T. Umamathi et al. / Portugaliae Electrochimica Acta 44 (2026) 337-350

Mild Steel Corrosion Inhibition in Ground Water by an Aqueous Extract of *Ocimum Basilicum* Leaves

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Received 06/05/2024; accepted 20/10/2024
https://doi.org/10.4152/pea.2026440502

Abstract

CI of MS in ground water by AEOBL and Zn was herein investigated by WL method and PDP. It was concluded that MS corrosion resistance strengthened with AEOBL and Zn presence, due to an increase in LPR and a decrease in I_{corr}. Therefore, AEOBL and Zn inhibited metal corrosion and acted as good CI. SP showed a SE between AEOBL active principles and Zn ion. FTIR spectral analysis revealed that a protective film was formed on the MS surface by coordination of Fe^{2+} ion with polar O atoms of phenolic and ether groups, pi electrons of the double bonds and O of methoxy group. Each functional group contributed to the protective film formation, which also contained $Zn(OH)_2$. SEM images confirmed the formation of a protective film on the MS surface. PDP revealed that LPR increased from 760 to 4561 Ohm/cm^2 and I_{corr} decreased from 5.941×10^{-5} to 1.006×10^{-5} A/cm^2. The findings may be useful in water towers where ground water is used for cooling.

Keywords: AEOBL; CI; FTIR; ground water; MS; PDP; SEM; SP; WL method; $Zn(OH)_2$.

Introduction[•]

Corrosion is a natural process in which a refined metal is converted to a more chemically stable form, such as an oxide, hydroxide or sulphide. It is the gradual decomposition of materials (usually metals) due to chemical and/or electrochemical reactions with their surroundings. There are several methods used to control corrosion, such as the use of CI, which is a substance that, by its chemical activity, reduces CR of a metal surface exposed to an aggressive medium, thus extending the service life of metallic components. In the corrosion process, usually there is loss of electrons in contact with a medium such as an electrolyte. The CI supply electrons

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[•] The abbreviations list is in pages 347-348.

onto the metal surface, thereby decreasing the release rate of electrons from it. In this respect, CI can be considered as electron pumpers. The recent trend is the use of aqueous extract of natural plant materials as CI [1-10], which usually contain many active principles that are organic molecules with hetero atoms such as O, S and N. The electrons on the hetero atoms from active molecules in plants extracts are donated onto metal ions, hindering the formation of complexes from these ions. Highly efficient green CI for MS in sulfuric acid has been reported by [1]. Petals of *B. glabra* have been investigated as green CI for MS, at different Ct and temperatures, by PPD, EIS and gravimetric methods. SEM and metallurgical microscopy have been used for surface studies. A relation between IE(%) obtained from theoretical and experimental techniques has been established.

Inhibition properties of *Vang* tea-water extract for CS corrosion in acidic environments have been investigated by [2]. Electrochemical properties have been characterized by OCP, EIS, LPR and PDP. Post-immersion surface analyses have also been evaluated using SEM, ATR-FTIR and XPS.

The use of methanol extract of RR (*Işgın*) flower as a natural and promising CI for MS protection in a 1 M HCl solution has been investigated by [3]. In this study, the adsorption and protection ability of RR extract on MS corrosion in 1 M HCl solution have been investigated with the help of several electrochemical techniques. RR molecules interacted and bonded to the MS surface via physical and chemical interactions. RR could be suggested as a promising natural CI for MS protection in a 1 M HCl solution.

Electrochemical and DFT theoretical evaluation of *Randia monantha Benth* pulp and seed extract as eco-friendly CI for MS in a 1 M HCl solution have been reported by [4]. Their work has assessed IE(%) of *Randia m.* on A36 MS corrosion in a 1 M HCl solution through WL and electrochemical measurements such as PPD and EIS. Surface analysis was carried out using SEM and EDS. The corroded steel surface was examined by SEM and EDS, confirming the CI compounds adsorption. Chemical quantum calculations have allowed to determine the extract's electronic properties, explaining the CI/metal surface interactions and adsorptions modes.

Eco-friendly plant extracts have been tested by [5] as green CI for metals and alloys in corrosive mediums, with a focus on literature from past five years. Characterization techniques, including gravimetric analysis, EIS, kinetic and adsorption parameters, computational analysis and surface morphologies techniques, have been discussed to provide insights into the inhibition mechanism and morphological configuration.

MPE performance as a natural product for the CI of LCS in NaCl/Na_2S solutions has been investigated by [6], using chemical and electrochemical techniques to evaluate its IE(%). With higher Ct of MPE, IE(%) increased, reaching 91.2% at 300 ppm, whereas increased temperature favored its slow decrease.

Grafted biopolymers have been used by [7] as CI. Biopolymer-based products have fascinated and created interest in developing alternative technologies for removal of toxic metal ions and dyes from wastewater, due to their comparability, eco-friendliness, economical value, non-toxicity, high thermal stability, ease of operation and excellent sorption capacities. Grafted biopolymers tend to improve water solubility, stability, flexibility, moisture resistance, compatibility and biodegradability.

Phytochemical components of *Allium Jesdianum* flower have been used by [8] as effective corrosion-resistant materials for Fe(1 1 0), Al(1 1 1) and Cu(1 1 1). A comprehensive investigation of three phytochemicals present in this flower, such as Palmitic acid, a-Linolenic acid and Icosan-1-ol, has been made using computational methods (DMol3 and Monti Carlo simulations), to determine IE(%) for corrosion, chemical reactivity and adsorption behavior.

RR leaf extract has been examined by [9] on its adsorption, CI, stability and synergistic inhibition SE on MS in a 1 M HCl solution. Inhibition SE of iodide ions and stability of CI film surface have also been studied. The protection ability of extract was investigated using electrochemical techniques. The stability of surface film has been evaluated by PDP and potentiostatic studies. After being exposed to the corrosive medium, the metal surface has been examined using SEM, EDX, AFM and contact angle measurements.

Green CI for steel and other metals in basic media have been investigated by [10]. The study has aimed to briefly investigate the effectiveness of plant extracts used as CI for steel and other metals in basic media. CI have been recommended by various researchers as one of the ways to slow down CR and to reduce financial losses associated with it.

Herein, AEOBL was used as CI of MS immersed in ground water, so that the findings may be used in structures making use of ground water as cooling systems. Corrosion IE(%) was evaluated by WL method. Mechanistic aspects of the CI were investigated by PDP technique. The protective film was analyzed by FTIR and SEM. AEOBL's anodic/cathodic nature was investigated. Surface morphology characteristics were revealed by SEM and FTIR techniques.

Experimental

AEOBL

Ocimum basilicum, annual herb of the mint family (*Lamiaceae*) (Fig. 1), is grown for its aromatic leaves. OB is likely native to India and widely grown as kitchen herb. Its leaves are used fresh or dried to flavor meats, fish, salads and sauces. OB tea is a stimulant [11].

In this study, 50 g OB leaves (Fig. 1) were shade dried and boiled with distilled water. The suspended materials were filtered and made up to 100 Ml. From then on, AEOBL was used as CI.

Figure 1: Photo of OB leaves.

WL method

MS specimens in triplicate were immersed in 100 mL ground water with various Ct of AEOBL, for one day. The specimens' weight before and after immersion was determined using a Shimadzu balance, model AY62. Corrosion products were cleaned with Clarke's solution. CR was calculated using the following equation.

$$\mathrm{CR} = W/At \qquad (1)$$

where W is WL (mg), A is the specimen surface area (dm^2) and = IT (days).
CR was expressed in mdd [mg/(dm^2)(day)] units.
IE(%) was calculated using the relation:

$$\mathrm{IE}(\%) = [(CR_1\text{-}CR_2)/CR_1] \times 100\ \% \qquad (2)$$

where CR_1 and CR_2 are CR without and with AEOBL, respectively.

Polarization study

A CHI electrochemical work station with impedance model 660A was employed for this purpose. A three-electrode cell assembly was herein used (Fig. 2).

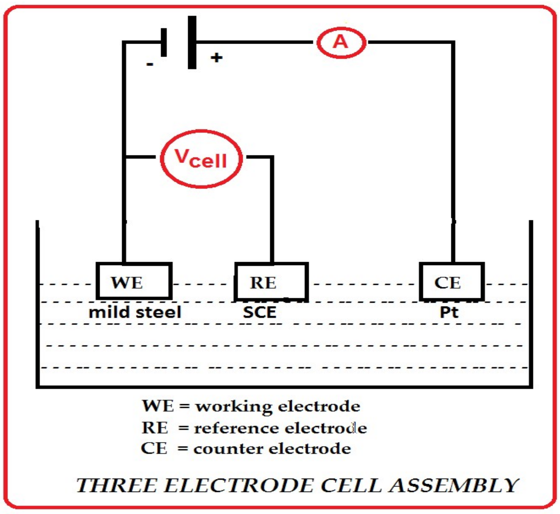


Figure 2: Circuit diagram of three-electrode cell assembly.

MS, SCE and Pt were used as working, reference and counter electrodes, respectively. From PDP corrosion parameters such as E_{corr}, I_{corr}, β_a, β_c and LPR were calculated. Scan rate (V/S) was 0.01. Hold time (Efcs) was zero and quit time (s) was two.

Surface characterization study

MS specimens were immersed in various test solutions for one day. Then, they were taken out and dried. The nature of the protective film formed on the MS surface was analyzed by surface characterization studies such as SEM and FTIR.

SEM

MS specimens immersed in various test solutions for one day were taken out, rinsed with double distilled water, dried and subjected to surface examination. MS surface morphology measurements were carried out by SEM using CAREL ZEISS make model EVO-18.

FTIR spectra

The protective film formed on the metal surface was carefully removed, mixed thoroughly with KBr and made in to pellets. Then, FTIR spectra were recorded in a Perkin - Elmer "Spectrum Two" spectrophotometer.

Results and discussion

WL method

IE(%) of AEOBL in controlling corrosion of MS immersed in ground water was evaluated by WL method. Results are given in Tables 1 and 2.

Table 1: CR (mdd) of MS immersed in ground water and IE(%) of AEOBL.

AEOBL (mL)	CR (mdd)	IE(%)	SC	Ct/ SC
0	16.58	-	-	-
2	6.30	62	0.62	3.23
4	5.47	67	0.67	5.97
6	4.80	71	0.71	8.45
8	3.81	77	0.77	10.39
10	2.98	82	0.82	12.19

Table 2: CR (mdd) of MS immersed in ground water and IE(%) of AEOBL + 50 ppm Zn^2. IT of 1 day.

AEOBL (mL)	Zn^{2+} (ppm)	CR (mdd)	IE(%)	SC	Ct/ SC
0	0	16.58	-	-	-
0	50	14.42	13	0.13	-
2	50	3.64	78	0.78	2.56
4	50	2.82	83	0.83	4.82
6	50	2.32	86	0.86	6.98
8	50	1.49	91	0.91	8.79
10	50	0.83	95	0.95	10.53

The effect of Zn^{2+} on IE(%) of AEOBL was also investigated. It was seen that IE(%) of CI increased with Zn^{2+}, which indicates a SE between both. That is, the CI mix showed better IE(%) than its individual elements. For example, IE(%) of 10 mL AEOBL was 82%, while that of 50 ppm Zn^{2+} was 13%. However, when combined, it was 95%.

SP

SP was used to determine SE existing between AEOBL and Zn^{2+}. SP can be calculated, using the following relationship.

$$S_I = 1 - \theta_{1+2} / 1 - \theta'_{1+2} \quad (3)$$

$$\theta = IE\%/100,\ \theta_{1+2} = (\theta_1 + \theta_2) - (\theta_1 \times \theta_2) \quad (4)$$

where θ_1, θ'_{1+2} and θ'_{1+2} are SC of OB, Zn^{2+} and combined ones, respectively.
SP indicate SE between CI. SP value was found to be greater than one, suggesting SE between AEOBL and Zn^{2+}. SP value is given in Table 3. When SP > 1, SE exists. From Table 3, it can be seen that SP value was greater than one. Thus, increased IE(%) caused by Zn^{2+} addition to OB was due to SE.

Explanation for SE between CI system and Zn^{2+}

When Zn^{2+} was added to AEOBL, Zn-CI complex was formed. In the solution bulk, Zn-CI bond was strong enough to carry it towards the MS surface, and weak enough to break on it to form the complex, Zn^{2+} being released. Moreover, in general, Fe-AEOBL complex was more stable than Zn-CI one. Anodic reaction was controlled by formation of Fe-CI complex on the anodic sites of the MS surface. The released Zn^{2+} has combined with OH^- on the MS surface (because this is a ground water system) to form $Zn(OH)_2$ on its cathodic sites. Thus, both anodic and cathodic reactions were controlled, accounting for SE between AEOBL and Zn^{2+} (Table 3).

Table 3: SP for MS immersed in ground water without and with AEOBL-Zn^{2+} (50 ppm).

AEOBL (mL)	CR (mdd)	IE(%)	SC	Ct/SC of Zn^{2+} (50 ppm)
2	0.62	0.13	0.78	1.50
4	0.67	0.13	0.83	1.68
6	0.71	0.13	0.86	1.80
8	0.77	0.13	0.91	2.22
10	0.82	0.13	0.95	3.13

Electrochemical study

Electrochemical study such as PDP was employed to evaluate corrosion protection of MS in ground water without and with OB-Zn^{2+} as CI.

Analysis of PDP curves

PDP study was used to detect the formation of a protective film on the MS surface. When a protective film was formed on the MS surface, LPR increased and I_{corr}

decreased [12-16]. PDP curves of MS immersed in various test solutions are shown in Figs. 3 and 4.

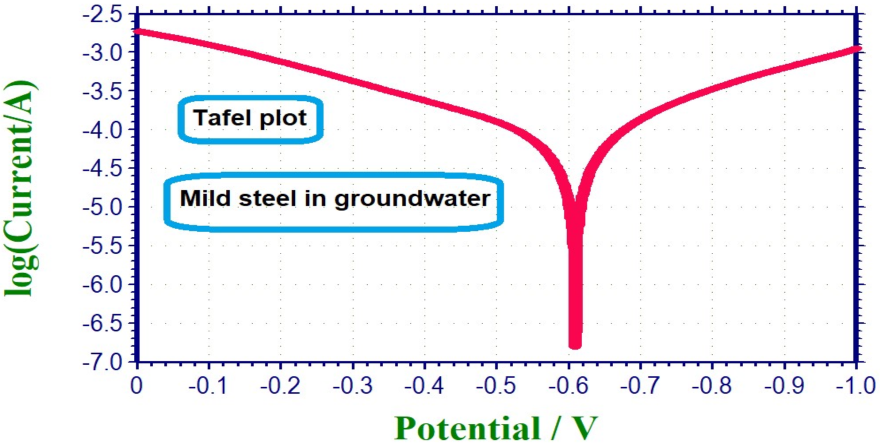


Figure 3: PDP curve of MS immersed in groundwater.

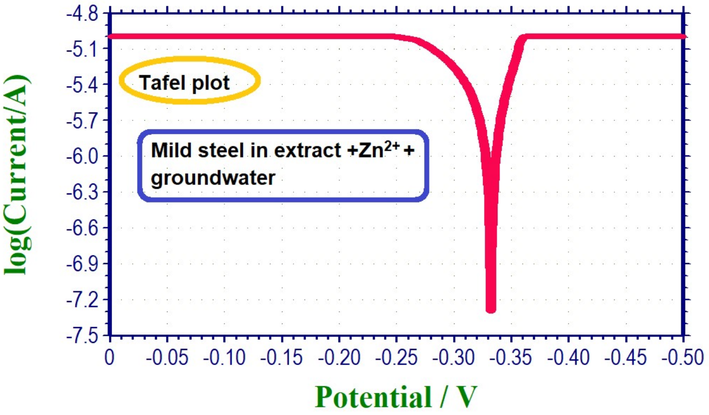


Figure 4: PDP curve of MS immersed in groundwater with CI - Zn^{2+} system.

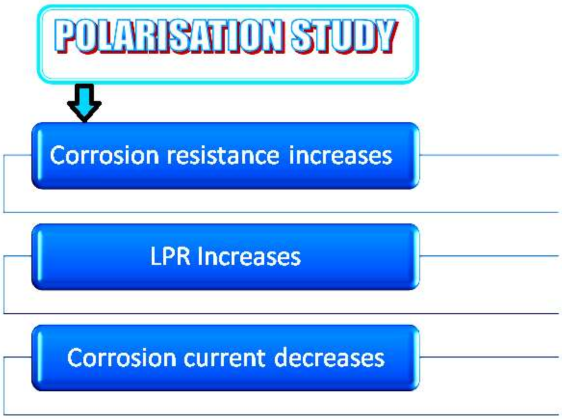
Corrosion parameters, namely, E_{corr}, β_c, β_a, LPR and I_{corr} are given in Table 4.

Table 4: Corrosion parameters of MS, immersed in various test solutions, obtained by PDP.

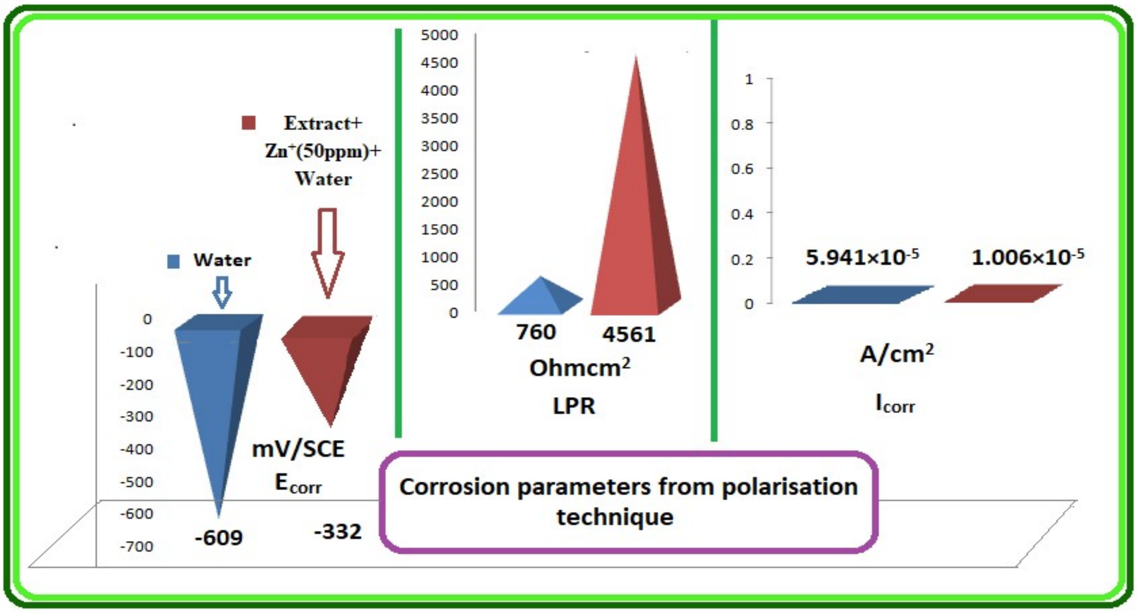
System	E_{corr} mV vs. SCE	β_c mV/decade	β_a mV/decade	LPR Ohm/cm^2	I_{corr} A/cm^2
GW	-609	187	232	760	5.941 x 10^{-5}
GW + AEOBL + Zn^{2+} (50 ppm)	-332	234	192	4561	1.006 x 10^{-5}

When MS was immersed in ground water, LPR value was 760 Ohm/cm^2 (Table 1). E_{corr} was -609 mV *vs.* SCE. I_{corr} value was 5.941 × 10^{-5}A/cm^2.

When MS was immersed in ground water with 10 mL AEOBL + Zn^{2+} (50 ppm), LPR value increased and I_{corr} decreased. This indicates that MS corrosion resistance increased, due to the formation of a protective film on its surface. E_{corr} moved from -609 to -332 mV *vs.* SCE, indicating an anodic shift. Hence, it was concluded that CI system mainly controlled anodic reaction. Various corrosion parameters are compared in Schemes A and B.



Scheme A: Correlation among corrosion parameters from PDP.



Scheme B: Comparison of corrosion parameters from PDP.

Analysis of FTIR spectra

The structures of various chemical components present in AEOBL [17] are shown in Fig. 5.

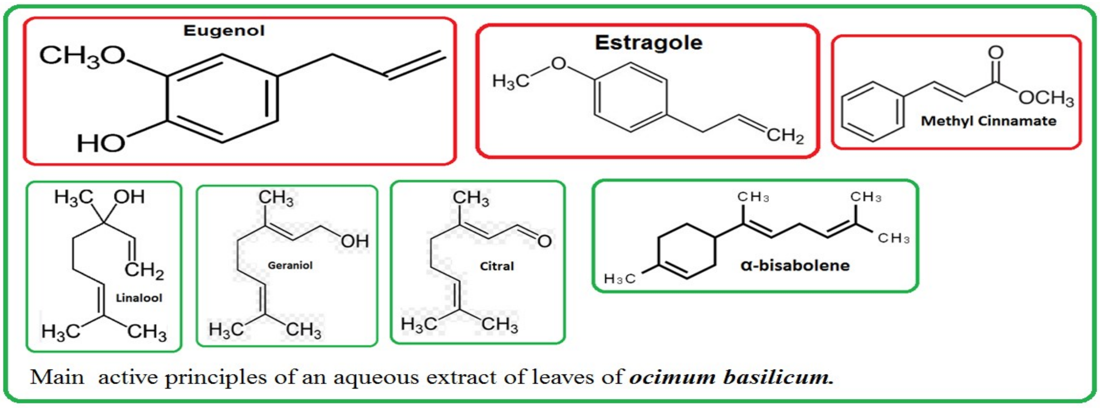


Figure 5: Main active principles of AEOBL.

The various functional groups and their stretching frequencies are given in Table 5.

Table 5: Various functional groups and their stretching frequencies.

Stretching/bending	**Range (cm^{-1})**	**Glass plate solid mass (cm^{-1})**	**Metal plate film (cm^{-1})**
Aromatic compound C-H bending	2000-1650	1637	1638
Alkyl aryl ether C-O stretching	1275-1200	1236	disappeared
Ketone C=O	1650	1637	1638
Phenolic -OH bending	1390-1310	1384	1384
Aromatic–CH bending	2000-1650	1637	1638
Aliphatic –CH	2800-2900	2923, 2853	2922, 2854
-OH	3550-3200	3422	3408
$Zn(OH)_2$			1383
Metal-O			674

A few drops of AEOBL were dried on a glass plate, for obtaining a desiccated mass, which was then mixed with KBr and made into a pellet. Its FTIR spectrum is shown in Fig. 6.

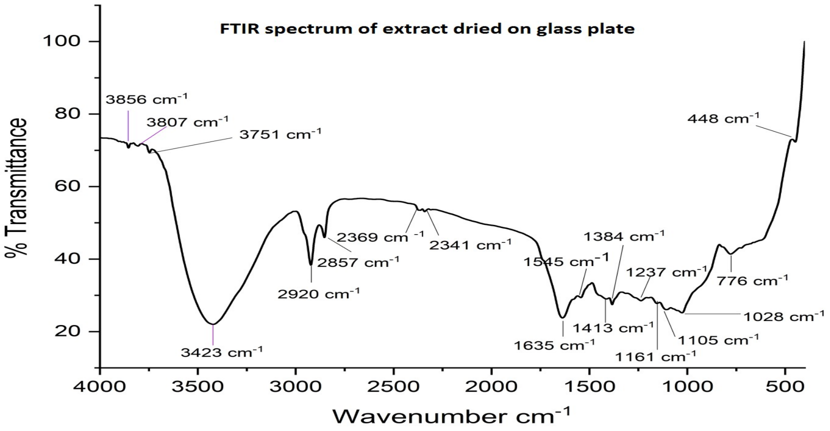


Figure 6: FTIR spectrum of AEOBL dried on glass plate.

FTIR spectrum (KBr) of the film formed on the MS surface after immersion in the solution containing ground water, AEOBL and Zn^{2+} is shown in Fig. 7. Table 5 reveals that the protective film was formed on the Ms surface by Fe^{2+} coordination with polar O atoms of phenolic group, ether group, pi electrons of double bonds and O of methoxy group. Each functional group contributed for the protective film formation. The peak at 1383 cm^{-1} was due to $Zn(OH)_2$. The peak at 674 cm^{-1} was caused by MS-O bond. This may be due to $Zn(OH)_2$ and FeO.

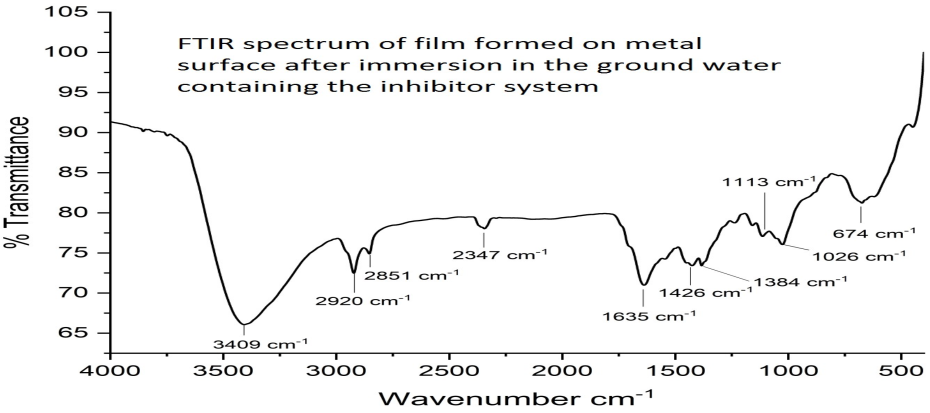


Figure 7: FTIR spectrum of the film formed on the MS surface after immersion in ground water with the CI system.

Analysis of SEM images

SEM images are used to analyze the surface morphology of the protective films in CI studies [18-20].

SEM images of MS - polished, in ground water and in CI system - are shown in Fig. 8. For polished MS, the surface was smooth. When it was in ground water, pits appeared due to corrosive ions like chloride. In the CI system, the pits disappeared due to the formation of a protective film on the MS surface.

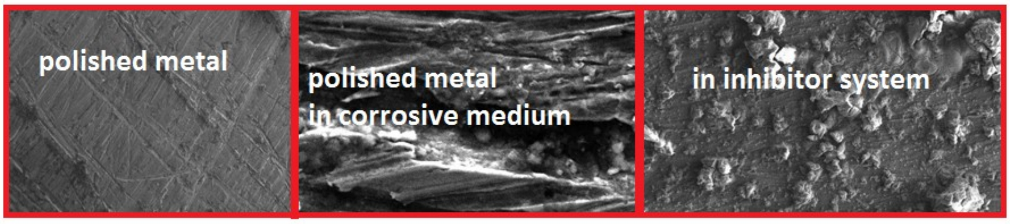


Figure 8: SEM images of various metal surfaces.

Conclusion

CI of MS in AEOBL and Zn^{2+} was investigated by WL method and PDP. It was inferred that corrosion resistance of MS increased with AEOBL and Zn^{2+}. This was revealed by an increase in LPR and a decrease in I_{corr}. Therefore, AEOBL and Zn^{2+} were able to inhibit MS corrosion and act as a good CI. SP revealed that SE existed between active principles of AEOBL and Zn^{2+}. FTIR spectral analysis revealed that the protective film was formed on the MS surface by coordination of Fe^{2+} ion with polar O atoms of phenolic group, ether group, pi electrons of double bonds and O of methoxy group. Each functional group contributed to the protective film formation. The protective film also contained $Zn(OH)_2$. SEM images confirmed the formation of a protective film on the metal surface. The findings may be useful in water towers where ground water is used for cooling.

Scope for further study

In future, the following study could be undertaken: Al, Cu and Zn can be used instead of MS, , simulated oil well water and sea water etc., can replace groundwater; surface analysis, such as AFM, EDAX, DLS, etc., can be employed.

Abbreviations

AC: alternating current
AEOBL: aqueous extract of *Ocimum basilicum* leaves
AFM: atomic force microcospy
ATR: attenuated total reflection
β_a: anodic Tafel slope
β_c: cathodic Tafel slope
C_{dl}: double layer capacitance
CI: corrosion inhibitor/inhibition
CR: corrosion rate
Ct: concentration
E_{corr}: corrosion potential
EDS: energy dispersive spectroscopy
EIS: electrochemical impedance spectra
FTIR: Fourier transform infra-red
HCl: hydrochloric acid
I_{corr}: corrosion current
IE(%): inhibition efficiency
IT: immersion time
LCS: low carbon steel
LPR: linear polarization resistance
MPE: *Mentha piperita* extract
MS: mild steel
Na_2S: sodium sulfide
NaCl: sodium chloride
OCP: open circuit potential

PDP: potentiodynamic polarization
Ppm: parts per million
R_{ct}: charge transfer resistance
SC: surface coverage
SCE: saturated calomel electrode
SE: synergistic effect
SEM: scanning electron microscopy
SP: Synergism parameters
WL: weight loss
XPS: X-ray photoelectron spectroscopy
Zn: zinc
$Zn(OH)_2$: zinc hydroxide

Authors' contributions

S. Rajendran: writing, correspondence. **B. Gomathi, A. L. Jewelcy, P. S. L. Kala**: recorded electrochemical studies, FTIR spectra, SEM. **T. Umamathi, N. Anitha, I. S. Vinnarasi**: conceptualization and validation; review and editing. All authors have read and agreed to the final version of the manuscript.

Acknowledgement

The authors are thankful to their managements for their help and support.

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