Electrochemical and Surface Analysis Studies on Corrosion Inhibition of Carbon Steel in Well Water By Curcumin

[1]R.Epshiba, [2]A.Peter Pascal Regis, [3]S.Rajendran

[1, 2]Department of Chemistry, St.Joesph's College, Trichy, Tamil Nadu,
[3]Department of Chemistry, RVS School of Engineering and Technology, Dindigul, Tamil Nadu
Email: [1]hephzi_r@aol.com

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Abstract—Sodium molybdate in the presence of Zn^{2+} ions is used to function as the corrosion inhibitors in the corrosion of carbon steel. The green inhibitor, curcumin was added to SM and Zn^{2+} at low concentrations to increase the inhibition efficiency. In this paper, detailed studies on curcumin as second synergist in the formulation containing relatively low levels of sodium molybdate (SM) and Zn^{2+} in the corrosion inhibition of carbon steel are presented. Thus, the ternary system containing 100 ppm of SM, 25 ppm of Zn^{2+} and 250 ppm curcumin is quite effective. AC impedance studies indicate that a protective film is formed on the metal surface. Potentiodynamic polarization studies show that this system works as an anodic inhibitor and controls the anodic reaction predominantly. FTIR spectra reveals that the protective film consists of SM-Fe^{2+}, Fe^{2+}-curcumin complex and $Zn(OH)_2$. The protective film formed on the metal surface are further analysed with the help of SEM and AFM.

Index Terms— Corrosion inhibition, Carbon steel, Sodium molybdate, curcumin, Synergistic effect.

I. INTRODUCTION

Corrosion is the deterioration of metal by chemical attack or reaction with its environment. It is a constant and continuous problem, often difficult to eliminate completely. Prevention of corrosion includes the usage of corrosion inhibitors, which form protective layers on the metal surface. The term green corrosion inhibitor or eco-friendly inhibitor refers to the substances that are biocompatibile in nature, environmentally acceptable, readily available and renewable source. Hence, the extracts of some common plants based chemicals and their by-products have been tried as inhibitors for metals under different environments[1-3]. A lot of natural products have been previously used as corrosion inhibitors for different metals in various environments [4-7]. In the present research work a "yellow coloring-matter" from the rhizomes of Curcuma longa (turmeric) named as curcumin has been taken as a corrosion inhibitor for carbon steel in well water. Johnsirani et al., have used an aqueous extract of curcumin dye, in controlling corrosion of carbon steel in sea water [8] Elmsellem. H et al., [9] investigated the inhibition effect of curcumin on the corrosion of mild steel in 1.0 M HCl solution. The present study evaluated the synergistic effect of the Sodium molybdate(SM)-Zn^{2+} system and estimated the influence of Curcumin(CM) on the inhibition efficiency [IE] of the SM-Zn^{2+} system. The mechanistic aspects of corrosion inhibition were evaluated by electrochemical studies and analysis of protective film by FTIR spectra, SEM and AFM techniques.

II. EXPERIMENTAL METHODS

A. Weight loss method

Stainless steel specimens of dimensions 4.0 X 1.0 X 0.2 cm, polished to a mirror finish and degreased with trichloroethylene in triplicate were immersed in 100 ml of well water and well water containing various concentrations of the inhibitors for 3 days. The weights of the specimens before and after immersion were determined using an analytical balance, Shimadzu AY62 model. Then the inhibition efficiency (IE) and corrosion rate (CR) was calculated using the equation.

$$IE = 100\,[1-(W_2/W_1)]\ \%$$

where W_1 is the corrosion rate in the absence of inhibitor and W_2 is the corrosion rate in the presence of inhibitor. Corrosion rate was calculated using the formula

Millimetre per year = 87.6 W / DAT

W = weight loss in milligrams

D = density of specimen g/cm^3 = 7.87 gm/cm^3

A = area of specimen = $10cm^2$ and

T = exposure in hours = 72 hrs.

B. Electrochemical and Impedance Measurements

Polarization and AC impedance studies were carried out in same Electrochemical Impedance Analyzer model CHI 660A using a three electrode cell assembly. The working electrode was used as a rectangular specimen of carbon steel with one face of the electrode of constant 1cm^2 area exposed. A saturated calomel electrode (SCE) was used as reference electrode. A rectangular platinum foil was used as the counter electrode. Polarization curves were recorded after doing iR compensation. The corrosion parameters such as Tafel slopes (anodic slope b_a and cathodic slope b_c), corrosion current (I_{Corr}) and corrosion potential (E_{Corr}) values were calculated. The AC impedance parameters charge transfer resistance (R_f) and the double layer capacitance (C_{dl}) were calculated.

C. Surface characterization studies

FTIR Spectra were recorded in a Perkin - Elmer spectrum spectrophotometer. The surface morphology measurements of the carbon steel were examined by SUPRA 55 Field Emission Scanning Electron Microscope (FESEM). All SEM micrographs of carbon steel are taken at a magnification of X=2500. Atomic force microscope (AFM) using (NT-MDT Ireland model) was used to observe the sample's surface in tapping mode, using cantilever with Silicon nitride, Single crystal, N-type tips. The scanned area in the images are 3 µm x 3 µm at the scan rate of 1 Hz/second. All solutions were prepared using well water collected from chennai, Tamil Nadu, India.

III. RESULT AND DISCUSSION

A. Analysis of Result of the weight loss method

The inhibition efficiencies (IE) and corrosion rates(CR) of sodium molybdate(SM)-Zn^{2+}-Curcumin system in controlling the corrosion of carbon steel immersed in well water for a period of 3 days in the absence and the presence of inhibitors is given in Table 1 and is shown in Fig.1. In order to examine the role of curcumin in the ternary inhibitor formulation, experiments were conducted with curcumin alone and with SM(100ppm) and Zn^{2+}(25ppm) in a wide concentration range.

Table 1. Corrosion Rates (CR) and Inhibition efficiencies (IE) of Carbon Steel in Well Water, in the absence and the presence of Inhibitors obtained by Weight-Loss Method

SM (ppm)	Zn^{2+} (ppm)	Curcumin (ppm)	IE (%)	CR (mm/y)
0	0	0	-	0.1174
100	0	0	22	0.0986
100	25	0	23	0.0903
0	0	250	38	0.0727
100	25	50	70	0.0352
100	25	100	75	0.0293
100	25	150	81	0.0223
100	25	200	86	0.0164
100	25	250	89	0.0129
100	0	250	54	0.0540
0	25	250	47	0.0622

It is observed that, 100 ppm of SM and 25 ppm Zn^{2+} has 23% IE and 250ppm of Curcumin has 38% IE. But the formulation consisting of 100 ppm of SM, 25ppm Zn^{2+} and 250ppm of Curcumin has 89%. However, such an efficiency is not obtained with combinations of SM and Curcumin even at relatively high concentrations. Thus, it may be concluded that Zn^{2+} is the primary synergist and Curcumin is the secondary synergist and both play a significant synergistic role. Hence, the highest IE is obtained at such low concentrations of each of the components in the ternary inhibition formulation. It also shows that the corrosion rate decreases as the concentration of Curcumin increases [10].

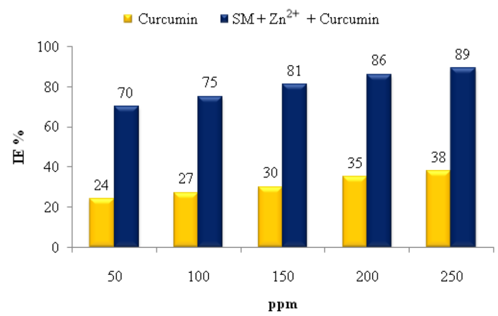


Fig.1. Inhibition efficiency (IE) of SM- Zn^{2+}-Curcumin system in the corrosion of carbon steel immersed in well water (Immersion period-3 days)

B. Analysis of polarization curves

The polarization study has been used to investigate the formation of protective film on metal surface [11,12]. The polarization curves of carbon steel immersed in well water in the presence and absence of inhibitors are shown in Fig 2. The corrosion parameters are given in Table 2. When carbon steel is immersed in well water environment, the corrosion potential is –763 mV vs SCE. The formulation consisting of 100 ppm of SM, 25ppm of Zn^{2+} and 250ppm of Curcumin shifts the corrosion potential to -713 mV vs SCE. This suggests that this formulation controls the anodic reaction predominantly. Both the anodic and cathodic Tafel slopes are shifted in the presence of the inhibitor. However, a shift of anodic Tafel slope (from 148 mV /dec to 268 mV /dec) is much higher as compared to a shift of the cathodic Tafel slope(from64 mV /dec to 141 mV /dec) for the blank and SM (100ppm)- Zn^{2+}(25ppm)- Curcumin (250ppm) formulations. Thus, it can be concluded that this inhibitor formulation acts as an anodic inhibitor and controls the anodic reaction predominantly.

Table 2. Corrosion parameters of carbon steel immersed in well water in the absence and presence of SM-Zn^{2+}-Curcumin system obtained from polarization method

System	E_{corr} mV vs SCE	b_a mV/dec	b_c mV/dec	LPR cm^2	I_{corr} A/cm^2
Well Water (WW)	-763	148	64	496.9	3.92 x 10^{-5}
WW + SM (100ppm) + Zn^{2+} (25ppm) + CM (250ppm)	-713	268	141	1584.8	2.010 x 10^{-5}

When carbon steel immersed in well water, the corrosion current, I_{corr} is $3.92x10^{-5}$ A/cm^2. When SM (100ppm)-Zn^{2+} (25 ppm)-Curcumin (250ppm) is added to well water, the Corrosion Current decreases to 2.010 $x10^{-5}$ A/cm^2. The significant reduction in corrosion current indicates a decrease in corrosion rate in the presence of the inhibitor. Linear polarization resistance (LPR) value increases from 496.9 cm^2 to 1584.8 cm^2. This indicates that the protective film is formed on the metal surface [13,14].

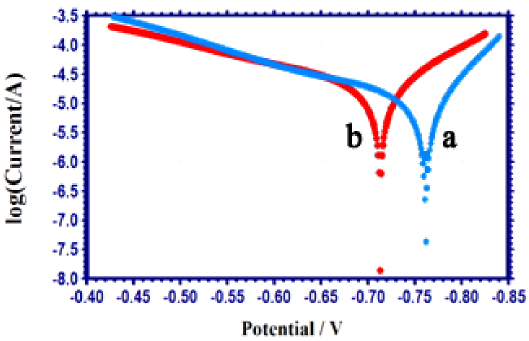


Fig. 2 Polarization curves of carbon steel immersed in various test solutions

a) Well water

b) Well water containing 100ppm of Sodium molybdate, 25ppm of Zn^{2+} and 250ppm of Curcumin

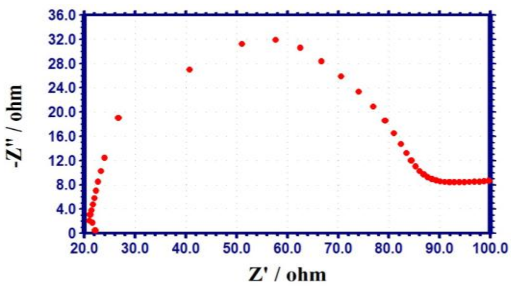
C. Analysis of AC Impedance spectra

AC impedance spectra have been used to detect the formation of the film on the metal surface [15,16]. The Nyquist plots are shown in Fig 3. The AC Impedance parameters are given in Table 3. If a protective film is formed, the charge transfer resistance increases and double layer capacitance value decreases [17,18].

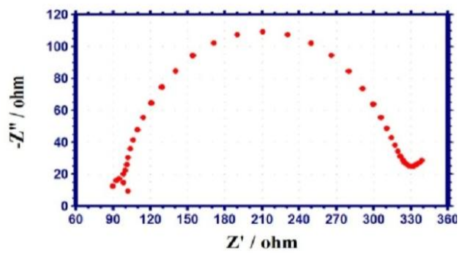
When carbon steel is immersed in well water, the R_t value is 79.71 ohm cm^2 and C_{dl} value is 5.21 x 10^{-8} F cm^{-2}. When the SM (100ppm), Zn^{2+} (25ppm) and curcumin (250ppm) are added to well water, the R_t value increases from 79.71 ohm cm^2 to 247.12 ohm cm^2 and C_{dl} value decreases from $5.21x10^{-8}$ F cm^{-2} to $1.679x10^{-8}$ F cm^{-2}. This suggests that a protective film is formed on the surface of the metal and this accounts for the high inhibition efficiency of SM-Zn^{2+} -Curcumin system.

Table 3 Corrosion parameters of carbon steel in well water in the absence and presence of inhibitors, obtained by AC Impedance method

System	R_t, (ohm cm^2)	C_{dl}, (F cm^{-2})
Well water (WW)	79.71	$5.21x10^{-8}$
WW + SM + Zn^{2+} + SM	247.12	$1.679x10^{-8}$



(a)



(b)

Fig. 3 Nyquist plots of (a) Carbon steel immersed in well water (b) Carbon steel immersed in well water + SM (100ppm) + Zn^{2+} (25ppm) + Curcumin(250ppm)

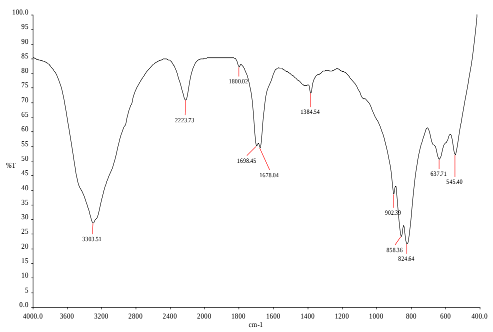
D. Analysis of FTIR spectra

FTIR spectra have been used to analyze the protective film formed on metal surface [19,20]. FTIR spectrum of pure sodium molybdate is given in Fig. 4a. The Mo-O stretching frequency appears at 824 cm^{-1}.

FTIR spectrum of the Curcumin is shown in Fig. 4b. The –OH stretching frequency appears at 3508 cm^{-1}. The C=O stretching frequency appears at 1739 cm^{-1}. The asymmetrical C-O-C stretching frequency of aryl alkyl ethers appears at 1276 cm^{-1}. The band at 1026 cm^{-1} corresponds to the symmetrical C-O-C stretching of alkyl aryl ether [21].

The FTIR spectrum of the protective film formed on the surface of the metal after immersed in the solution containing 100ppm of SM, 25ppm of Zn^{2+} and 250ppm of curcumin is shown in Fig. 4c. The stretching frequency of Mo-O has been shifted to 872 cm^{-1}. This indicates that the oxygen atom of molybdate has coordinated with Fe^{2+} formed on the metal surface resulting in the formation of Fe^{2+}-MoO_4^{2-} complex on the anodic sites of the metal surface.

It is found that the -OH has shifted from 3508 cm^{-1} to 3406 cm^{-1}. The C=O stretching frequency has decreased from 1739 cm^{-1} to 1708cm^{-1}. The asymmetrical C-O-C stretching frequency of alkyl aryl ether has disappeared. The symmetrical C-O-C stretching of alkyl aryl ether has shifted from 1026 cm^{-1} to 1024 cm^{-1}. It was inferred that curcumin has coordinated with Fe^{2+} through the phenolic oxygen, ethereal oxygen, and carbonyl oxygen resulting in the formation of the Fe^{2+}-Curcumin complex on the anodic sites of the metal surface. The peak at 532cm^{-1} is due to Zn-O band. The peak at 3406cm^{-1} is due to –OH stretching. Hence it is confirmed that $Zn(OH)_2$ is formed on the cathodic sites of the metal surface [22]. Thus, the FTIR spectral study leads to the conclusion that the protective film consists of the Fe^{2+}-SM complex, Fe^{2+}-CM complex and $Zn(OH)_2$ [23].



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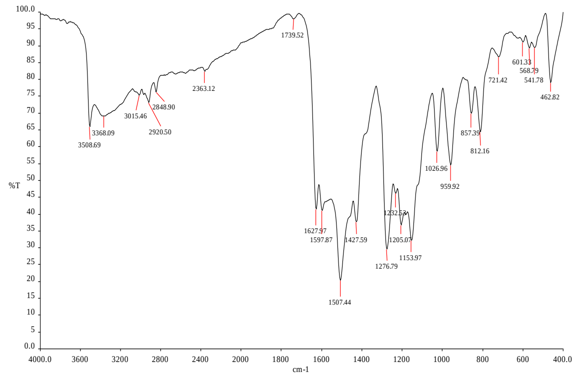

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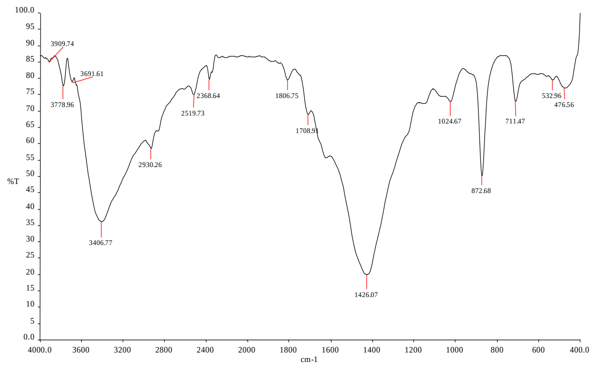


(c)

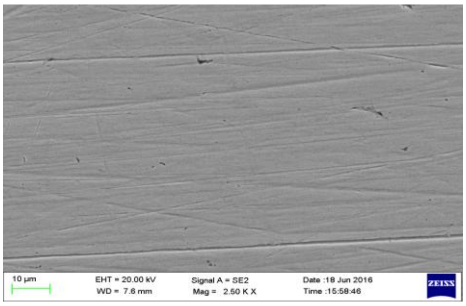
Fig. 4 FTIR Spectra of (a) Pure sodium molybdate (b) Pure Curcumin (c) Film formed on metal surface after the immersion in well water containing SM (100ppm), Zn^{2+} (25ppm) and Curcumin (250ppm)

E.SEM analysis of metal surface

SEM provides a pictorial representation of the surface. To understand the nature of the surface film in the absence and the presence of inhibitors and the extent of corrosion of carbon steel, the SEM micrographs of the surface are examined [24,25]. The SEM images of magnification(X 2500) of carbon steel specimen and carbon steel specimen immersed in well water for one day in the absence and presence of inhibitor system are shown in Fig.5 as images (a,b,and c) respectively.

The SEM micrographs of polished carbon steel surface (control) in Fig.5a illustrate the smooth surface of the metal. These show the absence of any corrosion products formed on the metal surface. The image 5b denote the SEM micrographs of carbon steel surface immersed in well water. They show the type of rough surface characteristic of the uniform corrosion of the metal surface in well water, indicating in an inhibitor free solution, the surface is highly corroded.

The image 5c confirms that in the presence of 100ppm of SM, 25ppm of Zn^{2+} and 250ppm curcumin in well water, the rate of corrosion is suppressed, as can be seen from the decrease in corroded areas. This is as a result of the formation of insoluble complex on the surface of the metal (SM-Zn^{2+} and Fe^{2+}-CM inhibitor complex) and the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of carbon steel [26,27].



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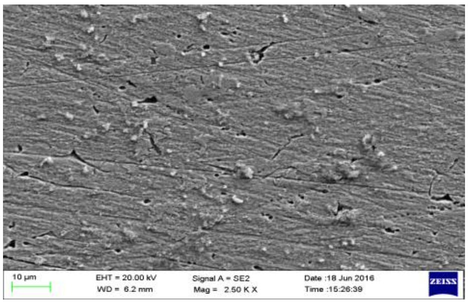

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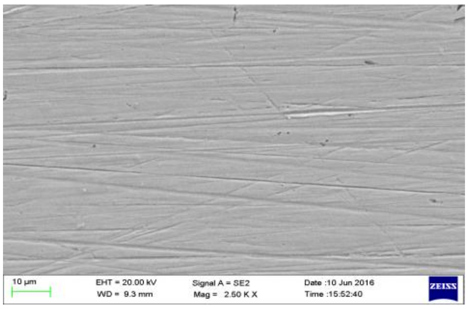


(c)

Fig. 5 SEM analysis of (a) Carbon steel Magnification X 2500 (control) (b) Carbon steel immersed in well water Magnification X 2500(blank) (c) Carbon steel immersed in well water containing 100ppm SM-25ppm of Zn^{2+}-250ppm Curcumin Magnification X 2500

F.AFM analysis of metal surface

AFM is a powerful technique to investigate the surface morphology at nano–to micro–scale and has become a new choice to study the influence of inhibitor on the generation and the progress of the corrosion at the metal/solution interface [28,29]. AFM image analysis was performed to obtain the average roughness, R_a (the average deviation of all points roughness profile from a mean line over the evaluation length), root-mean-square roughness, R_q (the average of the measured height deviations taken within the evaluation length and measured from the mean line) and the maximum peak-to-peak(P-P) height values (largest single peak-to-peak height in five adjoining sampling heights) [30]. R_q is much more sensitive than R_a to large and small height deviations from the mean [31].

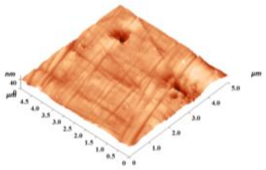
Table 4 is the summary of the average roughness (R_a), rms roughness (R_q) maximum peak-to-peak height (P-P) value for carbon steel surface immersed in different environments. The three dimensional (3D) AFM morphologies for polished carbon steel surface (reference sample), carbon steel surface immersed in well water (blank sample) and carbon steel surface immersed in well water containing SM(100 ppm)-Zn^{2+}(25 ppm)-Curcumin(250ppm)are shown in Fig.6a, 6b and 6c.

The value of Rq, Ra and P-P height for the polished carbon steel surface (reference sample) are 2.25382 nm, 1.76027nm and 22.6017nm respectively, which shows a more homogeneous surface, with some places in which the height is lower than the average depth. Fig. 6a displays the uncorroded metal surface. The slight roughness observed on the polished carbon steel surface is due to atmospheric corrosion. The rms roughness, average roughness and P-P height values for the carbon steel surface immersed in well water are 18.1644nm, 14.9076nm and 111.572nm respectively. These data suggest that carbon steel surface immersed in well water has a greater surface roughness than the polished metal surface. This shows that the unprotected carbon steel surface is rougher and is due to the corrosion of the carbon steel in well water. Fig.6b displays the corroded metal surface with few pits.

Fig.6c shows the steel surface after immersion in well water containing SM (100ppm) + Zn^{2+}(25ppm) + Curcumin (250ppm). The presence of SM (100ppm) + Zn^{2+} (25ppm) + Curcumin (250ppm) in well water reduces the R_q by a factor of 4.92532 nm from 18.1644 nm and the average roughness is significantly reduced to 3.41842 nm when compared with 14.9076 nm of carbon steel surface immersed in well water. The maximum peak-to-peak height also reduced to 55.2593 nm from 111.572 nm. These parameters confirm that the surface appears smoother. The smoothness of the surface is due to the formation of a compact protective film of Fe^{2+}-SM complex and Fe^{2+}-Curcumin complex and $Zn(OH)_2$ on the metal surface thereby inhibiting the corrosion of carbon steel. Also the above parameters observed are somewhat greater than the AFM data of polished metal surface which confirms the formation of the film on the metal surface, which is protective in nature.

Table 4. AFM data for carbon steel surface immersed in inhibited and uninhibited environments

Samples	**RMS (R_q) Roughness (nm)**	**Average (R_a) Roughness (nm)**	**Maximum peak-to-peak height (nm)**
Polished carbon steel (control)	2.25382	1.76027	22.6017
Carbon steel immersed in well water (blank)	18.1644	14.9076	111.572
Carbon steel immersed in well water containing SM (100ppm) + Zn^{2+}(25ppm)+ CM(250ppm)	4.92532	3.41842	55.2593



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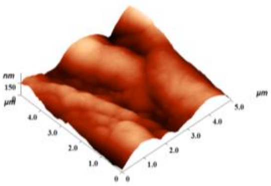

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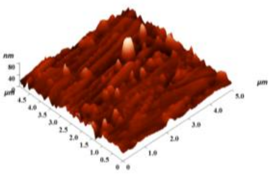

(c)

Fig. 6. Three dimensional AFM images of the surface of (a) Polished carbon steel (control) (b) Carbon steel immersed in well water (blank) (c) Carbon steel immersed in well water containing SM(100ppm) + Zn^{2+} (25ppm) + Curcumin(250ppm)

G. Mechanism of Corrosion Inhibition

In order to explain the experimental results, the following mechanism of corrosion inhibition is proposed. The mechanistic aspect of the inhibition of carbon steel in well water by SM- Zn^{2+} and Curcumin can be explained in terms of complexation and adsorption.

1) Before immersion of carbon steel in well water environment, Zn^{2+}, SM and curcumin- Zn^{2+}form complexes, viz., Zn^{2+}-SM, Zn^{2+}-Curcumin, and Zn^{2+}-SM-Curcumin respectively. These complexes are in equilibrium in the solution with free Zn^{2+}, SM, and curcumin ions.

2) During the immersion of carbon steel in this solution, the Zn^{2+}–SM, Zn^{2+}-Curcumin complexes diffuse from the bulk of the solution towards metal surface that reduces metal dissolution; this may be due to adsorption and complex formation at the surface with the combined application of SM, Zn^{2+} and Curcumin.

3) The corresponding anodic and cathodic reactions of the metal can be generalized as follows: The Zn^{2+}–SM, Zn^{2+}-Curcumin complex diffuses from the bulk solution to the surface of the metal and is converted into a Fe-SM complex, Fe-Curcumin complex which is more stable than Zn^{2+}-SM, Zn^{2+}-Curcumin . The released Zn^{2+} causes $Zn(OH)_2$ precipitation at the local cathodic sites.

4) Hence, Zn^{2+} inhibits the local cathodic region and the local anodic region is inhibited by curcumin and SM. Thus, the protective film formed on the metal surface consists of Fe^{2+}-SM complex, Fe^{2+}-curcumin complex and $Zn(OH)_2$.

5) The FTIR spectra of the surface film suggests the formation of these complexes and the presence of $Zn(OH)_2$ in the surface.

IV. CONCLUSION

A formulation consisting of SM , Zn^{2+} and curcumin can be used as a potent inhibitor to prevent the corrosion of carbon steel in well water. The ternary system SM (100ppm)-Zn^{2+}(25ppm)-curcumin(250 ppm) is effective and has 89% IE. Significant synergism was attained by the combined application of SM-Zn^{2+}-curcumin. Polarization studies reveal that SM-Zn^{2+}-curcumin functions as an anodic inhibitor and controls the anodic reaction predominantly. FTIR spectra reveals that the protective film consists of Fe^{2+}-SM complex and Fe^{2+}-curcumin complex and $Zn(OH)_2$. AC impedance spectra suggests that a protective film is formed on the metal surface and it is confirmed by the SEM micrographs and AFM images.

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