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Synergistic Influence Of Sodium Meta Vanadate On Corrosion Inhibition Efficiency Of 1-Benzyl-3-Hydroxy-1-H-Indazole On Mild Steel In Aqueous Medium Containing 60 ppm Cl^- Ion

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Abstract : The inhibition efficiency of 1-Benzyl-3-Hydroxy-1-H- Indazole (BHI) –Zn^{2+} system and the synergist sodium meta vanadate (SMV) is controlling corrosion of mild steel in an aqueous solution containing 60 ppm of Cl^- ion has been evaluated by weight loss method 120 ppm of BHI has 71.4% inhibition efficiency addition of 100 ppm of SMV has 82.6% Inhibition efficiency. Synergistic effect exists between inhibitor system and sodium meta vanadate. Mechanistic aspects of corrosion inhibition have been studied by electrochemical studies like polarization and electrochemical impedance spectroscopy. Surface analytical techniques; FT-IR, AFM were carried out for revealing protective film formation. Atomic absorption spectroscopy study showed that the inhibition efficiency increases with increasing inhibitor concentration.
Key words : Synergism, inhibition efficiency, FT-IR, AFM, potentiodynamic polarization study.

Introduction:

Mild steel is an important metal which finds wide application in industries due to its excellent mechanical properties and low cost[1]. The recent trend is towards environment friendly inhibitor[2,23-29]. Trillion of rupiahs have been spent to overcome the corrosion problems[3]. One of the most effective ways to protect the corrosion, especially the internal parts of mild steel pipeline scaling in cooling water system is the use of organic corrosion inhibitors[4,5,6]. Organic inhibitor that exhibit one or more polar functions (N,O,S) and the heterocyclic compounds with polar groups and π electrons have been quite effective in corrosion protection[7]. This inhibition efficiency usually attributed to the specific interactions that occur between functional group and hetero atom with the metal surface due to their lone pair of electrons[8] and the influence on the change of corrosion potential[9]. The absence and presence of the protective film on the surface of metal is responsible for controlling the behavior of corrosion because of its action as a protective barrier against corrosive attack[10,11]. Synergists are still used to increase further efficiency of corrosion inhibitor. In the present work the influence of synergist SMV on a new organic inhibitor BHI and Zn^{2+} ion in controlling the corrosion of mild steel in neutral aqueous environment containing low chloride has been studied by non electrochemical methods and electrochemical methods (potentiodynamic polarization, electrochemical impedance). Surface analytical techniques; FT-IR, AFM were used to investigate the nature of protective film formed on metal surface.

Experimental

1. Material preparation

Mild steel specimen were chosen from same sheet of composition 0.1% C, 0.026% S, 0.06% P, 0.04%Mn and rest iron and the dimensions of 1.0 X 4.0 X 0.2 cm. Carbon steel specimens were mechanically abraded[12] degreased, rinsed, dried and weighed before immersion in the experimental solution. All the chemicals and reagents used were Analar / Sigma Aldrich grade.

2. Weight loss measurements

Weight loss measurements were carried in triplicate in absence and presence of various concentrations of BHI, BHI+Zn^{2+}, SMV, BHI+Zn^{2+}+SMV solution in 60 ppm Cl^- ion at ambient temperature. We evaluated the synergistic effect of SMV on BHI+Zn^{2+} in aqueous media. Illustrated data are mean values of obtained result

$$IE(\%) = \frac{W_1 - W_2}{W_1} X\,100$$

Where W_1 and W_2 are the weight loss of carbon steel in absence and presence of inhibitor respectively.

3. Synergism Parameter (S_I)

Synergism parameter is indication if synergistic effect existing between the inhibitors.

$$SI = \frac{1\text{-}\theta 1+2}{1\text{-}\theta' 1+2}$$

Where θ_1 is surface coverage of SMV, θ_2 is surface coverage of BHI+Zn^{2+}, θ'_{1+2} is combined surface coverage of BHI and SMV.

4. Statistical analysis - F-Test (ANOVA)

Analysis of variance established to know the synergistic effect existing between SMV and inhibitor system is statistically significant[13-15].

5. Electrochemical measurements

Electrochemical measurements were done by means of SOLARTRON CHI760D and Corrware software were used for data acquisition and analysis.

5.1 Potentiodynamic polarization curves

Polarization experiments were carried out in a conventional three- electrode glass cell with platinum counter electrode under saturated calomel electrode as reference with the luggin capillary bridge. The cathode plot was always determined first; the open circuit potential was then reestablished and anode plot was determined. The anodic and cathodic polarization curves were recorded at constant scan rate of 0.01 mV/ sec. Inhibition efficiencies were determined from corrosion current calculated by tafel extrapolation method. The inhibition efficiency (IE) was calculated using the following equation

$$IE\ (\%) = \frac{I_1 - I_2}{I_1} X100$$

Where I_1 and I_2 are the corrosion current density of carbon steel in the absence and presence of inhibitor, respectively.

5.2 Electrochemical impedance spectroscopy

Impedance spectra were obtained in the frequency range of 100 kHz – 0.1 Hz. A sine wave with 5mV amplitude was used to perturb the system[16,17]. SCE was used as a reference and a platinum plate was used as a counter electrode. All potential were reported versus SCE. The charge transfer resistance (R_{ct}) values were obtained from the diameter of the semicircles of the Nyquist plot. (IE) of the inhibitor was calculated from R_{ct} values using the following equation:

$$IE\ (\%) = \frac{R_{ct}(I) - R_{ct}}{R_{ct}\ (I)}\ X100$$

6. Atomic Absorption Spectroscopy

AAS was conducted by using Atomic Absorption Spectrometer (SHIMADZU model –AA-6300), at PSG college of Arts and Science, Central research lab, Coimbatore was used for estimating the amount of dissolved iron in corrodant solution containing 60 ppm Cl^- ion, BHI+Zn^{2+}, BHI+Zn^{2+}+SMV after exposure the mild steel specimen for 3 hours. From the amount of dissolved iron the IE was calculated

$$IE\ (\%) = \frac{B - A}{B} X\ 100$$

Where B is the amount of dissolved iron in absence of inhibitor, A is the amount of dissolved iron in presence of inhibitor.

7. Surface Examination Studies

7.1 Fourier Transforms Infrared (FT-IR) spectroscopy

For FT-IR studies, analytical SHIMADZU MIRacle 10 instrument was used. The spectra of all studied samples were measured in the range of 400-4000 cm^{-1}.

7.2 Atomic Force Microscopy (AFM) analysis of metal surface

Atomic Force Microscopy is a powerful technique for the gathering of roughness statistics from a variety of surfaces. AFM is becoming an accepted method of roughness investigation[18].

Result and Discussion

Gravimetric Analysis

The gravimetric measurements were carried out to calculate the CR and IE for the mild steel in an aqueous solution (60 ppm Cl^- ion) in absence and presence of various concentrations of BHI, BHI+Zn^{2+}, synergist sodium meta vanadate (SMV) and BHI+Zn^{2+}+SMV. The values are tabulated in Table [1-4]. From Table 1 (Fig.1) the IE of BHI (120 ppm) was 61.4% and BHI+Zn^{2+} (120 ppm +20 ppm) (Table 2,Fig. 2) IE were 71.4%. The inhibition efficiency from weight loss studies of SMV (100 ppm) individually was 74.2% (Table 3,Fig. 3).The influence of SMV on the inhibitor system (BHI+Zn^{2+}) showed an increased IE of 82.6% (Table 4,Fig. 4 (a),(b)). This shows synergistic effect existing between the SMV over BHI+Zn^{2+}.

Table 1: Influence of IE on BHI

S.No	Conc. of BHI (ppm)	Conc. of Zn2+ (ppm)	CR (mmpy)	Surface coverage (θ)	IE (%)
1.Blank	0	0	0.2604	-	-
2.	20	0	0.2045	0.2100	21
3.	40	0	0.1822	0.3000	30
4.	60	0	0.1673	0.3570	35.7
5.	80	0	0.1302	0.5000	50
6.	100	0	0.1190	0.5420	54.2
7.	120	0	0.1004	0.6140	61.4
8.	140	0	0.1153	0.5570	55.7
9.	160	0	0.1488	0.4280	42.8

Table 2: influence of IE on BHI and Zn^{2+} system

S.No	Conc. Of SMV (ppm)	CR (mmpy)	Surface coverage (θ)	IE (%)
1 .	0	0.2604	-	-
2.	10	0.09297	0.6430	64.3
3.	20	0.08925	0.6570	65.7
4.	40	0.08739	0.6640	66.4
5.	60	0.08553	0.6700	67
6.	80	0.08180	0.6850	68.5
7.	100	0.06694	0.7420	74.2
8.	120	0.0744	0.7140	71.4
9.	140	0.1059	0.5930	59.3

Table 3: Influence of IE on Various concentrations of SMV

Conc. Of BHI (ppm)	Conc. Of Zn^{2+}									
	0 ppm		20 ppm		40 ppm		80 ppm		120 ppm	
	CR (mmpy)	IE (%)	CR (mmpy)	IE (%)	CR (mmpy)	IE (%)	CR (mmpy)	IE (%)	CR (mmpy)	IE (%)
0	0.2604	-	0.0967	62.8	0.1041	60	0.1116	57.1	0.1190	54.2
20	0.2045	21	0.0948	63.6	0.1023	60.7	0.1078	58.6	0.1152	55.7
40	0.1822	30	0.09297	64.3	0.1004	61.4	0.1041	60	0.1078	58.6
60	0.1673	35.7	**0.08925**	**65.7**	0.0967	62.8	0.09855	62.1	0.1059	59.3
80	0.1302	50	0.08553	67	0.09297	64.3	0.0967	62.8	0.1041	60
100	0.1190	54.2	0.0818	68.5	0.08925	65.7	0.09390	63.9	0.0948	63.6
120	0.1004	**61.4**	**0.0744**	**71.4**	0.0818	68.5	0.08553	67	0.08739	66.4
140	0.1153	55.7	0.1041	60	0.0967	62.8	0.09297	64.3	0.08925	65.7
160	0.1488	42.8	0.1376	47.1	0.1302	50	0.1190	54.3	0.1190	54.2

Table 4: Influence of SMV on IE of inhibitor system (BHI +Zn^{2+})

S.No	Conc. of BHI (ppm)	Conc. of Zn^{2+} (ppm)	Conc. Of SMV (ppm)	CR (mmpy)	Surface coverage (θ)	IE (%)
1. Blank	0	0	0	0.2604	-	-
2.	0	20	0	0.0967	0.6280	62.8
3.	120	0	0	0.1004	0.6140	61.4
4.	120	20	0	**0.0744**	**0.7140**	71.4
5.	120	20	20	0.0669	0.7420	74.2
6.	120	20	40	0.6322	0.7570	75.7
7.	120	20	80	0.05578	0.7850	78.5
8.	120	20	100	0.0446	0.8280	82.6
9.	120	20	120	0.05206	0.8000	80

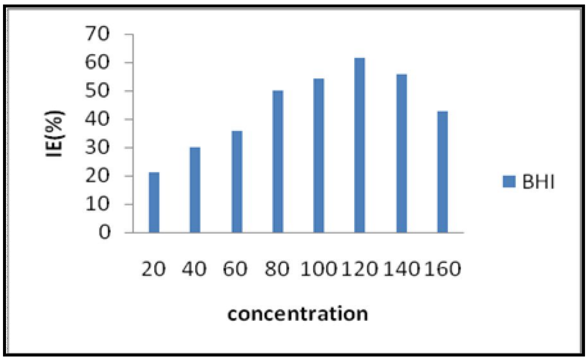


Figure 1: Plot of IE Vs Various concentration of BHI

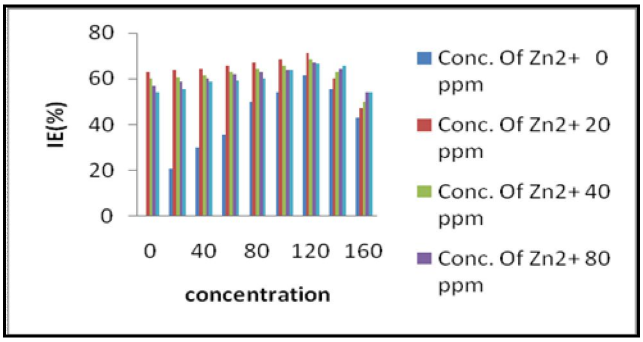


Figure 2: Plot of IE Vs Various concentrations of BHI +Zn^{2+} system

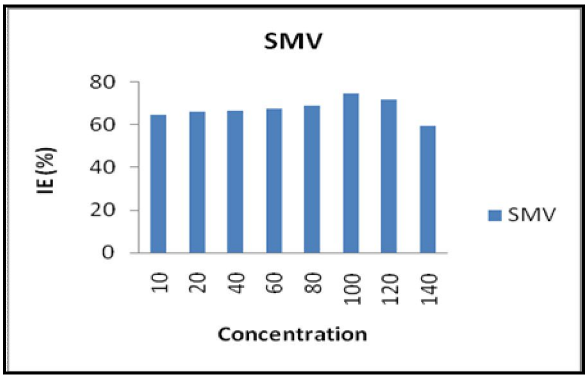
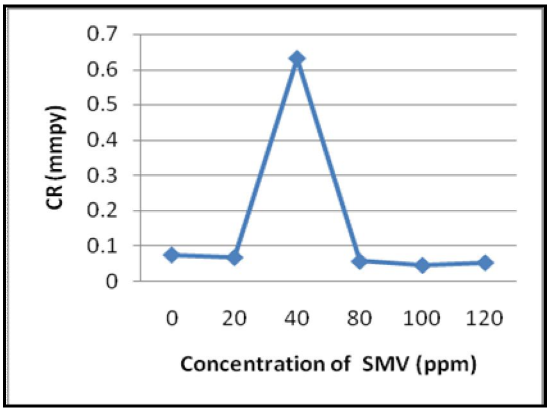


Figure 3: Plot of IE on Various concentrations of SMV



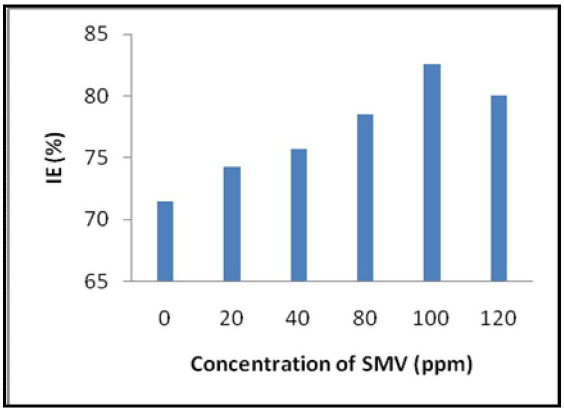


Figure 4:(a) plot of CR Vs Conc.of.SMV **4(b) plot of IE Vs Conc.of.SMV**

Synergism parameter:

The values of synergism parameters are shown in Table 5.When S_I >1, this points to synergistic effect. S_I approaches 1 when no interaction existing between the inhibitor compounds. In the case of S_I <1 negative interaction of inhibitor prevails (CR increases). In present study synergism exists between SMV and BHI+Zn^{2+}system.

Table 5: Synergistic parameters

Source of variance	Sum of squares (SS)	Degree of freedom (df)	Mean square (MS)	F-Test	Level of significance
Between samples	SSC= 349.3	K-1=1	MSC=349.3	FC= MSC/MSE 349.3/6.878 =50.78	P>0.05
Within samples	SSE=55.03	N-K=10-2=8	MSE=6.878		

Table 6: Variance of ANOVA

SMV (θ_1)	Zn^{2+} (θ_2)	BHI + Zn^{2+} +SMV (θ'_{1+2})	S_I $(1-\theta_{1+2})/(1-\theta'_{1+2})$
0.742	0.614	0.742	2.6
0.742	0.628	0.757	3.8
0.742	0.600	0.785	3.8
0.742	0.593	0.828	3.8
0.742	0.571	0.800	3.8

Analysis of variance (ANOVA):

F-Test is used if the synergistic effect exists between inhibitors is statistically significant. The F-value calculated for SMV+ BHI-Zn^{2+} system is 50.78 (Table 6) greater than the critical value 5.32 for 1,8 degrees of freedom at 0.05 level of significance. Hence it is concluded the synergistic effect existing between SMV and BHI-Zn^{2+} is statistically significant.

Electrochemical measurements

Polarization studies

The corresponding electrochemical parameters namely corrosion current densities (I_{corr}), Corrosion potential (E_{corr}), cathodic,anodic Tafel slope (b_c), (b_a) derived from polarization curve are presented in Table 7. From the results I_{corr} value decreases and LPR values increases for inhibitor and SMV compared to blank, this shows adsorption of the inhibitor molecule through active centers[19]. E_{corr} values are shifted more anodically, also Tafel constants b_a and b_c obtained suggest that the inhibitors are mixed type and inhibit corrosion by blocking the active sites[20]. The IE obtained from weight loss measurement for inhibitor system and synergist is an excellent agreement with IE electrochemical study. Though the reaction is anodically predominant the Tafel plot (Fig.5) shifts to cathodic side showing that the inhibitor is mixed type inhibitor.

Table 7: Potentiodynamic polarization study of carbon steel immersed in various test solution

Inhibitor system	Ecorr (mV/SCE)	Tafel polarization parameters				
		Bc (mV/dec)	ba (mV/dec)	Icorr (µA/cm2)	IE (%)	LPR (Ohm/cm2)
Blank	-0.586	4.833	4.960	6.686x10-6	-	6511
BHI+Zn^{2+}	-03541	6.279	4.111	1.966x10-6	71.16	21285
BHI+Zn^{2+}+ SMV	-0.315	6.025	4.370	$1.335X10^{-6}$	80.42	31337

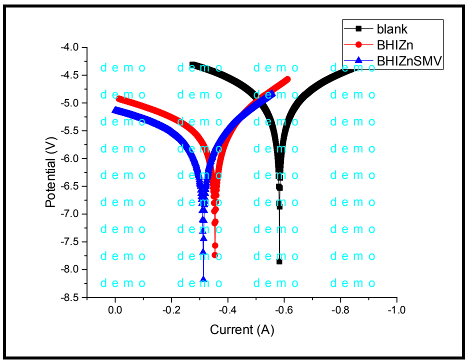


Figure 5: Tafel plot for blank, (BHI 120 ppm + Zn^{2+} 20 ppm), SMV (100 ppm)+ (BHI 120 ppm + Zn^{2+} 20 ppm)

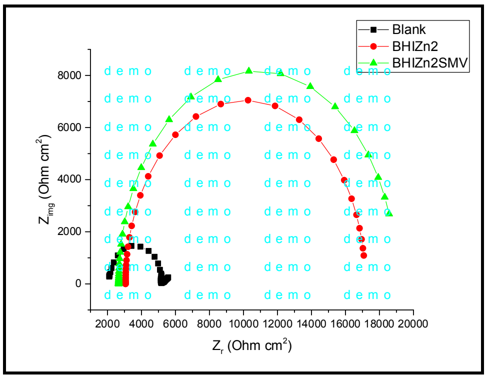


Figure 6: Nyquist plot of carbon steel immersed in various test solution

Electrochemical impedance study

AC impedance has been study to detect the formation of film on the metal surface. If a protective film is formed, the charge transfer resistance increases and double layer capacitance value decreases[21]. The AC impedance spectra of carbon steel immersed in various solutions are shown in Fig.6 and R_{ct}, C_{dl} values in Table 8 which suggests that R_{ct} increases and C_{dl} decreases conforming film formation. The IE found was an agreement with weight loss IE.

Table 8: AC impedance Parameters of carbon steel immersed in various test solution

Inhibitor System	R_{ct} (Ω cm^2)	F(max)	C_{dl} (F cm^{-2})	IE(%)
Blank	3125.759	1555.508	$3.275X10^{-8}$	-
BHI+Zn^{2+}	14123.752	7035.141	$1.601X10^{-8}$	77.9
BHI+Zn^{2+}+SMV	16442.209	8089.794	$1.19 X10^{-8}$	81

Table 9: Amount of dissolved mild steel present in the corrosive solution with and without inhibitors

Name	concentration (ppm)	Amount of mild steel in corrodant (mg/l)	Inhibition efficiency (%)
Blank	60 ppm Cl^- ion	1.31	-
Cl^- +BHI +Zn^{2+}	60 ppm + 120 ppm +20 ppm	0.31	76
Cl^- +BHI +Zn^{2+} +SMV	60 ppm + 120 ppm +20 ppm+100 ppm	0.11	92.3

Atomic Absorption Spectroscopy

The IE of the inhibitor system and SMV towards the dissolution iron was calculated by measuring the dissolved iron in the corroding solution with and without inhibitor and SMV. From results presented in Table 9, increase in concentration of inhibitor decrease the amount of dissolved iron resulting in increased IE. The IE obtained by this technique was in good agreement with that obtained from conventional method.

FT-IR Spectroscopy

The FT-IR Spectra (KBr pellet) of the film formed on the carbon steel surface after immersion in the solution containing 100 ppm of SMV, 120 ppm of BHI and Zn^{2+} shown in figure 7. The OH Stretching frequency has been shifted from 3352cm^{-1} to 3326 cm^{-1} and N-H stretching shifted from 3352 cm^{-1} to 3355 cm^{-1}.The VO_3^- stretching frequency of SMV shifted from 1382 cm^{-1} 1385 cm^{-1} resulting in the formation of Fe^{2+}-SMV complex[22]. The N=N peak shifted from 1638 cm^{-1} to 1640 cm^{-1}and C=C stretching frequency shifted from1637 cm^{-1} to1643 cm^{-1}. The peak at 1637 cm^{-1} is due to Zn $(OH)_2$.

The FT-IR suggest that the protective film may consists of [Fe^{2+}/Zn^{2+}-SMV –BHI], $Zn(OH)_2$ and considerably less amount of oxides & hydroxides of Iron.

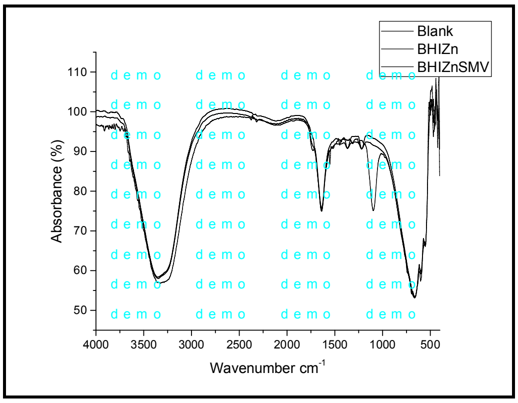


Figure 7: FTIR spectra for blank, (BHI 120 ppm + Zn^{2+} 20 ppm), SMV (100 ppm)+ (BHI 120 ppm + Zn^{2+} 20 ppm)

Atomic Force Microscopy

In the presence and absence of inhibited system the 2D,3D AFM morphology for polished metal, mild steel metal surface immersed in 60 ppm Cl^- ion, (Cl^-+BHI+Zn^{2+}) system and Cl^-+BHI+Zn^{2+}+SMV and their R_q , Ra,(P-V) are shown in Figure 8 (a), (b), (c) and (d) and Table 10.

Table 10: AFM data for mild steel metal surface immersed in inhibited and uninhibited environments

Samples	RMS (Rq) Roughness (nm)	Average (Ra) Roughness (nm)	Max. Peak to Valley Height (nm)
Polished mild steel (control)	117.798	91.6251	839.505
Mild steel immersed in 60 ppm Cl-ion (blank)	166.961	129.812	1570.22
Mild steel immersed in 60 ppm Cl- +BHI (120 ppm)+Zn2+ (20 ppm)	140.612	113.027	900.148
Mild steel immersed in 60 ppm Cl- +BHI (120 ppm)+Zn2+ (20 ppm)+SMV(100 ppm)	77.564	55.86	816.707

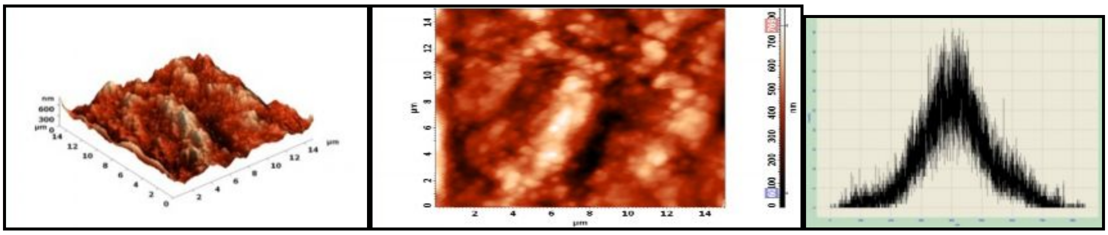


Figure 8 (a): AFM images for polished mild steel

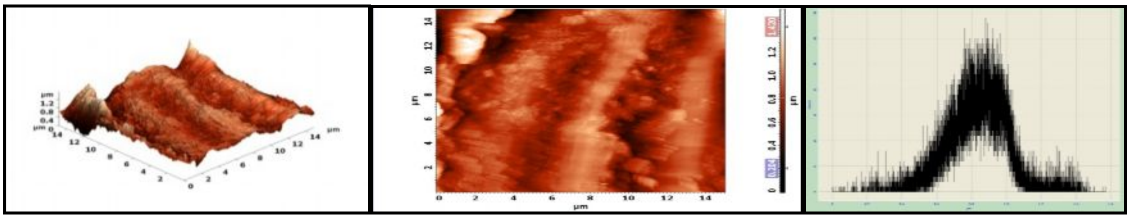


Figure 8 (b): AFM images for blank

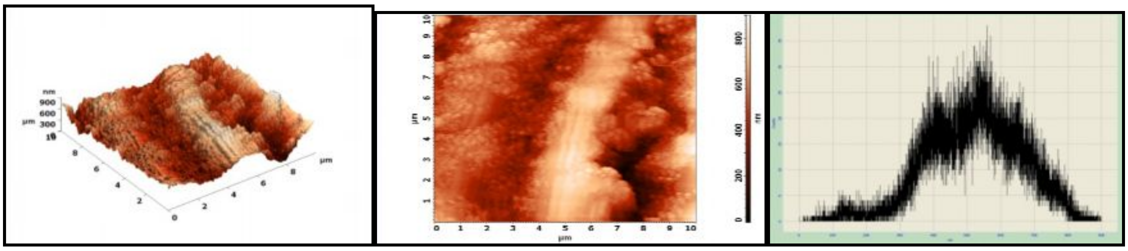


Figure 8 (c): AFM images for inhibitor system (BHI +Zn^{2+})

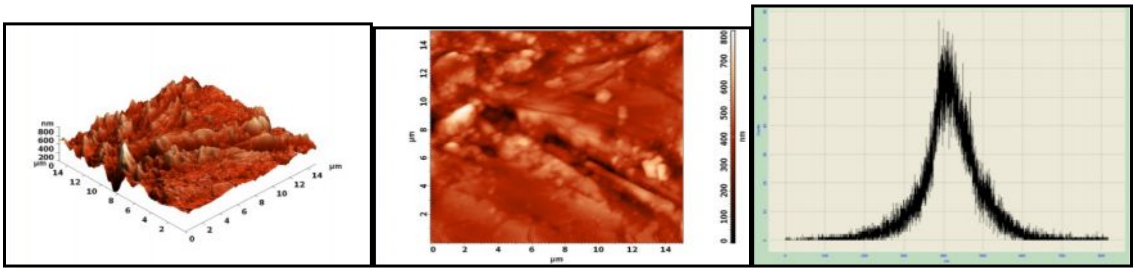


Figure 8 (d): AFM images for inhibitor system (BHI +Zn^{2+}) and SMV

The average roughness (R_a), root mean square roughness R_q and the maximum peak-to-valley(P-V) height value,for polished and inhibited system were found to be less when compared to that of corroded system (blank) conforming the protective film formation in2D,3D images as shown in Figure 8 (a) (c),(d).

Conclusion

The Present study leads to the following conclusion: the formulation consisting of [120ppm BHI+20ppm Zn^{2+}] and[100ppm SMV] offers 82.6% IE to mildsteel immersed in aqueous solution containing 60ppm cl^{-1}.EIS studies shows the mixed inhibitor character ,and protective film formation on the mildsteel surface. FT-IR reveal that protective film consists of Fe^{2+}+SMV complex and Fe^{2+}+BHI complex. AFM also confirm protective film formation and AAS showed that the inhibition efficiency increases with increasing inhibitor concentration.

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