# **Electrochemical study on the corrosion of rusted carbon steel in dilute NaCl solutions**

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## Abstract

**Purpose** – The purpose of this study was to investigate the corrosion of rusted carbon steel in dilute NaCl solution, with the purpose of exploring the effect of the rust layer on metal corrosion and establishing a corrosion model for rusted iron.

**Design/methodology/approach** – The corrosion behavior of rusted carbon steel in dilute NaCl solution was studied by means of weight-loss determinations, scanning electron microscopy, Raman spectrometry and electrochemical techniques.

**Findings** – The results indicated that carbon steel had a similar corrosion behavior in all three NaCl solutions. The iron rust, which consisted of a thin  $\gamma$ -FeOOH layer and a thick Fe<sub>3</sub>O<sub>4</sub> layer, can facilitate the corrosion process of carbon steel via reduction of  $\gamma$ -FeOOH and the large area cathode of Fe<sub>3</sub>O<sub>4</sub>. Hence, the corrosion rate of carbon steel was accelerated significantly and finally was determined by the limiting diffusion rate of oxygen. **Originality/value** – A corrosion model of rusted carbon steel was established, suggesting that iron rust formed in all slightly acidic waters with low alkalinity probably promotes the corrosion of carbon steel. Anti-corrosion measures for iron in this type of solution, such as desalination water, should be aimed to reduce the promotional effect of the rust layer on metal corrosion.

Keywords Carbon steel, Dilute NaCl solution, Rust layer, Corrosion behavior, Alkalinity

Paper type Research paper

# 1. Introduction

The reverse osmosis (RO) and multistage flash (MSF) processes are the two main industrial processes used for seawater desalination (Malik et al., 2010). RO product water has a total dissolved solids (TDS) of about 200-500 mg·L<sup>-1</sup>, while the MSF product water exhibits the content of TDS of 10-50 mg·L<sup>-1</sup> due to the difference of their desalting principles (Malik et al., 2006; Van der Bruggen and Vandecasteele, 2002). Carbon steel is widely used as the material of transmission pipelines for desalination of water, especially in China. It has been reported that the carbon steel pipe suffers severe corrosion in both types of desalinized water, but the corrosion behaviors of carbon steel are still not clearly understood (Marangou and Savvides, 2001; Hu et al., 2012; Wang et al., 2011). The desalinized water produced either from the RO or the MSF process can be considered as dilute NaCl solution with different concentrations, because the main dissolved salt in them is NaCl. Therefore, studying the corrosion of carbon steel in dilute NaCl solutions can be conducive to understanding the corrosion behaviors of carbon steel in both types of desalinized water. To date, few researchers have focused on the corrosion of rusted carbon steel in dilute NaCl solutions.

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Anti-Corrosion Methods and Materials 61/3 (2014) 139–145 © Emerald Group Publishing Limited [ISSN 0003-5599] [DOI 10.1108/ACMM-05-2013-1261] In the present investigation, the corrosion behavior of carbon steel in dilute NaCl solutions was studied by weight-loss determinations, scanning electron microscopy (SEM), Raman spectrometry (RS) and electrochemical techniques. This study researched the corrosion of rusted carbon steel, with the purpose of exploring the effect of the rust layer on corrosion of the metal and establishing a corrosion model for rusted iron.

# 2. Experimental procedures

## 2.1 Materials and solutions

Q235A carbon steel was used as the test material. The dimensions of the test specimens were  $40 \times 13 \times 2 \text{ mm}^3$ , and the main constituents were (in weight per cent) C 0.10, Si 0.18, Mn 0.30, S 0.02, P 0.02 and Fe balance. All of the specimens were ground to 1000 grit emery paper, and then scrubbed with anhydrous ethyl alcohol and acetone. Afterward, the specimens were put into a dryer, and their size and weight were measured before use.

The NaCl test solutions, with concentrations of 0.001, 0.005 and 0.010 mol·L<sup>-1</sup>, were prepared from reagent grade NaCl and deionized water.

## 2.2 Weight-loss tests

Weight-loss tests were carried out using a rotary coupon rack. The rotary coupon rack had 12 rotating bars that moved clockwise with a rotating speed ranging from 45 to 180 rpm. The specimens were suspended at the bottom of the bars and rotated in the dilute NaCl solutions. The experimental conditions were chosen according to China National Standard GB/T 18175-2000 as follows: the rotation speed of the bars was 95 rpm; the testing temperature was 30°C.

After exposure, some specimens were washed with deionized water to clean their surfaces, and then were dried for 12 hours before weighing. The corrosion rate of the carbon steel was calculated from the weight loss of the specimens. The calculation formula of corrosion rate was as follows:

$$V = \frac{87600(W_0 - W_1)}{\rho St}$$
(1)

where V was the corrosion rate of carbon steel,  $\text{mm}\cdot\text{y}^{-1}$ ;  $W_0$  was the initial mass of specimen, g;  $W_1$  was the final mass of specimen removing rust layer after experiment, g;  $\rho$  was the density of metal, g·cm<sup>-3</sup>; S was the surface area of specimen, cm<sup>2</sup>; and t was the immersion time, h.

To study the characteristics of iron rusts, the other specimens with corrosion products covered were used to perform the rust layer analysis tests.

#### 2.3 Rust layer analysis

The SEM examination was conducted using a QUANTA 200, manufactured by FEI (The Netherlands), and RS was performed using an RM-1000, manufactured by Renishaw (the United Kingdom) to establish the analysis of the morphology and composition of rust layer on metal surface.

The cross-sectional morphology of rust layer was photographed using the SEM. The components of the inner and the outer rust layers were analyzed using RS. Raman excitation was performed using an  $Ar^+$  laser with a wavelength of 514.5 nm and a laser power of < 0.4 mW.

#### 2.4 Electrochemical tests

Electrochemical tests were performed using a PGSTAT128N Autolab electrochemical workstation (Metrohm, China Ltd.). A three-electrode system was used during these tests. The working electrodes were the carbon steel samples covered by epoxy resin, and their working area was 1 cm<sup>2</sup>. All of the working electrodes were ground to a 1,000 grit finish using emery paper and were scrubbed with anhydrous ethyl alcohol and acetone before use. The reference electrode was a saturated calomel electrode and the auxiliary electrode was a platinum electrode. The testing medium, with a temperature of  $25 \pm 1^{\circ}$ C, was 0.005 mol·L<sup>-1</sup> NaCl solution.

Before taking electrochemical measurements, some working electrodes were immersed in 0.005 mol·L<sup>-1</sup> NaCl solution using the rotary coupon rack to form rust layers. The test conditions were as follows: the measuring time of the open circuit potential (OCP) was one hour; the measuring frequency for the AC impedance measurements was 100 kHz to 0.01 Hz; the potentiodynamic polarization curves were made from OCP to -0.35 V (vs OCP), after stabilization, from OCP to +0.30 V (vs OCP), with a scanning rate of 0.33 mV·s<sup>-1</sup>.

# 3. Results

#### 3.1 Corrosion rate of carbon steel

A total of 12 different immersion durations were used for the weight-loss test for specimens: 2, 6, 12, 24, 48, 72, 120, 168, 240, 360, 480 and 720 hours. The results of the tests are shown in Figure 1.

As indicated in Figure 1, it can be clearly observed that the corrosion rates of carbon steel in all three solutions had a steep

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Figure 1 Relationship between the corrosion rate of carbon steel and the immersion time in dilute NaCl solutions with different concentrations



rise during initial immersion duration and stabilized subsequently at about  $1.35 \text{ mm} \cdot \text{y}^{-1}$ . The increase in corrosion rate in 0.010 mol·L<sup>-1</sup> NaCl solution was much larger than that in 0.001 mol·L<sup>-1</sup> NaCl solution, suggesting that the corrosion of carbon steel was enhanced by the chloride ions during the initial immersion (Zheng and Yang, 2010).

The increase in the corrosion rate of the carbon steel was accompanied by the accumulation of the rust layer on metal surface. Therefore, it could be assumed that the rust layers formed in dilute NaCl solutions could accelerate the corrosion of carbon steel. In addition, the corrosion processes would be controlled by the same factor(s), which tended to make the corrosion rates of steel consistent. Because carbon steel had similar corrosion behavior in all three dilute NaCl solutions, 0.005 mol·L<sup>-1</sup> NaCl solution was chosen as the main corrosion media for the rest of the experiments.

#### 3.2 Basic characteristics of rust layer

#### 3.2.1 Analysis results of SEM

The cross-section morphology of the rust layer for the 480-hour immersion in 0.005 mol· $L^{-1}$  NaCl solution was photographed by SEM. Experimental results are shown in Figure 2.

As shown in Figure 2, the rust of carbon steel had a two-layer structure, i.e. a thick inner layer and a thin outer layer. It was found by observing the structure of the rust that the outer rust layer was yellow and the inner rust layer was black. After removing the rust layer, no pitting of the metal surface was observed by the naked eye, indicating that the rust layer could inhibit the effect of chloride ions on the corrosion of carbon steel (Zheng and Yang, 2010).

#### 3.2.2 Analysis results from RS

To explore the components of the outer and inner rust layer, the rust layer for 480-hour immersion in 0.005 mol·L<sup>-1</sup> NaCl solution was analyzed using RS. The findings are shown in Figure 3(a) and 3(b).

According to the characteristic Raman peaks of the iron oxides (Singh et al., 2008; Zhang et al., 2011), it was

**Figure 2** Cross section morphology of rust layer for 480-hour immersion in  $0.005 \text{ mol} \cdot \text{L}^{-1}$  NaCl solution



**Figure 3** Raman spectra of (a) outer rust layer and (b) inner rust layer for 480-hour immersion in 0.005 mol·L<sup>-1</sup> NaCl solution



concluded that the main components of the outer and inner rust layers were  $\gamma$ -FeOOH and Fe<sub>3</sub>O<sub>4</sub>, respectively. The content of  $\alpha$ -FeOOH was very low. Therefore, it was known that the yellow  $\gamma$ -FeOOH existed mainly in the outer rust layer, and the black Fe<sub>3</sub>O<sub>4</sub> predominated in the inner rust layer.

In consequence, it is known that the rust layer on the carbon steel consisted of a thin  $\gamma$ -FeOOH layer and a thick Fe<sub>3</sub>O<sub>4</sub> layer in dilute NaCl solutions, which is similar to that formed in RO product water (Hu *et al.*, 2012). Considering the change trend in the corrosion rate of carbon steel, it could be assumed that the rust layer, containing a large quantity of Fe<sub>3</sub>O<sub>4</sub>, a small quantity of  $\gamma$ -FeOOH and a little amount of  $\alpha$ -FeOOH, could accelerate the corrosion of carbon steel. Hence, the electrochemical characteristics of rust layers were investigated in Section 3.3.

## 3.3 Electrochemical characteristics of rust layer

3.3.1 Results of polarization curve measurement

Polarization curves for electrodes with different immersion times were conducted, and the results are shown in Figure 4.

As indicated in Figure 4, the anodic current of electrodes decreased slowly with the formation of rust layers, indicating that the rust layer could partially suppress the anodic reaction (Wang *et al.*, 2010). Compared to the cathodic current of the bare electrode (0.5-hour immersion), the cathodic currents of the rusted electrodes were much larger, which indicated that the rust layer participated in cathode reaction to facilitate the cathodic process. This phenomenon had been suggested to be attributed to the reduction of ferric rust (Wang *et al.*, 2010; Li *et al.*, 2008). According to the results of RS, it was assumed that the ferric rust, which was reduced easily in 0.005 mol·L<sup>-1</sup> NaCl solution, was  $\gamma$ -FeOOH.

## 3.3.2 Results of AC impedance measurements

Impedance measurements of electrodes for different immersion times were performed, and the results are shown in Figure 5.

Only partial representative results are presented in Figure 5 to simplify the Nyquist diagram. For a bare steel (0.5-hour immersion), its Nyquist diagram illustrated only one double-





Figure 5 Nyquist diagram of electrodes for various immersion durations



layer capacitive semicircle. As the immersion time was extended, each Nyquist curve of rusted electrodes consisted of a capacitance arc and a line.

The equivalent circuit for the naked electrode is shown in Figure 6(a). Each Nyquist curve for the rusted electrodes in dilute NaCl solution consisted of a single capacitance arc and a line, which was similar to that in the 3 per cent NaCl solution (Hu *et al.*, 2013). Hence, the equivalent circuit shown in Figure 6(b) is proposed for the rusted electrodes. The fitted results are summarized in Table I.

As shown in Table I, the value of transfer resistance  $(R_i)$  decreased sharply at the beginning, and then trended to level off. The change trend of  $R_i$  values showed that the rust layer could promote the corrosion process of carbon steel significantly during initial immersion, and then the corrosion rate of the steel would stabilize at a high value with the thickening of the rust layer. This conclusion was consistent with the result presented in Figure 1.

The Warburg coefficient  $(Y_0)$  could represent the diffusion ability of oxygen. The experiment results in Table I showed that the value of  $Y_0$  was about 0.1  $\Omega^{-1} \cdot \text{cm}^{-2} \cdot S^{-0.5}$ , which was much larger than the  $Y_0$  value for oxygen transfer in the rust

**Figure 6** Equivalent circuits of electrodes.  $R_s$ : resistance of solution;  $R_t$ : transfer resistance;  $C_d$ : capacitance of the electric double layer; and  $Z_d$ : diffusion impedance



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Table I Analytical results of Nyquist curves of electrodes for various immersion durations

Time/hour	$R_s/(\Omega \cdot cm^2)$	$R_t/(\Omega \cdot \mathrm{cm}^2)$	$Y_0/(\Omega^{-1} \cdot cm^{-2} \cdot S^{-0.5})$
0.5	171.3	2,425	_
36	246.7	617	0.08610
72	244.3	161.7	0.09290
120	189.4	125.7	0.09741
168	226.3	66.3	0.11915
240	225.7	51.6	0.13725
380	208.5	62.1	0.10539
480	186.3	49.1	0.12732
720	182.4	68.3	0.10871
800	200.6	75.2	0.08608

layer (Bai *et al.*, 2006; Melchers, 2003). Furthermore, the  $Y_0$  value for rusted electrode in the dilute NaCl solution is similar to that in the 3 per cent NaCl solution (Hu *et al.*, 2013). These results indicate that the diffusion process of oxygen in solution determined the corrosion of rusted carbon steel, suggesting that oxygen could be reduced directly on the surface of the rust layer.

As shown in Figure 2, the iron rust consisted of a thick inner layer and a thin outer layer. To explore the effect of each layer on metal corrosion, the electrode with an immersion time of 800 hours was treated as follows, before AC impedance measurements were made: removal of the outer layer, removal of the inner layer (without rust layer covered). The surface morphologies of electrode are shown in Figure 7. The results of the AC impedance tests are shown in Figure 8 and Table II.

As shown in Table II, the  $R_t$  value became much larger when the outer rust layer was removed, proving that the outer rust layer could promote the corrosion of carbon steel. The  $R_t$ value of an electrode covered by the inner rust layer was much smaller than that of a bare electrode, suggesting that the inner rust layer also played a significant role in accelerating the corrosion of carbon steel.

Therefore, it is found that the formation of both the outer rust layer ( $\gamma$ -FeOOH layer) and the inner rust layer (Fe<sub>3</sub>O<sub>4</sub> layer) facilitated the corrosion process of carbon steel in dilute NaCl solutions.

## 4. Discussion

After carbon steel is immersed in dilute NaCl solutions, the corrosion reactions can occur as follows: Anode region:

$$Fe \rightarrow Fe^{2+} + 2e$$
 (2)

Cathode region:

$$O_2 + 2H_2O + 4e \rightarrow 4OH^-$$
(3)

Before a rust layer is formed, chloride ions are easy to absorb onto the metal surface. By accelerating the dissolution of the iron, chloride ions can facilitate the corrosion of the carbon steel (Zheng and Yang, 2010). Hence, the corrosion rate of carbon steel in 0.010 mol·L<sup>-1</sup> NaCl solution is much larger than that in 0.001 mol·L<sup>-1</sup> NaCl solution during the early stages of immersion.

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Figure 7 Pictures of electrode (a) with rust layer covered; (b) removal of the outer rust layer; and (c) removal of the inner rust layer



Figure 8 Nyquist plots of carbon steel electrode after rust layer treatment



 Table II Analytical results of Nyquist curves of electrodes after rust layer treatment

Surface state	$R_s/(\Omega \cdot cm^2)$	$R_t/(\Omega \cdot cm^2)$	$Y_0/(\Omega^{-1} \cdot \mathrm{cm}^{-2} \cdot \mathrm{S}^{-0.5})$
Rust layer			
covered	200.6	75.2	0.08608
Removal of the outer layer	268.9	407.6	0.10632
Removal of the inner layer	195	2,781	_

 $Fe^{2+} + 2Cl^- + 4H_2O \rightarrow FeCl_2 \cdot 4H_2O$  (4)

$$\text{FeCl}_2 \cdot 4\text{H}_2\text{O} \rightarrow \text{Fe(OH)}_2 + 2\text{Cl}^- + 2\text{H}^+ + 2\text{H}_2\text{O}$$
 (5)

The ferrous ions (Fe<sup>2+</sup>), which do not form compounds with chloride ions, can form hydrated ions (FeOH<sup>+</sup>) in solution. The intermediate corrosion products FeOH<sup>+</sup> and Fe(OH)<sub>2</sub> can be oxidized quickly by O<sub>2</sub> and transform into  $\gamma$ -FeOOH (Zou *et al.*, 2010).

The results of electrochemical measurements indicate that the  $\gamma$ -FeOOH layer can promote the corrosion of carbon steel, which suggests that  $\gamma$ -FeOOH can be reduced in dilute NaCl solution. The reduction reaction for  $\gamma$ -FeOOH is:  $3\gamma - \text{FeOOH} + \text{H}^+ + \text{e} \rightarrow \text{Fe}_3\text{O}_4 + 2\text{H}_2\text{O}$  (6)

Therefore, the black inner rust layer (Fe<sub>3</sub>O<sub>4</sub> layer) appears quickly, and the content of Fe<sub>3</sub>O<sub>4</sub> keeps increasing with extended immersion time. Hence, the two-layer rust deposit consisted of a thin  $\gamma$ -FeOOH layer and a thick Fe<sub>3</sub>O<sub>4</sub> layer, which was formed on the metal surface in dilute NaCl solution. Evans (1965) concluded that ferrous ions and electrons could pass through an Fe<sub>3</sub>O<sub>4</sub> layer, and hence the cathodic reaction would occur on the surface of the rust layer. The experimental results in Table II indicate that the inner rust layer (i.e. the Fe<sub>3</sub>O<sub>4</sub> layer) can accelerate significantly the corrosion of carbon steel. Additionally, it has been proved that oxygen can participate in the reaction on the surface of the rust layer. Therefore, it can be concluded that the Fe<sub>3</sub>O<sub>4</sub> layer works as a large cathode area and oxygen is reduced on its surface in dilute NaCl solutions.

Due to the reduction of the  $\gamma$ -FeOOH layer and the large cathode area of Fe<sub>3</sub>O<sub>4</sub> layer, the corrosion process of carbon steel is accelerated greatly. Finally, the corrosion rate of the carbon steel is determined by the limiting diffusion rate of oxygen from solution to inner rust layer.

The limiting diffusion rate current density can be expressed as:

$$i_L = -nFD\frac{C_0}{\delta} \tag{7}$$

where D is the diffusion coefficient of oxygen;  $C_0$  is the concentration of oxygen in solution; and  $\delta$  is the thickness of diffusion layer.

In equation (7), D and  $C_0$  are directly related to the solution temperature;  $\delta$  is directly related to the rotating speed of specimens and the solution temperature. When all experimental conditions are unchanged, the values of all parameters in equation (7) are constant, and hence  $i_L$  is a certain value. In consequence, the corrosion rate of carbon steel in all dilute NaCl solutions tends to be uniform no matter how big the concentration of chlorine ions. These conclusions are consistent with the experimental results presented in Figure 1.

The corrosion model for rusted carbon steel in dilute NaCl solutions can be expressed as shown in Figure 9.

The Nernst equation for the reduction reaction of  $\gamma$ -FeOOH can be expressed as:

Figure 9 Corrosion model of rusted carbon steel in dilute NaCl solutions



$$\mathbf{E}_{\text{FeOOH/Fe}_{3}O_{4}} = \mathbf{E}_{\text{FeOOH/Fe}_{3}O_{4}}^{\theta} - \frac{2.303\text{RT}}{\text{F}}\text{pH}$$
(8)

As shown in equation (8), the reduction potential of  $\gamma$ -FeOOH is related directly to the electrolyte pH. The lower the pH value of electrolyte, the higher is the value of the reduction potential of  $\gamma$ -FeOOH. When the reduction potential of  $\gamma$ -FeOOH is higher than the corrosion potential of the carbon steel,  $\gamma$ -FeOOH begins to work as an oxidant and is reduced. The dilute NaCl solutions are slightly acidic because it is easy for CO<sub>2</sub> to dissolve into them. It is known that the slight acidity of dilute NaCl solutions is good for the reduction reaction of  $\gamma$ -FeOOH. Hoerlé *et al.* (2004) also proved that the rate of  $\gamma$ -FeOOH reduction was directly proportional to electrolyte pH.

Additionally, Lahav and Birnhack (2007) found that iron was corroded badly in natural low salinity terrestrial waters that were slightly acidic with low alkalinity. Zhang *et al.* (2011) concluded that the rust layer consisted of a  $\gamma$ -FeOOH layer and an Fe<sub>3</sub>O<sub>4</sub> layer in the 0.5 M NaCl and 0.05 M Na<sub>2</sub>SO<sub>4</sub> solution with a pH at 6-7, and considered that the Fe<sub>3</sub>O<sub>4</sub> layer could work as a large cathode area. Previous research by the present authors also proved that the rust layer formed in 3 per cent NaCl solution can facilitate the corrosion process of carbon steel (Hu *et al.*, 2013). Therefore, it is probable that the rust layer generated in all slightly acidic waters with low alkalinity can promote the corrosion of carbon steel via reduction of  $\gamma$ -FeOOH.

To slow down corrosion, anti-corrosion measures for iron in these types of solutions should be aimed to reduce the promotional effect of the rust layer on the corrosion process. Hence, adding corrosion inhibitors, which can isolate the metal and the rust layer from the solution, into the corrosion media is a promising option.

## 5. Conclusion

Due to the slight acidity of dilute NaCl solutions, the reduction potential of  $\gamma$ -FeOOH will be higher than the corrosion potential of carbon steel. Hence,  $\gamma$ -FeOOH can work as an oxidant and will be reduced, and then the Fe<sub>3</sub>O<sub>4</sub> layer can be generated quickly at the interface between the

 $\gamma$ -FeOOH layer and the metal substrate. The  $\gamma$ -FeOOH layer facilitates the corrosion of the carbon steel via reduction of the  $\gamma$ -FeOOH. Meanwhile, the Fe<sub>3</sub>O<sub>4</sub> layer works as a large cathode area and oxygen is reduced on its surface. As a result, the corrosion rate of carbon steel can be accelerated significantly and is determined overall by the limiting diffusion rate of oxygen from solution to Fe<sub>3</sub>O<sub>4</sub> layer. Because the reduction potential of  $\gamma$ -FeOOH is related to the electrolyte pH, which is directly related to the alkalinity of solution, it is probable that iron rust formed in all slightly acidic waters with low alkalinity can promote the corrosion of carbon steel via the reduction of  $\gamma$ -FeOOH.

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