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Studies on the effect of Zn ions on the corrosion behavior of iron-base alloys relevant to desalination and power plant conditions

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ABSTRACT

The corrosion behavior of mild steel and AISI 304L SS in the presence of dissolved Zn ions under different experimental conditions have been investigated. Experimental conditions include: variation in aqueous medium, Zn ions concentration, pH, dissolved oxygen, and flow condition. The variation in corrosion rate in the presence of varying concentrations of Zn ions was determined using weight loss technique and spectrophotometric analysis of iron ions entered into the test solutions after completion of immersion. The pH of the test solutions was also monitored during the entire period of immersion. The Zn ions present in the test solutions after completion of immersion were estimated using atomic absorption spectrophotometer (AAS). Open circuit potential (OCP) measurements and potentiodynamic polarization (PDP) measurements were carried out to find out the role of Zn ions on the corrosion behavior of mild steel and SS 304L. The surface morphology of steel before and after immersion in aqueous solution containing Zn ions was also examined using scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS). The influence of Zn ions on the corrosion behavior of mild steel is quite pronounced and show interesting trends whereas SS 304L is unaffected both in distilled water or artificial seawater.

Keywords: Mild steel; SS 304L; Corrosion; Zn ions; Immersion tests; Electrochemical tests

1. Introduction

In multistage flash (MSF) desalination plant, heat exchangers and flash chambers are the two important sections which are most prone to corrosion. For flash chambers, carbon steel is the most common material of construction; it is used as such or cladded with stainless steel. In the distillers, the recirculating brine generally contains high concentration of heavy metal ions like Cu, Ni, and Zn [1,2]. The source of Cu and Ni is the cupronickel condenser tubes in the distiller which are subjected to vapor phase corrosion leading to the contamination of brine and condensate with Cu and Ni. The source of Zn is generally the dosing chemicals. An investigation of internal corrosion problems in water distribution systems confirmed that the contamination of brine and condensate with heavy metals may lead to the deposition of these metals on the flash chamber components and may accelerate corrosion [3]. Further, in dual purpose power/water co-generation plants, the condensate from the desalination plants is returned to the boiler and the carryover metal ions are deposited over the water touched parts of the boiler leading to

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their corrosion [4,5]. Under normal operating conditions of boiler, a dark-colored magnetite film is primarily formed on the internal surface of boiler tubes. The magnetite films act as a protective barrier and provide protection to the underlying metals and prevent serious metal loss. The condenser and feed water heater alloys may also get corroded as a result of air leakage and consequently dissolved Cu and Ni are also transported and deposited in the boiler. Zn from dosing chemicals is essentially present in the deposits in significant concentration. The analysis of steam side scales of water wall tubes from a number of power plants operating in Saudi Arabia has revealed the presence of high concentrations of carryover Cu, Ni, and Zn in the deposits [5–8]. The deposits are easily removed showing a corroded metal surface. The role of carryover metals present in the deposit on the corrosion behavior of underneath metal has yet not been systematically investigated.

A seawater desalination plant offers numerous corrosion problems due to its process conditions, including factors such as temperature and pH, and operation in relatively aggressive environments consisting of seawater, seawater-air and salt-air aerosols, corrosive gases, slow moving or stagnant liquids, or deposit forming liquids [9]. Plain carbon steels, low- and high-alloy steels, martensitic, austenitic, and ferritic stainless steels are some of the materials that are generally used in desalination and power plants for fabrication of various components. During their service life, these materials pick up metal ions from the surrounding environment, which migrates in to the matrix and causes damage [10]. A survey of literature shows that limited references are available dealing with the effect of Cu and Ni on the corrosion behavior of carbon steel and stainless steel under desalination and power plant conditions [11]. But the role of Zn on the corrosion behavior of carbon steel and stainless steel under such condition is perhaps yet to be investigated.

The present work is concerned with the studies on the effect of zinc ions on corrosion behavior of mild steel and SS 304L under different experimental conditions relevant to desalination and power plants. The experimental conditions which have been taken into account are the nature of aqueous medium, metal ion concentration, pH, oxygen, and flow condition. The results of the study shall provide important information about the role of Zn ions carryover on the failure of components used in desalination and power plant.

2. Material and methods

2.1. Preparation of specimens

Commercial grade mild steel and AISI 304L SS were used for the immersion and electrochemical tests. The composition of steels as analyzed by optical emission spectrophotometer is given in the Table 1. For immersion tests coupons of dimension (4.0 × 3.2 × 0.13) cm were cut from the sheet. To hold the specimens, a hole of 1.5 mm diameter was made near the edge. The specimens were then machined and abraded on 180 grit SiC paper to simulate near service conditions. The abraded specimens were washed and degreased with ethyl alcohol and dried up and then used for immersion tests. Before commencing the immersion, the specimens were subjected to above treatments and freshly used with no further storage. For electrochemical tests, circular samples with 1.5 cm diameter were cut from the mild steel and SS 304L sheets. A conducting wire was spot welded to one face of the specimens for the electrical connection and the specimens were then mounted in a cold setting resin to provide a crevice free mount. The exposed circular face was ground on 180 grit SiC paper. The ground specimens were washed, degreased, and dried up.

2.2. Preparation of test solutions

The test solutions were made up with distilled water and artificial seawater. The artificial seawater was prepared in the laboratory following ASTM designation D1141. The composition of artificial seawater is given in Table 2 [12]. The solutions containing 10 ppb, 100 ppb, 1 ppm, 10 ppm, 20 ppm, 30 ppm, 40 ppm, and 100 ppm of Zn were prepared in distilled water and artificial seawater using analar grade CDH chemicals. The pH of the solutions was adjusted using appropriate chemicals. For test solution made up with distilled water a pH of 4.0, 6.5, and 8.5 was maintained whereas for the test solution made up with artificial seawater a pH of 8.2 was maintained.

2.3. Immersion test procedures

Immersion tests were performed in accordance with ASTM designation G31-72 (Re-approved 1990). After taking the initial weight and dimensions, the coupons were hanged in the test solution containing varying concentration of Zn ions. In order to avoid galvanic and crevice corrosion, the coupons were loosely tied with the help of nylon thread and immersed in the solution. A blank experiment was also carried out for the comparison purpose. The immersion tests were carried out under both static and dynamic conditions. Under static condition, the immersion tests were carried out under both aerated

Table 1
Composition of steel as analyzed by optical emission spectrometer

Steel	% Composition of elements									
	C	Si	Mn	P	S	Cr	Ni	Mo	Cu	Fe
Mild Steel	0.206	0.004	0.166	0.028	0.044	0.078	0.088	0.114	0.052	Bal
AISI 304L	0.048	0.440	1.587	0.028	0.007	18.002	7.635	0.353	0.326	Bal

Table 2
Chemical compositions of artificial seawater [12]

Compound	Concentration (g/l)
NaCl	24.53
$MgCl_2$	5.20
Na_2SO_4	4.09
$CaCl_2$	1.16
KCl	0.695
$NaHCO_3$	0.201
KBr	0.101
H_3BO_3	0.027
$SrCl_2$	0.025
NaF	0.003

and deaerated conditions. Under aerated condition the tests runs were of 24, 360, and 720 h duration for mild steel and 720, 4320, and 8760 h durations for SS 304L. However, under deaerated condition the tests runs were of 12 h duration. Under dynamic (aerated) conditions the runs were of 24 h duration. After completion of the immersion test, the specimens were taken out and cleaned following ASTM designation G1-90 and observed for any localized attack. The average corrosion rate was determined using the following relationship:

$$\text{Corrosion rate (mpy)} = \frac{534W}{\rho AT}$$

where W = mass loss in mg; A = area in inch^2; T = time of exposure in h; and ρ = density in g/cm^3.

2.4. Solution analysis of metal ion

Corrosion rate of mild steel and SS 304L was also measured from the determination of total iron ions (Fe^{2+}/Fe^{3+}) entered into the test solution during the course of corrosion [13,14]. The analysis was performed spectrophotometrically using a calibration curve prepared from standard solutions. The 1, 10-orthophenanthroline was used to produce a strongly colored complex with Fe(II) ions. Hydroxyl amine (HCL salt) was added as a reducing agent before color is developed to provide a measure of total Fe ions present into the solution. The pH of the solution was adjusted to a value between 6 and 9 by adding sodium acetate buffer. The samples were immersed in triplicate and average corrosion rate was calculated. The uncertainty for three replicate measurements was less than 5%. A double beam spectrophotometer (Model: Elico-SL-169 UV–visible spectrophotometer) was used for analysis of iron ions in the test solutions. The corrosion rate was calculated using the following relationship:

$$\text{Corrosion rate (mpy)} = \frac{14.4m}{d \times s \times t}$$

where 'm' is the mass of corroded metal (calculated from the total iron content determined in the test solution); 's' is the area of the test metal in m^2; 't' is the exposure time in h; and 'd' is the density in g/cm^3.

2.5. Monitoring of pH

The pH of the different test solutions containing the immersed mild steel and SS 304L samples was measured on daily basis and continued for 1 month.

2.6. Estimation of metal ions in the test solution

The amount of Zn ions present in the test solutions after the completion of immersion was estimated using atomic absorption spectrophotometer. A GBC 902 double-beam atomic absorption spectrophotometer was used to determine the concentration of zinc. The idea was to know the amount of Zn adsorbed/entrapped on the substrate.

2.7. Electrochemical tests

The effect of Zn ions on the corrosion behavior of mild steel and SS 304L was also investigated by carrying out electrochemical tests like free corrosion potential measurements and potentiodynamic polarization measurements. The tests were carried out in both distilled water and artificial seawater containing varying concentration of Zn ions at room temperature.

2.7.1. Free corrosion potential measurement

The free corrosion potential measurements (E_{corr}) were carried out in the absence and presence of varying concentration of Zn ions at room temperature under static condition. The change in voltage against SCE used as reference electrode was plotted vs. time. The steel specimen was connected to a wire having alligator clip on both the ends. One end of the alligator clip was attached to the steel specimen and placed into the test solution and other end was connected to a multimeter.

2.7.2. Potentiodynamic polarization measurements

The potentiodynamic polarization measurements were conducted on EG&G potentiostat/galvanostat Model 263A. The experiments were carried out using a corrosion cell from EG&G; model K 0047, with Ag/AgCl electrodes (saturated KCl) as reference and Pt wire as counter electrode. The potentiodynamic polarization measurements were carried out using a scan rate of 0.166 mV commencing at a potential above 250 mV more active than stable open circuit potential. To observe the effect of Zn in a given aqueous medium potentiodynamic curves were obtained under similar experimental condition except a periodic change in Zn ions concentration. However, before starting the scanning at each concentration, the specimen was stabilized for about 1 h for attaining a steady state which was shown by a stable potential.

2.8. Scanning electron microscope (SEM) and energy dispersive X-ray spectroscopy (EDS)

The surface morphology of the steel samples before and after immersion in distilled water and artificial seawater containing Zn ions were studied, using SEM (Model: LEO Electron Microscopy Ltd. Cambridge, England, and JEOL JSM-6510LV—with EXS INCA, Oxford, England). EDS analysis was also used for element analysis of the corroded steel surface.

3. Results and discussion

The effect of Zn ions on the corrosion behavior of mild steel and SS 304L was studied at room temperature under different experimental conditions using immersion and electrochemical techniques. Mild steel shows a complex dependence of corrosion rate on pH. In pH range 5–9 (near neutral pH range) the direct role of pH on the corrosion rate of steel has not been established [15]. The major reaction governing corrosion in most practical application in this pH range is the reduction of oxygen present in the solution. The pH of the test solutions with immersed steel samples was monitored during the entire immersion period. At pH 4.0 there was a slight increase in the pH of the solutions during early period of immersion. Thereafter, there was no appreciable effect of immersion period on the pH of the test solutions (Fig. 1). Therefore, under static condition, except for the test solutions at pH 4.0 the pH range under study is not expected to play a significant role in corrosion and any change in the corrosion behavior of steel may be expected to be brought about by other experimental variables such as presence of Zn ions and dissolved oxygen in the aqueous medium. Considering the effect of Zn ion concentration on the corrosion rate of mild steel for different immersion period in both the aqueous medium under aerated static and dynamic conditions. The immersion period does not appear to significantly affect the corrosion rate of mild steel in the presence of Zn ions. Further, the effect of Zn ion concentrations on the corrosion rate of mild steel is not much pronounced except at higher concentration of the metal.

The typical corrosion rate vs. Zn ion concentration plot for mild steel under aerated, static conditions for 1 month immersion period is shown in Fig. 2. In acidic medium, at 100 ppm Zn there is a slight decrease in corrosion rate, whereas in near neutral or basic medium there is an increase in corrosion rate at the same concentration. In static condition there appears to be no significant effect of aqueous medium on the corrosion rate of mild steel. The estimation of Zn in the test

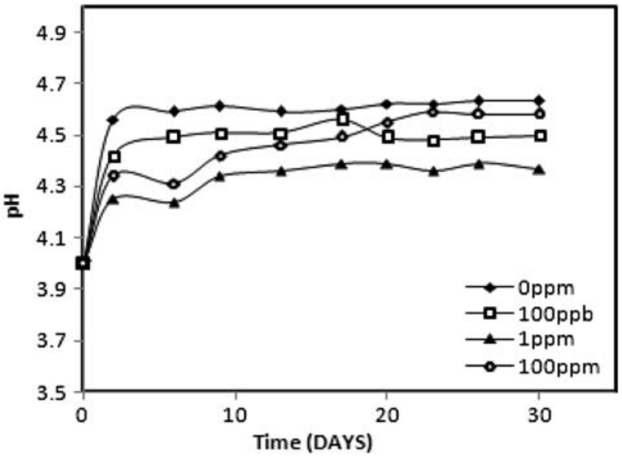


Fig. 1. pH vs. time plot in the absence and presence of varying concentration of Zn ions for mild steel immersed in distilled water at pH 4.0.

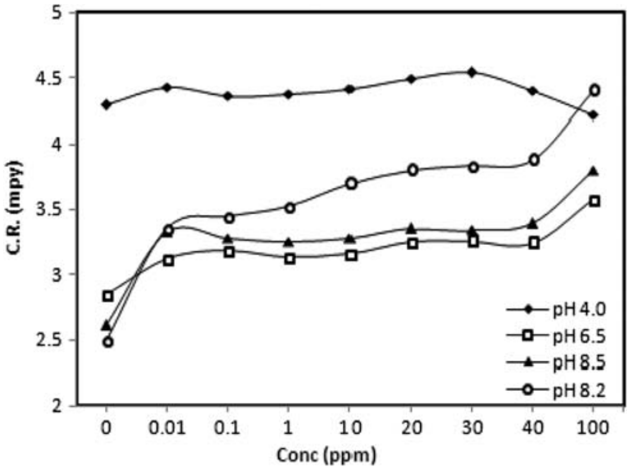


Fig. 2. Corrosion rate vs. Zn ion concentration plot for mild steel immersed in distilled water and artificial seawater under aerated, static conditions at different pH for 720 h immersion period.

solutions after completion of immersion indicates that most of the Zn ions added during the immersion is present in aqueous medium. In acidic medium, in the presence of dissolved oxygen and Zn ions, the corrosion of mild steel may be accounted by the following anodic and cathodic reactions:

$$\text{Anodic reaction:} \quad Fe \rightarrow Fe^{2+} + 2e^- \tag{1}$$

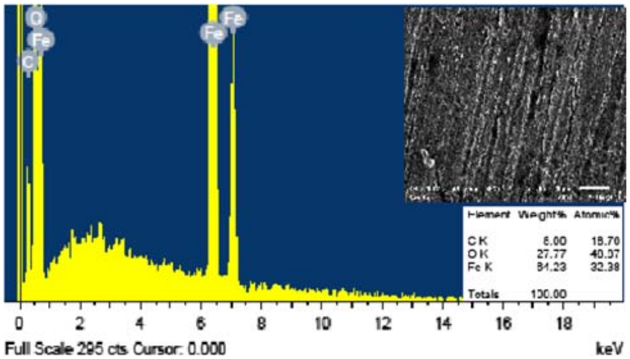
$$\text{Cathodic reactions:} \quad 2H^+ + 2e^- \rightarrow H_2 \tag{2}$$

$$1/2O_2 + H_2O + 2e^- \rightarrow 2OH^- \tag{3}$$

$$Zn^{2+} + 2e^- \rightarrow Zn \tag{4}$$

However, Zn being anodic [electrode potential of Zn = $E_{Zn^{2+}/Zn}$ (−0.760 V)] to mild steel is unlikely to get reduced and deposited on the steel thereby denying the occurrence of Eq. (4). Therefore, the presence of Zn ions in the solutions is not expected to increase the rate of iron dissolution. However, at acidic pH, Zn will be electrostatically attracted to the cathodic sites on the mild steel surface thereby, blocking the reaction path of iron dissolution [16]. The effect is less pronounced at lower Zn ion concentration. This is the reason that in the presence of lower concentration of Zn the corrosion rate is almost unaffected, whereas at higher concentration of Zn a slight reduction in corrosion rate is observed.

However, EDS analysis of the mild steel surface (Fig. 3) did not show the presence of any Zn on the surface. This may be attributed to the fact that under



Element	Weight%	Atomic%
C K	6.00	18.70
O K	27.77	40.07
Fe K	64.23	32.38
Totals	100.00	

Fig. 3. EDS analysis of mild steel immersed in distilled water for 24 h duration containing 100 ppm Zn ion.

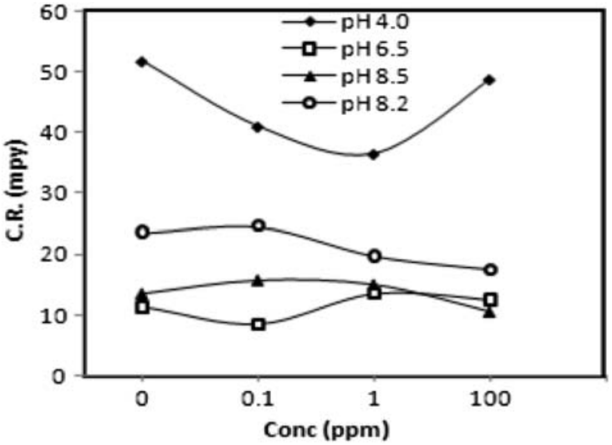


Fig. 4. Corrosion rate vs. Zn ion concentration plot for mild steel immersed in distilled water and artificial seawater under aerated, dynamic condition for 24 h duration.

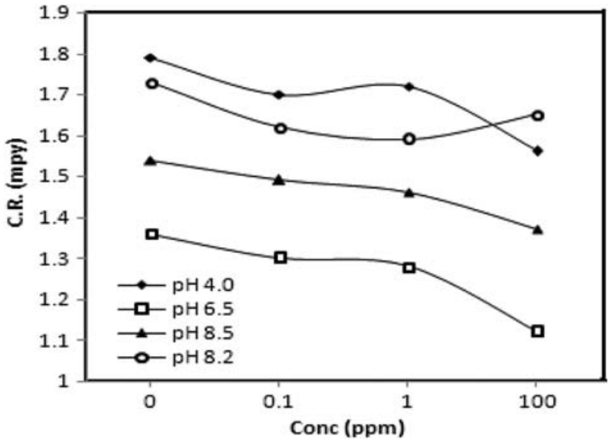


Fig. 5. Corrosion rate vs. Zn ion concentration plot for steel immersed in distilled water and artificial seawater under deaerated, static conditions for 12 h duration.

Table 3
Effect of Zn on the corrosion rate of SS 304L under different experimental conditions at room temperature

Aqueous medium	pH	Zn ions conc. (ppm)	Corrosion rate as obtained by weight loss measurements (mpy)			Corrosion rate as obtained by solvent analysis of iron ions (mpy)			Conc. of Zn ion left over in the test solutions (ppm)		
			720 h	4320 h	8760 h	720 h	4320 h	8760 h	720 h	4320 h	8760 h
Distilled water	4.0	Blank	0.0	0.0	0.0	0.0	0.0	0.0	–	–	–
		1	0.011	0.0059	0.0063	0.0103	0.0055	0.0058	0.7	0	0
		10	0.0092	0.011	0.0086	0.0088	0.0097	0.0083	9.2	9	8.5
		20	0.0074	0.0091	0.0082	0.0072	0.0087	0.0078	19.1	18.7	18
		30	0.012	0.0078	0.0062	0.0118	0.009	0.0066	28.1	28.9	28
		40	0.014	0.0086	0.0058	0.0126	0.013	0.0053	38.9	38	37.1
		100	0.0095	0.017	0.0067	0.0098	0.0154	0.0063	98.7	97.1	97.5
Distilled water	6.5	Blank	0.0	0.0	0.0	0.0	0.0	0.0	–	–	–
		1	0.0065	0.0085	0.0073	0.064	0.0078	0.0071	0.8	0	0
		10	0.0099	0.009	0.0079	0.0096	0.0085	0.0076	9.5	9.1	8.2
		20	0.0453	0.0085	0.0042	0.0397	0.0088	0.0037	19.2	18.7	17.9
		30	0.0079	0.0069	0.0038	0.0074	0.0066	0.0033	28.3	28.7	27.5
		40	0.0184	0.014	0.0087	0.0183	0.0137	0.082	39.6	37.4	37
		100	0.0256	0.0078	0.0097	0.0261	0.0077	0.094	98	97.2	96.6
Distilled water	8.5	Blank	0.0	0.0	0.0	0.0	0.0	0.0	–	–	–
		1	0.0429	0.0088	0.0078	0.0417	0.0079	0.0073	0	0	0
		10	0.0115	0.0069	0.0032	0.0106	0.0068	0.0031	8.2	7.9	6.7
		20	0.0224	0.0096	0.0091	0.0218	0.0099	0.0089	19.5	18.4	17.2
		30	0.0122	0.0084	0.0069	0.0119	0.0081	0.0067	29	28.2	27.1
		40	0.0174	0.0076	0.0087	0.0171	0.0075	0.084	38.4	37.6	36.7
		100	0.0298	0.0078	0.0095	0.0287	0.078	0.0088	97.1	97	96.9
Artificial seawater	8.2	Blank	0.0	0.0	0.0	0.0	0.0	0.0	–	–	–
		1	0.0114	0.0088	0.0096	0.011	0.0086	0.0097	0	0	0
		10	0.0162	0.0085	0.0058	0.0158	0.0083	0.0057	8.3	8	7.7
		20	0.0183	0.0077	0.0039	0.0179	0.0071	0.0036	18.9	18.6	17.9
		30	0.0273	0.0089	0.0067	0.0269	0.0086	0.0065	29.1	28.2	27.4
		40	0.0306	0.0086	0.0058	0.0297	0.0085	0.0059	38	37.7	36.6
		100	0.0321	0.0095	0.0078	0.0286	0.0093	0.0077	98.2	97.1	96.1

experimental conditions Zn was physically adsorbed and removed during the cleaning of the specimen. At pH 7 and above, Zn ions in the solutions are precipitated as ZnO or $Zn(OH)_2$ thereby denying the electrostatic attraction between Zn ions and the cathodic sites at steel surface. This prevented the adsorption of Zn on the cathodic site thereby increasing the corrosion rate of mild steel. Under dynamic condition, in addition to the presence of Zn ions the agitation of solution and presence of some fine suspended solid particles may also affect the corrosion rate. In presence of dissolved oxygen the corrosion of carbon steel is governed by cathodic diffusion and solution agitation is expected to cause an increase in corrosion rate. This accounts for the observed higher corrosion rate of carbon steel under dynamic condition (Fig. 4). The slightly higher corrosion rates observed in raw seawater than distilled water under dynamic condition may be accounted due to the presence of aggressive chloride ions present in artificial seawater.

Considering the results of immersion test on the corrosion rate of mild steel under static and deaerated conditions (Fig. 5), there is an obvious decrease in the corrosion rate of mild steel, due to absence of cathodic reaction involving reduction of oxygen. Considering the results of immersion test for SS 304L in both distilled and artificial seawater. There is no effect of Zn ions on the corrosion rate of SS 304L. The observed behavior is due to the absence of anodic reaction as SS 304L remains unaffected in both distilled water and artificial water (Table 3). The results of corrosion rate as estimated by solvent analysis of iron ions is consistent with the results obtained by the weight loss measurements.

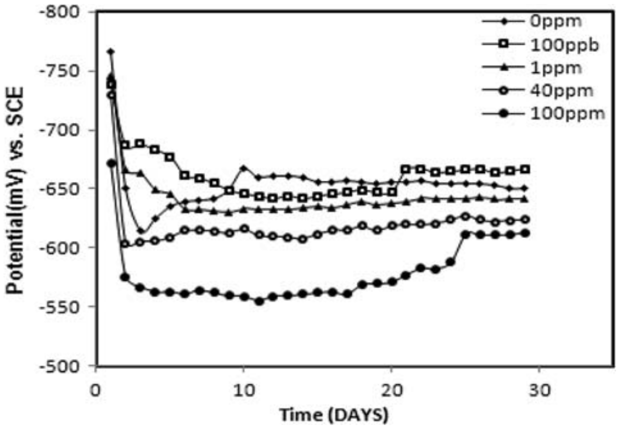


Fig. 6. E_{corr} vs. time plot for mild steel immersed in distilled water at pH 4.0 containing various concentration of Zn ion.

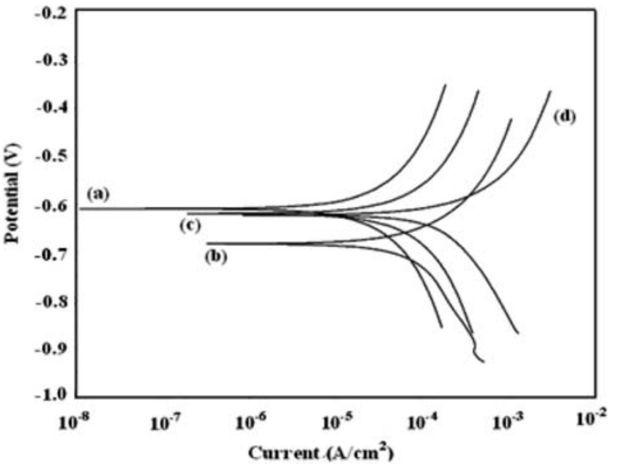


Fig. 7. Potentidymanic polarization curves for mild steel in distilled water (pH = 4.0) in the absence and presence of varying concentration of Zn (a) blank (b) 100 ppb (c) 1 ppm (d) 100 ppm.

Considering the results of OCP measurements the presence of Zn in the aqueous medium does not significantly affect the E_{corr} of mild steel except at pH 4.0 (Fig. 6).

At pH 4.0, in presence of Zn ions, an initial decrease in negative potential was observed in the presence of 100 ppb, 1 ppm, 40 ppm, and 100 ppm of zinc, and a potential below −650 mV (steady potential of mild steel in the absence of Zn ions) is maintained for the time period extending 30 days. The nobler shift in E_{corr} in the presence of 100 ppm Zn ions confirms that the adsorbed Zn ions on the mild steel depressed the anodic current of the corrosion reaction and offered resistance to corrosion. The above observations support the results of immersion test at pH 4. At other pH, in general, a negative shift in E_{corr} is observed.

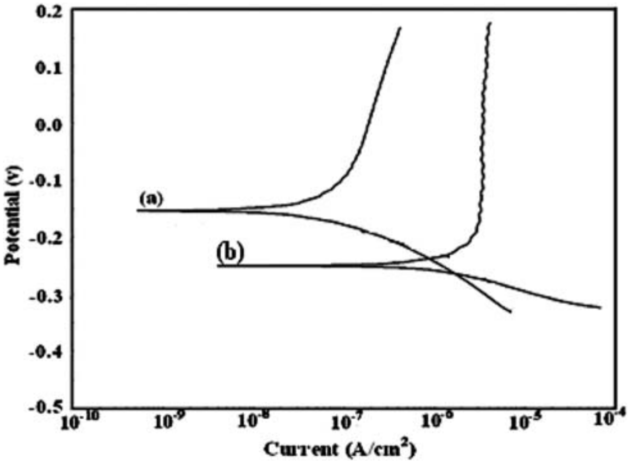


Fig. 8. Potentidymanic polarization curves for SS 304L steel in distilled water (pH = 6.5) in the absence and presence of varying concentration of Zn (a) blank (b) 100 ppm.

Table 4
Corrosion parameters for mild steel and SS 304L in distilled water as obtained by potentiodynamic polarization measurements

Alloy	Aqueous medium	pH	Zn ion conc. (ppm)	E_{corr} (mv)	I_{corr} (A/cm^2)	Corrosion rate (mpy)
Mild steel	Distilled water	4.0	0 ppm	−610	4.7×10^{-4}	107.7
			100 ppb	−682	5.6×10^{-4}	129.0
			1 ppm	−620	8.5×10^{-4}	197.0
			100 ppm	−623	7.7×10^{-4}	178.2
SS 304L	Distilled water	6.5	0 ppm	−151	1.0×10^{-7}	0.024
			100 ppm	−256	2.4×10^{-6}	0.55

A negative shift in E_{corr} is attributed to the activation of corrosion process at the metal surface.

The potentiodynamic polarization curves for mild steel and SS 304L are shown in Figs. 7 and 8. The corrosion parameters as derived by the potentiodynamic curves are shown in Table 4.

Considering the potentiodynamic polarization curves for mild steel, at pH 4, in the presence of Zn ions the corrosion potential (E_{corr}) of steel was found to shift towards more negative values relative to the potential of mild steel in the absence of Zn ions. Also, the corrosion current density (I_{corr}) increases in the presence of Zn ions resulting in an increased corrosion rate. The result is contrary to the results of weight loss measurements. The increased value of corrosion rate in the presence of Zn is explained on the basis of instantaneous measurement of the corrosion parameters and rapid rate of electron transfer reactions. The identical trend is observed in case of SS 304L.

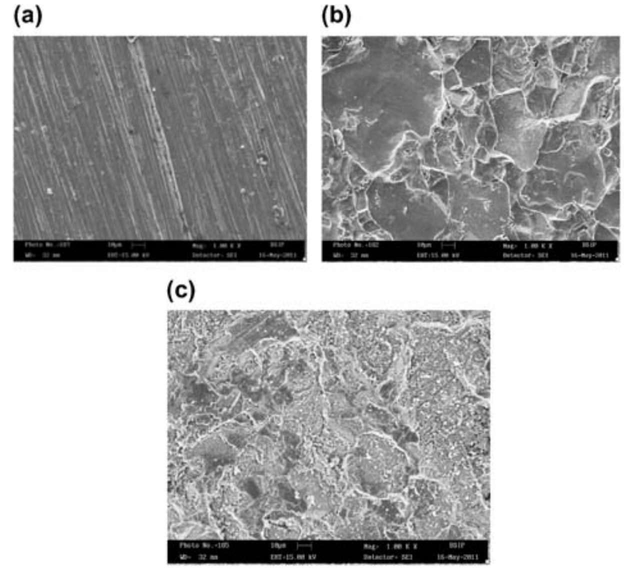


Fig. 9. SEM micrographs of (a) mild steel (b) mild steel immersed in distilled water at pH 6.5 containing 100 ppm Zn ion (c) mild steel immersed in artificial seawater at pH 8.2 containing 100 ppm Zn ion.

To observe the effect of Zn ions on the localized corrosion behavior of steel the surface morphology of the corroded steel surface was also examined using scanning electron microscopy (SEM) and the micrographs are produced in Fig. 9. The SEM micrographs did not show any evidence of localized attack in the presence of metal ions.

4. Conclusion

1. (1) The result of immersion tests showed that under static condition, in the presence of dissolved oxygen, effect of Zn ion concentrations on the corrosion rate of mild steel is not much pronounced except at higher concentration of the metal.
2. (2) In acidic medium a slight decrease in corrosion rate is observed, whereas in near neutral or basic medium an increase in corrosion rate is observed. There appears to be no significant effect of immersion period and aqueous medium on the corrosion rate of mild steel.
3. (3) Under aerated dynamic condition, the corrosion rate of mild steel is dependent on the solution agitation and nature of aqueous medium.
4. (4) Under deaerated static condition, the corrosion rate of mild steel is considerably lowered in both distilled water and artificial seawater as compared to aerated condition.
5. (5) SS 304L is unaffected in both distilled and artificial seawater and the presence of Zn ions in the medium showed no effect on the corrosion rate.
6. (6) The results of immersion tests find support from free corrosion potential measurements. The corrosion parameters for mild steel at pH 4 as derived from potentiodynamic polarization measurements showed contrary results as compared to the results of immersion tests. The increased value of corrosion rate in the presence of Zn is explained

on the basis of instantaneous measurement of the corrosion parameters and rapid rate of electron transfer reactions.

(7) The microscopic examination of the mild steel coupons subjected to immersion in distilled and artificial seawater did not show any evidence of localized attack in the presence of different concentration of Zn ions.

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