Inhibitive action of the catechol-zinc system in controlling the corrosion of carbon steel

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Received December 17, 2010; Accepted January 4, 2011

The inhibition efficiencies of inhibitor systems constituting various combinations of catechol and zinc ions in controlling corrosion of carbon steel immersed in ground water for 3 days was evaluated by the weight-loss method. 100 ppm of Zn^{2+} has only 15% inhibition efficiency (IE). When 100 ppm of catechol was added, the IE increased to 90%. Weight-loss method, polarization studies and AC impedance spectra were employed. The nature of the protective film formed on the metal surface was analyzed by FTIR spectroscopy. The protective film was found to consist of Fe²⁺ - catechol complex. Synergism parameters and analysis of variance (ANOVA) were used to evaluate the synergistic effect existing between the inhibitors.

Keywords: Carbon steel, corrosion inhibition, catechol, F-test, synergistic effect, FTIR.

1. INTRODUCTION

Corrosion and scaling of carbon steel may be inhibited by the use of inhibitors. It has been observed that the effect of corrosion inhibitors is always caused by change in the state of the protected surface due to adsorption or formation of hardly soluble compounds with metal cations. A review including extensive listing of various types of organic inhibitors has been published [1]. The most often used corrosion inhibitors are nitrogen, sulphur, oxygen and phosphorous containing compounds [2-6]. These compounds get adsorbed onto the metal from the bulk of the environment and form a film at the metal surface. The inhibition efficiency (IE) increases in the order O<N<S<P [7]. The corrosion inhibition of metals in acidic media by different types of organic compounds has been widely studied [8-13]. The inhibition action of organic molecules is primarily due to their adsorption on the surface of the metal through the presence of active centres. Rodge et al. [14] have evaluated the effect of phenol on the corrosion of mild steel in nitric acid of various concentrations. The percent loss in weight was found to increase linearly with increase in acid concentration. The IE values were ordered as follows: p-cresol>mcresol>2-naphthol>phenol>1-naphthol>2-

nitrophenol. Muller *et al.* have studied the use of phenol and substituted phenols such as amino phenols and nitro phenols for aluminium pigment in acid and alkaline medium as corrosion inhibitors

[15]. Viswanathan et al. [16] have studied the inhibitory action of methoxy phenol (MPH) and nonyl phenol (NPH) on the corrosion of N80 steel in 15% HCl. MPH and NPH have shown maximum inhibition at about 83 and 78% inhibitor concentration in the acid, respectively, after 6 h exposure test at ambient temperature. Kulkarni et al. [17] have studied the inhibition of the corrosion of mild steel in nitric acid, sulphuric acid and hydrochloric acid media by phenols. The trend observed was: p-cresol>m-cresol>phenol>2naphthol>1-naphthol>2-nitrophenol. Among naphthols, 2-naphthol was stronger inhibitor than 1naphthol. In general, 2-naphthol and p-cresol were found to be stronger over the range of concentrations of acids used.

The corrosion inhibition characteristics of *m*nitrophenol- Zn^{2+} , thiophenol- Mn^{2+} in ground water and hydroquinone- Zn^{2+} in well water was studied by Benita *et al.* [18-20] in controlling the corrosion of carbon steel. Most of the industries require water for cooling purpose. The major problems in the industrial use of the cooling water systems are the corrosion of the metal equipment and the scale formation. Hence, an attempt was made to assess the inhibition efficiency of catechol with zinc ions in controlling the corrosion of carbon steel in ground water.

The aim of the present study was:

1. To evaluate the inhibition efficiency of catechol in controlling the corrosion of carbon steel immersed in ground water in the presence and absence of zinc ions;

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2. To evaluate the synergistic effect of catechol and zinc ions by determining synergism parameters;

3. To investigate whether the synergistic effect established for this inhibitor system is statistically significant or not by means of the F-test using analysis of variance (ANOVA);

4. To propose a suitable mechanism of corrosion inhibition based on the results obtained from the weight-loss method, FTIR, AC impedance and polarization studies.

2. METHODS AND MATERIALS

Carbon steel specimens (containing 0.03% S, 0.05% P, 0.5% Mn, and 0.15% C) of dimensions $1.0 \times 4.0 \times 0.2$ cm were used in the weight-loss study. Carbon steel encapsulated in Teflon, polished to mirror finish and degreased with trichloroethylene, with surface area of the exposed metal surface 1 cm², was used in the electrochemical studies.

The experiments were carried out at room temperature (36° C). Three carbon steel specimens were immersed in 100 ml of solutions containing ground water and various concentrations of catechol in the absence and presence of Zn^{2+} (ZnSO₄.7H₂O) for a period of 3 days. The weights of the specimens before and after immersion were determined using a Shimadzu balance AY62. Inhibition efficiency (IE) was calculated from the relationship IE = (1-W₂/W₁) × 100, where W₁ = corrosion rate in the absence of inhibitor, and W₂ = corrosion rate in the presence of inhibitor.

After being immersed in the test solutions, the specimens were taken out and dried. The nature of the film formed on the surface of the metal specimens was analyzed by surface analysis technique. IR spectra were recorded with the Perkin Elmer 1600 spectrophotometer. The FTIR spectrum of the protective film was recorded by carefully removing the film, mixing it with KBr and preparing a pellet.

The polarization study was carried out in an H and CH electrochemical workstation impedance analyzer Model CHI 660A provided with iR compensation facility, using a three-electrode cell assembly. Carbon steel was used as working electrode, platinum as counter electrode and saturated calomel electrode (SCE) as reference electrode. After having done iR compensation, polarization study was carried out at a sweep rate of 0.01 V/s. The corrosion parameters such as linear polarization resistance (LPR), corrosion potential E_{corr} corrosion current I_{corr} and Tafel slopes (b_c and

 b_a) were measured. During the polarization study, the scan rate (V/s) was 0.01; hold time at Ef (s) was zero and quiet time (s) was 2. The values of I_{corr} were used to calculate the inhibition efficiency using the following equation:

$$IE\% = [(I_{corr}^0 - I_{corr}^1)/I_{corr}^0] \times 100,$$

where I_{corr}^0 and I_{corr}^1 are the corrosion current density values in the absence and presence of inhibitor, respectively.

AC impedance spectra were recorded on the instrument used for polarization study, using the same three-electrode cell assembly. The real part (Z') and the imaginary part (Z'') of the cell impedance were measured in Ohms for various frequencies. The charge transfer resistance (R_t) and double layer capacitance (C_{dl}) values were calculated.

 $R_t = (R_s + R_t) - R_s$, where R_s = solution resistance;

 $C_{dl} = \frac{1}{2}\pi R_t f_{max}$, where $f_{max} = maximum$ frequency.

The R_t values estimated from the abovementioned equivalent circuit were used to calculate IE% according to the following equation:

$$IE\% = [(R_{t}^{1} - R_{t}^{0})/R_{t}^{1}] \times 100,$$

where R_t^0 and R_t^1 are the charge transfer resistances in the absence and presence of additive, respectively.

Carbon steel specimens immersed in a blank and in an inhibitor solution for a period of one day were taken out, rinsed with redistilled water, dried and observed in a scanning electron microscope to examine the surface morphology. The surface morphology measurements of the carbon steel were performed on a Hitachi S-3000 H computercontrolled scanning electron microscope.3.

3. RESULTS AND DISCUSSION

The ground water used in this study is given in Table 1. The corrosion rates of carbon steel, immersed in ground water in the absence and presence of catechol and Zn^{2+} , determined by the weight-loss method, are presented in Table 2. The inhibition efficiencies are presented in Table 3.

It is seen from Table 2 that when carbon steel was immersed in ground water, the corrosion rate was 139.29 mpy. Upon addition of various concentrations of catechol, the corrosion rate slowly decreased, i.e., the metal was protected against corrosion.

The influence of a divalent metal ion, Zn^{2+} , on the inhibition efficiency of catechol in controlling

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Table I. Water Quality Parameters							
Parameters	Value						
рН	7.30						
Total Dissolved Solids	1236 ppm						
Chloride	232 ppm						
Sulphate	75 ppm						
Total Hardness	416 ppm						
Conductivity	1792 µmhos/cm						

the corrosion of carbon steel, is given in Table 3. It is seen that the addition of 250 ppm of catechol alone gives a maximum IE of 52% and additions of various concentrations of Zn^{2+} ions to ground water offer a maximum IE of 15% at 100 ppm of Zn^{2+} . The combination of Zn^{2+} and catechol provides for better IE. For example, 100 ppm of Zn^{2+} gives an IE of 15%; 100 ppm of catechol gives an IE of 32%, while their simultaneous use offers a maximum inhibition efficiency of 90%. The improvement in

Table 2. Corrosion rates (CR) obtained from catechol- Zn^{2+} system, when carbon steel is immersed in ground water. Inhibitor: Catechol + Zn^{2}

Catechol (ppm)	Corrosion rate (CR) mpy*							
-	0	10	25	50	75	100		
0	132.29	132.32	128.14	126.75	122.57	118.39		
50	78	78	32	17	12	13		
100	59	78	81	22	58	8		
150	48	66	35	27	17	22		
200	43	43	48	32	23	25		
250	41	61	50	37	28	17		

*) mpy- mils per year

Table 3. Inhibition efficiencies (IE) obtained from catechol- Zn^{2+} system, when carbon steel is immersed in ground water. Inhibitor: Catechol + Zn^{2}

Catechol (ppm)	Corrosion rate (CR) mpy*					
-	0	10	25	50	75	100
0	0	5	8	9	12	15
50	10	10	64	81	87	85
100	32	10	6	75	83	90
150	45	23	25	69	81	75
200	50	50	44	64	73	71
250	52	29	42	58	67	80

Inhibitor: Catechol +Zn²

 Table 4
 Synergism parameters derived from inhibition efficiencies of Catechol–Zn²⁺ system.

Catechol						Zn^{2}					
(ppm)	0	10	S_I	25	S_I	50	S_I	75	S_{I}	100	S_I
	(ppm)	(ppm)		(ppm)		(ppm)		(ppm)		(ppm)	
0	0	0	5	-	8	-	9	-	12	-	15
50	50	10	10	0.95	64	2.30	29	1.15	81	4.16	85
100	100	32	23	0.72	6	0.66	67	1.87	33	0.89	90
150	150	45	50	0.68	25	0.67	76	2.08	81	2.54	75
200	200	50	29	0.95	44	0.82	72	1.62	73	1.62	71
250	250	52	27	0.64	42	0.76	64	1.21	67	1.28	80

the protection efficiency was attributed to the synergistic effect resulting from the combination of the two inhibitors, Zn^{2+} and catechol, which form a complex. As a result of the complex formation, the inhibitor molecules are readily transported from the bulk of the solution to the metal surface [21-23].

Synergism parameters (S_I) were calculated using the relation

$$S_1 = \frac{1 - \theta_{1+2}}{1 - \theta'_{1+2}}$$

where:

$$\theta_{1+2} = (\theta_1 + \theta_2) - (\theta_1 \theta_2)$$

 θ_1 = Surface coverage (θ) of inhibitor catechol

 θ_2 = Surface coverage (θ) of inhibitor Zn²⁺

 θ'_{1+2} = Combined surface coverage (θ) of inhibitor catechol and Zn²⁺.

Synergism parameters are indicative of the synergistic effect existing between two inhibitors [24-26]. The values of the synergism parameters (Table 4) are greater than unity, indicating a synergistic effect existing between Zn^{2+} and various concentrations of catechol.

To investigate whether the influence of Zn^{2+} on the inhibition efficiency of catechol is statistically significant, analysis of variance (F-test) was carried

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Source of	Sum of	Degrees of	Mean	F	Level of
variance	squares	freedom	square		significance
Between	300	1	300	1.09	P < 0.05
Within	2197.68	8	274.71		
Between	2.16	1	2.16	0.005	P < 0.05
Within	3386.15	8	423.27	64.79	
Between	3267	1	3267	1.74	P < 0.05
Within	1877.68	8	234.71		
Between	3137.64	1	3137.64	7.64	P < 0.05
Within	1812.83	8	410.71		
Between	5292	1	5292	20.72	P < 0.05
Within	2043.68	8	255.46		
	Source of variance Between Within Between Within Between Within Between Within Between Within	Source of varianceSum of squaresBetween300Within2197.68Between2.16Within3386.15Between3267Within1877.68Between3137.64Within1812.83Between5292Within2043.68	Source of varianceSum of squaresDegrees of freedomBetween3001Within2197.688Between2.161Within3386.158Between32671Within1877.688Between3137.641Within1812.838Between52921Within2043.688	Source of variance Sum of squares Degrees of freedom Mean square Between 300 1 300 Within 2197.68 8 274.71 Between 2.16 1 2.16 Within 3386.15 8 423.27 Between 3267 1 3267 Within 1877.68 8 234.71 Between 3137.64 1 3137.64 Within 1812.83 8 410.71 Between 5292 1 5292 Within 2043.68 8 255.46	Source of variance Sum of squares Degrees of freedom Mean square F Between 300 1 300 1.09 Within 2197.68 8 274.71 Between 2.16 1 2.16 0.005 Within 3386.15 8 423.27 64.79 Between 3267 1 3267 1.74 Within 1877.68 8 234.71 Between 3137.64 1 3137.64 7.64 Within 1812.83 8 410.71 1 Between 5292 1 5292 20.72 Within 2043.68 8 255.46 1

Table 5. Distribution of F – value between the inhibition efficiencies of various concentrations of catechol (0 ppm Zn^{2+}) and the inhibition efficiencies of catechol in presence of 10, 25, 50, 75, 100 ppm Zn^{2+} .

out [27-28]. The results are given in Table 5. As can be seen, the influence of 10, 25, 50, 75 and 100 ppm of Zn^{2+} on the inhibition efficiencies of 50, 100, 150, 200, 250 ppm of catechol is investigated. The obtained F-value of 1.09 for 10 ppm Zn^{2+} , 0.005 for 25 ppm Zn^{2+} and 1.74 for 50 ppm Zn^{2+} is not statistically significant, since it is less than the critical F-value of 5.32 for 1.8 degrees of freedom at a 0.05 level of significance. Therefore, it is concluded that the influence of 10 ppm Zn^{2+} , 25 ppm Zn^{2+} and 50 ppm Zn^{2+} on the inhibition efficiencies of various concentrations of catechol is not statistically significant.

The obtained F-value of 7.64 for 75 ppm of Zn^{2+} and 20.72 for 100 ppm Zn^{2+} is statistically significant, since it is greater than the critical Fvalue of 5.32 for 1.8 degrees of freedom at a 0.05 level of significance. Therefore, it is concluded that the influence of 75 ppm Zn^{2+} and 100 ppm Zn^{2+} inhibition efficiencies on the of various concentrations of catechol is statistically significant.

The potentiodynamic polarization curves of carbon steel immersed in ground water in the absence and presence of inhibitors are shown in Fig.1. The corrosion parameters such as corrosion potential (E_{corr}), corrosion current (I_{corr}), Tafel (LPR) are given in Table 6. When carbon steel was immersed in ground water, the corrosion potential slopes (b_a, b_c) and linear polarization resistance was -807 mV vs SCE. In presence of the inhibitors (100 ppm of catechol and 100 ppm of Zn^{2+}), the corrosion potential shifted to the anodic side (-690 mV vs SCE). The electron transfer became more difficult, since the metal surface has become nobler. This fact was supported by the observation that the LPR value increased from 4.95×10^4 Ohm cm² to 1.069×10^5 Ohm cm² and the corrosion current



Fig. 1. Polarization curves of carbon steel immersed in test solution. (a) Ground water (b) Ground water containing 100 ppm of catechol and Zn^{2+} .



Fig.2. AC impedance spectra of carbon steel immersed in test solution.(a) Ground water (b) Ground water containing 100 ppm of catechol and Zn^{2+}

decreased from 7.157×10^{-7} A/cm² to 3.965×10^{-7} A/cm². These results suggest that a protective film (probably of Fe²⁺-catechol complex) is formed on the metal surface, thus preventing the electron transfer process and corrosion [29].



Table 6. Corrosion parameters of carbon steel immersed in ground water in the absence and presence of inhibitors obtained by potentiodynamic polarization method. Inhibitor system: catechol + Zn^{2+} .

Fig. 3. Analysis of Bode plot. (a)The Bode plot of carbon steel immersed in Ground water. b) The Bode plot of carbon steel immersed in Ground water containing 100 ppm of catechol and Zn^{2+} .

The IE% calculated by the weight-loss method and by the polarization study slightly differed and this difference may be attributed to the change in surface state during polarization, which plays an important role in the adsorption process [30].

When carbon steel was immersed in ground water, the R_t value was found to be 162.23 Ohm cm². The C_{dl} value was 3.14×10^{-8} F/cm². Upon addition of 100 ppm of catechol and 100 ppm of Zn²⁺, the R_t value increased to 2869.0 Ohm cm². This pointed to the formation of a protective film on the metal surface, probably by deposition of Zn

$$Fe \rightarrow Fe^{2+} + 2e^{-}$$
$$Zn^{2+} + 2e^{-} \rightarrow Zn^{2+}$$

on the metal surface, which prevented further corrosion.

The prevention of corrosion was further supported by the decrease in the double layer capacitance value (from 3.14×10^{-8} F/cm² to 1.78×10^{-9} F/cm²) is given in Table 7. The polarization study rapidly measures the rate of electron transfer reactions, while AC impedance spectra shown in Fig.2 record the prolonged formation of a protective film on the metal surface. This behaviour means that the film obtained acts as a barrier to the corrosion process and clearly proves the film formation and adsorption of inhibitor on the metal surface [31].

System	$R_t \Omega cm^2$	C_{dl} F/0.00785 cm ²	Impedance $\log (z/\Omega)$	IE %
Ground water	162.23	3.14x10 ⁻⁸	2.287	
Ground water + 100 ppm of catechol + 100 ppm of Zn ^{$2+$}	2869	1.78x10 ⁻⁹	3.496	94.3

 Table 7. AC- Impedance parameter of carbon steel immersed in the presence of inhibitors obtained from AC impedance spectra.

The corresponding Bode plots are shown in Fig. 3a. It is observed that in the absence of inhibitors the real impedance value (log Z) is 2.28 Ohm. In the presence of inhibitors this value increases to 3.49 (see Fig. 3b). The increase in the impedance value suggests a high protective efficiency of this system. The plot obtained in the presence of inhibitor is characterized by a single time constant. This indicates the formation of a homogeneous film on the metal surface [32,33].



Fig.4b. The FTIR spectra of the film formed on the surface of carbon steel after immersed in ground water containing catechol (100 ppm) and $Zn^{2+}(100 \text{ ppm})$.

The FTIR spectrum of pure catechol (KBr) is shown in Fig.4a. The OH stretching frequency appeared at 3314 cm^{-1} . The C=C ring stretching frequency appeared at 1588.55 cm⁻¹. The FTIR spectrum of the film formed on the metal surface after immersion in the solution containing ground water, 100 ppm of catechol and 100 ppm of Zn^{2+} is shown in Fig 4b. It is seen that the OH stretching frequency has shifted from 3314 to 3432 cm⁻¹ and the C=C ring stretching frequency has shifted from 1588.55 to 1597 cm^{-1} [34]. These observations indicated that catechol coordination to Fe²⁺ through the oxygen atoms of the phenolic group and the π electrons of benzene ring resulted in the formation of a Fe²⁺-catechol complex at the anodic sites of the metal surface. The peak at 1354 cm⁻¹ was due to Zn(OH)₂ formed on the cathodic sites of the metal surface [35].

The SEM images (magnification $2000\times$) of carbon steel specimens immersed in ground water for 1 day in the absence and presence of inhibitor system are shown in Fig. 5(a-c) respectively.

The SEM micrograph of polished carbon steel surface (control) in Fig. 5a shows the smooth surface of the metal with no corrosion products on the metal surface. On the SEM micrograph of carbon steel surface immersed in ground water (Fig. 5b) rough metal surface is seen which indicates the corrosion of carbon steel in ground water. Fig. 5c reveals that in the presence of 100 ppm of catechol and 100 ppm of Zn^{2+} in the water, the surface coverage increases, which in turn results in the formation of an insoluble complex on the surface of the metal (catechol- Zn^{2+} inhibitor complex) and the surface is covered by a thin layer of inhibitors which effectively controls the dissolution of carbon steel [36].

Mechanism of corrosion inhibition

The results of the weight-loss study revealed that the formulation consisting of 100 ppm of catechol and 100 ppm of Zn^{2+} offers an inhibition

efficiency of 90%. Results of the polarization study showed that this formulation acts as an anodic inhibitor. The FTIR spectra showed that Fe^{2+} -catechol complex and $Zn(OH)_2$ are present on the inhibited metal surface.



Fig. 5. SEM micrographs of : (a)Carbon steel (control); Magnification-X 2000; (b) Carbon steel immersed in well water; Magnification-X 2000; (c) Carbon steel immersed in well water containing catechol (100 ppm) + $Zn^{2+}(100 \text{ ppm})$; Magnification–X 2000.

In order to explain all observations in a holistic way, a suitable mechanism of corrosion inhibition was proposed as follows.

When a carbon steel specimen is immersed in the neutral aqueous environment, the anodic reaction is:

$$Fe \rightarrow Fe^{2+} + 2e^{-}$$

and the cathodic reaction is

$$2H_2O + O_2 + 4e^- \rightarrow 4OH^-$$

• When the formulation consisting of 100 ppm of catechol and 100 ppm of Zn^{2+} was prepared, the Zn^{2+} - catechol complex was formed in solution.

• When carbon steel was immersed in this solution, there was diffusion of Zn^{2+} -catechol complex towards the metal surface.

• On the metal surface, it was converted to Fe^{2+} -catechol complex. Zn^{2+} was released and Zn (OH)₂ was formed:

• Thus the protective film consists of Fe^{2+} catechol complex formed on anodic sites of the metal surface and is more stable than the Zn^{2+} catechol complex [37-38] and the $Zn(OH)_2$ precipitate in the local cathodic regions.

4. CONCLUSIONS

The inhibition efficiency of the catechol - Zn^{2+} system in controlling the corrosion of carbon steel in ground water was evaluated by the weight-loss method. The present study leads to the following conclusions:

• Weight-loss study revealed that the formulation consisting of 100 ppm of catechol and 100 ppm of Zn^{2+} had 90% inhibition efficiency in controlling corrosion of carbon steel immersed in ground water.

• Synergism parameters suggested that a synergistic effect exists between catechol and Zn²⁺.

• ANOVA test revealed that the synergistic effect of the above formulation is statistically significant.

• Polarization study revealed that this system acts as anodic inhibitor.

• AC impedance spectra (Bode plots) revealed that a protective layer is formed on the metal surface.

FTIR spectra revealed that the protective film consisted of Fe^{2+} -catechol complex and Zn (OH)₂.

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ИНХБИРАЩО ДЕЙСТВИЕ НА СИСТЕМА КАТЕХОЛ-ЦИНК ПРИ КОНТРОЛИРАНЕ НА КОРОЗИЯТА НА ВЪГЛЕРОДНА СТОМАНА

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Получена на 17 декември 2010; Приета на 4 януари 2011

(Резюме)

Инхибиращата ефективност на инхибиторни системи, състоящи се от различни комбинации на катехол и йони на цинк, при конролиране на корозията на въглеродна стомана, потопена в подпочвена вода, е оценена по метода на загубата на тегло. 100 ppm от Zn^{2+} имат само 15% инхибиращата ефективност (ИЕ). При добавяне на 100 ppm катехол, ИЕ се увеличава до 90%. Използвани са метод на загубата на тегло, поляризационни изследвания и импедансни спектри. Природата на предпазния филм, образуван върху повърхността на метала, се анализира чрез инфрачервена спектроскопия с Фурие трансформация (FTIR). Беше установено, че предпазния филм се състои от комплекс Fe^{2+} - катехол. За оценка на синергичния ефект на инхибиторите са използвани параметри на синергизъм и дисперсионен анализ.