence of any corrosion products formed on the surface.



**Figure 4: SEM image of:**

**(a) Polished carbon steel specimen,**

**(b) Carbon steel specimen in 60 ppm  $\text{Cl}^-$**

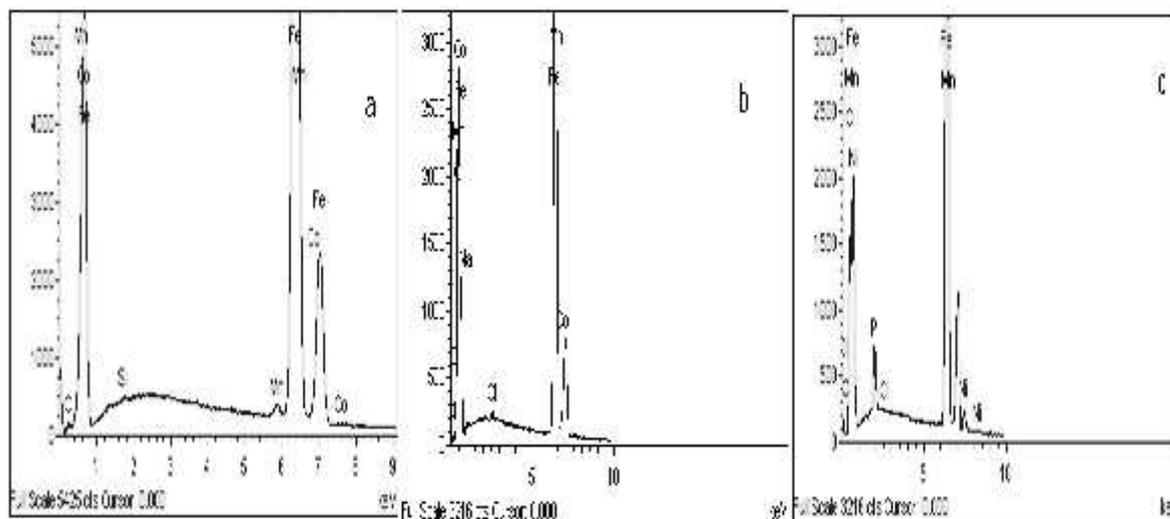
**(c)  $\text{Ni}^{2+}$  (50 ppm), 2-CIEPA (100 ppm) and SG (50 ppm)**

The SEM micrograph of carbon steel surface immersed in 60 ppm of chloride environment in figure 4b shows the roughness of the metal surface, which indicates the corrosion of carbon steel in chloride environment. Figure 4c indicates that in the presence of 50 ppm of SG, 100 ppm of 2-CIEPA and 50 ppm of  $\text{Ni}^{2+}$  mixture in chloride environment, the surface coverage increases which in turn results in the formation of insoluble complex of the metal surface

covered by a thin layer of inhibitors, which effectively controls the dissolution of the carbon steel.

### Analysis of EDS

The energy dispersive spectroscopy (EDS) of polished carbon steel (control) in figure 5a shows its composition. The EDS spectrum of chloride environment is shown in figure 5b. This indicates the presence of C, Co, Mn and Fe along with Chloride on the metal surface.



**Figure 5: EDS spectra of:**

**(a) Polished carbon steel specimen,**

**(b) Carbon steel specimen in 60 ppm  $\text{Cl}^-$**

**(c)  $\text{Ni}^{2+}$  (50 ppm), 2-CIEPA (100 ppm) and SG (50 ppm)**

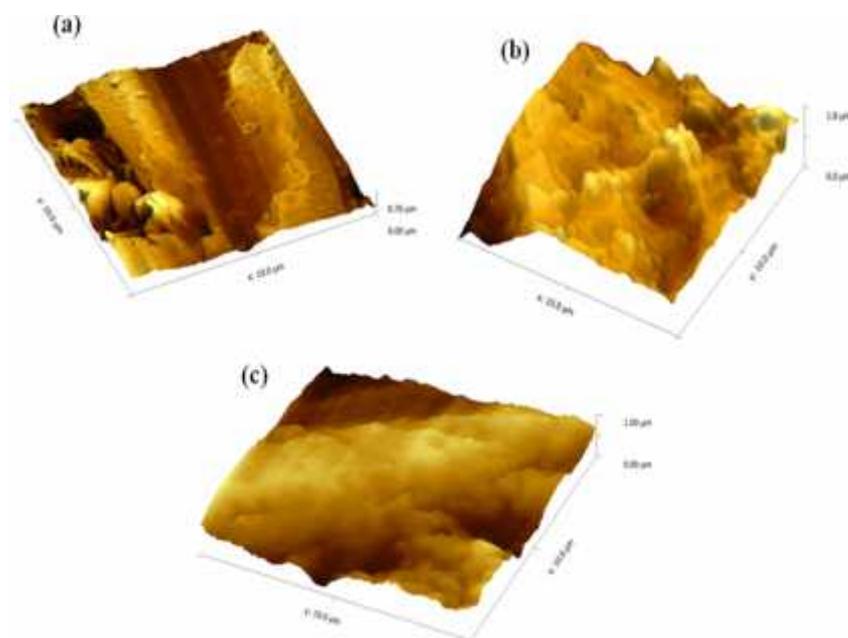
The EDS spectrum of film formed on the surface of carbon steel after immersion in chloride environment containing 50 ppm of SG, 100 ppm of 2-CIEPA and 50 ppm of  $\text{Ni}^{2+}$  is shown in figure 5c. This proves that the presence of phosphorous, nickel and oxygen atom of above mentioned Inhibitor system along with composition of carbon steel. This indicates that oxygen atom of functional group of SG has coordinated with  $\text{Fe}^{2+}$ , resulting in the formation of  $\text{Fe}^{2+}$ -SG complex and  $\text{Fe}^{2+}$ -2-CIEPA complex on the anodic sites of metal surface and  $\text{Ni}(\text{OH})_2$  formed on the cathodic sites of metal surface.

### Analysis of AFM

The three dimensional (3D) AFM morphologies for polished carbon steel surface (reference sample), carbon steel surface immersed in 60 ppm of chloride ion environment (blank sample) and carbon steel immersed in 60 ppm of chloride ion containing the formulation of

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100 ppm of 2-CIEPA, 50 ppm of  $\text{Ni}^{2+}$  and 50 ppm of SG are shown in figure 6. The average roughness ( $R_a$ ), rms roughness ( $R_q$ ) maximum peak-to-valley height (P-V) value for carbon steel surface immerse in different environment are shown in Table 6.



**Fig 6: AFM image of:**

**(a) Polished carbon steel specimen,**

**(b) Carbon steel specimen in 60 ppm  $\text{Cl}^-$**

**(c)  $\text{Ni}^{2+}$  (50 ppm), 2-CIEPA (100 ppm) and SG (50 ppm)**

The value of  $R_a$ ,  $R_q$  and P-V height for the polished carbon steel surface (reference sample) are 108 nm, 130 nm and 761 nm respectively, which shows a more homogeneous surface, with some places in where the height is lower than the average depth. Figure 5a displays the non-corroded metal surface. The slight roughness observed on the polished mild steel surface is due to atmospheric corrosion. The average roughness, rms roughness and P-V height values for the mild steel surface immersed in 60 ppm of chloride environment are 143 nm, 190 nm and 1758 respectively. These data suggests that carbon steel surface immersed in 60 ppm of chloride environment has a greater surface roughness than the polished metal surface, which shows that the unprotected mild steel surface is rougher and was due to the corrosion of carbon steel. Figure 5b displays corroded metal surface with few pits. The presence of 100 ppm of 2-CIEPA, 50 ppm of  $\text{Ni}^{2+}$  and 50 ppm of SG in 60 ppm of chloride environment reduces the  $R_q$  from 190 nm to 147 nm and the average roughness is significantly reduced to 120 nm when compared with 143 nm of carbon steel surface immersed in 60 ppm of chloride

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environment. The maximum peak-to-valley height also was reduced from 1758 nm to 995 nm.

**Table 6:** AFM data for carbon steel surface immersed in inhibited and uninhibited environments.

System	Average Roughness ( $R_a$ ) nm	Root-mean-square Roughness ( $R_q$ ) nm	Maximum peak-to valley height(P-V) nm
(a). Polished carbon steel	108	130	761
(b). 60 ppm $Cl^-$	143	190	1758
(c). 60 ppm $Cl^-$ + 50 ppm $Ni^{2+}$ + 100 ppm 2-CIEPA + 50 ppm of SG	120	147	995

These parameters confirm that the metal surface appears smoother. The smoothness of the surface is due to the formation of a compact protective film of  $Fe^{2+}$ -2-CIEPA complex,  $Fe^{2+}$ -SG and  $Ni(OH)_2$  on the metal surface thereby inhibiting the corrosion of carbon steel. The above parameters observed for carbon steel are somewhat greater than the AFM data of polished metal surface[19]. This confirms the formation of the protective film on the metal surface.

### Mechanism of corrosion inhibition

The Weight – loss study reveals that the formulation consisting of 100 ppm of 2-CIEPA, 50 ppm of  $Ni^{2+}$  and 50 ppm of SG has 98 % inhibition efficiency. Electrochemical studies such as polarization study and AC impedance spectra confirm the formation of a protective film on the metal surface in presence of inhibitors[20-27]. A mechanism of corrosion inhibition proposed based on experimental results.

In order to explain the above observations, the following mechanism of corrosion inhibition is proposed.

- When the environment consisting of 100 ppm of 2-CIEPA, 50 ppm of Ni<sup>2+</sup> and 50 ppm of SG are prepared, there is a formation of Ni<sup>2+</sup> -2-CIEPA complex and Ni<sup>2+</sup> - SG complex in solution.
- When the carbon steel introduced in this solution, there is diffusion of nickel complexes move towards the metal surface.
- On the metal surface, nickel complex is converted into iron complex on the anodic site.  

$$\text{Ni}^{2+} - 2\text{-CIEPA} + \text{Fe}^{2+} \rightarrow \text{Fe}^{2+} - 2\text{-CIEPA} + \text{Ni}^{2+}$$

$$\text{Ni}^{2+} - \text{SG} + \text{Fe}^{2+} \rightarrow \text{Fe}^{2+} - \text{SG} + \text{Ni}^{2+}$$
- The released Ni<sup>2+</sup> combined with OH<sup>-</sup> to form Ni(OH)<sub>2</sub> on the cathodic Sites
- $\text{Ni}^{2+} + 2\text{OH}^- \rightarrow \text{Ni(OH)}_2 \downarrow$
- Thus, protective film consists of Fe<sup>2+</sup> - 2-CIEPA complex, Fe<sup>2+</sup> - SG complex and Ni(OH)<sub>2</sub>.

## CONCLUSION

The present study leads to the following conclusions:

- ✓ The formulation consisting of 50 ppm of Ni<sup>2+</sup>, 100 ppm of 2-CIEPA and 50 ppm of SG offers 98% inhibition efficiency;
- ✓ The synergistic effect exists between 2-CIEPA – Ni<sup>2+</sup>- SG systems. The mixed inhibitors show better inhibition efficiency than individual.  
Polarization study reveals that this formulation controls the cathodic reaction predominantly.
- ✓ AC impedance spectra prove the protective film formation on the carbon steel.
- ✓ FTIR spectra reveals that the protective film consists of Fe<sup>2+</sup> - 2-CIEPA complex and Fe<sup>2+</sup> - SG complex formed on anodic sites of metal surface and Ni(OH)<sub>2</sub> formed on cathodic sites of metal surface.
- ✓ The SEM study proves the protective film formed on the metal surface.
- ✓ The EDS study determines the elements of 2-CIEPA - Ni<sup>2+</sup> - SG system on the carbon metal surface.
- ✓ The AFM study confirms the protective film formed on the metal surface due to the inhibitors system.

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