A Synergistic Acceleration of Corrosion of Q235 Carbon Steel Between Magnetization and Extracellular Polymeric Substances

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Abstract In this work, surface characterization and electrochemical measurement were employed to investigate the effects of magnetic field (MF) on the corrosion of Q235 carbon steel in a NaCl solution containing sulphate-reducing bacteria (SRB) or extracellular polymeric substances (EPS). Results demonstrated that a 150 mT MF enhanced steel corrosion in a SRB-containing NaCl solution by 202% calculated from weight loss with pitting corrosion as the main corrosion type. Either EPS or MF rendered steel corrosion, but a synergistic interaction between MF and EPS boosted up steel corrosion. This synergistic enhancement could be referred to the alteration in orientation of EPS induced by MF. The presence of higher percentage of chloride ions on the carbon steel surface manifested that MF initiated the erosion of chloride ions on the carbon steel coupon.

KEY WORDS: Sulphate-reducing bacteria; Magnetic field; Extracellular polymeric substances; Microbiologically influenced corrosion

1 Introduction

Microbiologically influenced corrosion (MIC) can be found in various environments including freshwater, seawater and petroleum chemical engineering [1–4]. It is estimated that 20–30% of corrosion damage is triggered by MIC, in which

sulphate-reducing bacteria (SRB) are blamed as the chief culprits [5–7]. SRB consumes sulphate as an electron acceptor for the utilization of organic compounds and produces sulphide [8, 9]. They can tolerate comparatively wide range of temperatures, pH values, chlorides and hexavalent chromium concentrations [10].

Extracellular polymeric substances (EPS) including polysaccharides, proteins, nucleic acids, lipids and other biological molecules [11, 12] play as the key constituents for the aggregation of microorganisms [13]. EPS, an extremely hydrated gel matrix, can encourage microbial cells to form stable and synergistic consortia and shield biofilm against biocides [14, 15]. Cathodic reduction of oxygen could be retarded by the adsorbed EPS film secreted by SRB [16], protecting the metal substrate from corrosion [17].

Recently, magnetic fields (MF) were employed as an innovated technique for biological wastewater treatment [18–20]. However, the MF engendered by industrial

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electromagnetic tools or provoked by magnetically water treatment would also control metal corrosion [21–25]. The MF can change the mass transfer rate by a convective force in an electrochemical system. This force (F) is given by:

$$F = q(V \times B), \quad (1)$$

where q is the charge (C) on the species; V is the velocity ($m\ s^{-1}$); and B is the magnetic field intensity (T). A local convection flow of the solution is owing to the momentum transfer between the moving species and the solvents [26]. This phenomenon satisfies the magnetohydrodynamic force (FMHD), which is given by:

$$FMHD = J \times B, \quad (2)$$

where J is the local flux of ions. The orientation of MF determines the mass transfer rate with respect to the ion flux. Thus, a Lorentz force, and then a MHD velocity, will be applied to any charged particle moving in the interfacial layer [26].

The MF can inhibit many types of microbes [27–30]. The amounts of bacteria cells and their viability decrease with an increased MF exposure time, elevated temperatures and higher MF intensities. Furthermore, the population of planktonic SRB decreased by 15% in an extremely low-frequency magnetic field for short exposure [28].

Our previous work found that a low-intensity MF of 4 mT could delay the formation of SRB biofilm and inhibited the corrosion of 304 stainless steel [31]. However, Bahaj *et al.* [32] demonstrated that a moderate intensity MF of 150 mT enhanced the corrosion rate of pure iron in the presence of SRB. They concluded that the increase of corrosion rate in both cases was due to the upsurge of bacterial adhesion and the prompt production of an anaerobic environment, a result of the synergistic interaction between MF and SRB. Al-Abbas *et al.* [26] similarly revealed that a 300 mT MF stimulated biofilm formation and enriched corrosion of pipeline steel caused by SRB.

Based on the significance of EPS in the bacteria attachment and biocorrosion, we further investigated the effects of synergistic interaction between magnetization and EPS secreted by SRB. The objective was to figure out the contribution of MF and EPS to steel corrosion when MF was used to control MIC.

2 Experimental

Q235 carbon steel was used for corrosion coupons with the chemical composition (wt%) of C 0.3, Si 0.01, Mn 0.42, S 0.029, P 0.01 and Fe balance. The working electrodes with a diameter of 15 mm and a thickness of 2 mm were used for electrochemical measurements. To measure weight loss, coupons with the size of 50 mm × 13 mm × 1.5 mm

were prepared. All the coupons were abraded with a series of SiC waterproof abrasive papers (600, 1000 and 1200) followed by metallographic abrasive paper. After abrasion, these coupons were washed and degreased in ethanol and distilled water, respectively. All coupons were finally dried using nitrogen gas.

SRB were isolated from the sludge in Sinopec oilfield (Shandong province, China) and have already been identified in a previous report [33]. API-RP38 culture medium was used, and the corresponding composition was ($g\ L^{-1}$): K_2HPO_4 0.01, $MgSO_4 \cdot 7H_2O$ 0.2, $(NH_4)_2Fe(SO_4)_2$ 0.2, NaCl 10, yeast extract 1.0, vitamin C 0.1, in addition to 4.0 ml L^{-1} sodium lactate (pH 7.2). Before inoculation, SRB culture medium was autoclaved at 121 °C for 20 min. SRB were incubated at 37 °C.

The extraction of EPS was achieved according to the method described by the previous report [34]. After 10 days of incubation, SRB culture medium was centrifuged at 14,000 rpm for 20 min. The supernatant was concentrated to 200 mL in an 80 °C water bath and then filtered using a sterile 0.22 μm membrane to separate microbial cells. The obtained EPS was preserved in a frozen state. UV–visible spectrophotometer (CARY 50 Scan, VARIAN) and an attenuated total reflection infrared (ATR-IR) spectroscope (VERTEX 70, Bruker) were used to analyse the composition of EPS.

An optical microscope (OM, VHX-1000, KEYENCE) was used to investigate the corrosion morphology (after removing the corrosion products) of Q235 carbon steel after 8 days of incubation in a 3 wt% NaCl solution in the presence of SRB. Furthermore, the carbon steel surfaces were observed using a scanning electron microscope (SEM, Sirion200, FEI). The corrosion products were analysed using an energy dispersive X-ray detector.

The schematic illustration for electrochemical measurements is presented in Fig. 1. Electrochemical workstation (CHI660C, Chenhua) was used for all

Fig. 1 Schematic diagram of set-up for electrochemical measurements with MF

electrochemical measurements along with Pt electrode and saturated calomel electrode (SCE) as the counter electrode and reference electrode, respectively. For the group in the presence of SRB, the 3 wt% NaCl solution consisting of 10% culture medium was used as electrolyte. For the group with EPS, the Q235 working electrodes immersed in a 3 wt% NaCl solution with 5% EPS to guarantee the adsorption equilibrium with EPS for 1 h before electrochemical tests. Then electrochemical measurement was executed in the 3 wt% NaCl solution only. The MF intensity employed was 150 mT. The perturbations in the frequency range from 100 kHz to 10 mHz at open-circuit potential (OCP) with 10 mV sinusoidal was used to perform electrochemical impedance spectroscopy (EIS). The polarization curves were conducted between +200 mV (vs. OCP) and −200 mV (vs. OCP) at a scan rate of 0.5 mV s^{-1} . The cyclic double-polarization measurements were measured in the range of −1.5 to 0.1 V (vs. OCP) at a scan rate of 0.5 mV s^{-1} . The starting point of the potential sweep was established in the cathodic region of the $E-i$ (E and i represent the potential and current density, respectively) data, which was liable to minimize the oxidation reaction of iron at the electrode surface. All the data were analysed by Zview2 and Cview2 software. Each test was performed in triplicate.

3 Results and Discussion

3.1 Steel Corrosion in an SRB-Containing Solution

3.1.1 Weight Loss and Morphology

Weight loss of coupons after 8 days of inoculation in the presence of SRB with and without MF in 3 wt% NaCl solution is presented in Table 1. In this work, the coupon without MF was as control. Compared with the control, the weight loss has increased by 202% under the MF of 150 mT, demonstrating that the MF extraordinarily accelerated steel corrosion in the presence of SRB. Figure 2 illustrates the corrosion morphologies of the coupon surfaces after removing biofilm. Pitting corrosion was the major corrosion type in the presence of SRB. Moreover, the number of corrosion pits per unit area and average size of the corrosion pits measured are shown in Table 1. With the

assistance of MF, not only the number of the pits on steel surface increased remarkably, but the sizes of the corresponding pits became considerably larger. MF with higher intensity of 50 mT was helpful to accelerate SRB corrosion. These results were in good agreement with the measurement results of weight loss. SRB is capable of promoting localized corrosion [35]. This is mainly due to the heterogeneous SRB biofilm [36], and SRB can obtain electrons from Fe^0 directly [37], resulting in pitting corrosion [4]. MF can influence SRB activity and the migration of corrosive ions [31, 38], which can be one of the important reasons that MF with the intensity of 50 mT can promote SRB corrosion.

3.1.2 Electrochemical Measurements

The polarization curves of Q235 carbon steel in a 3 wt% NaCl solution in the presence of SRB after 8 days of incubation are shown in Fig. 3, and the corresponding fitted results are listed in Table 2. It showed that the corrosion current density (i_{corr}) was larger than that without MF (Table 2), indicating that MF could promote SRB corrosion. The substantial change of b_c (b_c represents the cathodic Tafel slope) indicated that MF seriously influenced the rate of cathodic reduction process. In this neutral system, paramagnetism oxygen can easily migrate to the steel surface in the presence of MF [38], thus resulting in more consumption of oxygen. Then, the cathodic reaction will be promoted, leading to a higher cathodic current density. Therefore, MF undoubtedly changed the process of oxygen reduction or the diffusion of dissolved oxygen, which also has been verified in a previous report [38].

Figure 4 illustrates Nyquist plots with SRB in the absence and presence of MF after 8 days of incubation. The EIS data were fitted by the equivalent circuit, as shown in Fig. 5, where the parallel R_{ct} and C_{dl} denote the impedance of the double layer and the double layer capacitance, and the parallel R_f and C_f correspond to the impedance of the SRB biofilms and the capacitance of the biofilm. The corresponding fitted results of R_{ct} values are shown in Table 3. The R_{ct} values are closely related to the steel corrosion. The lower R_{ct} values can lead to higher steel corrosion rates [39]. The smaller R_{ct} values under MF of 150 mT indicated a higher corrosion rate, confirming that

Table 1 Weight loss of Q235 carbon steels after 8 days of incubation in the presence of SRB with and without MF in 3 wt% NaCl solution

MF (mT)	Average mass loss (g cm^{-2})	Average size (μm)	Number of pits (pit mm^{-2})
0	0.0213	6 ± 2	50 ± 10
150	0.0431	30 ± 10	550 ± 50

Fig. 2 Images of coupons surface after exposure to 3 wt% NaCl solution after removing biofilms and corrosion products in the presence of SRB after 8 days of incubation: **a** 0 mT MF; **b** 150 mT MF

Fig. 3 Polarization curves of Q235 carbon steel in 3 wt% NaCl solution in the presence of SRB after 8 days of incubation

MF accelerated SRB corrosion. This also kept consistency with the results of the polarization curve measurements.

3.2 Corrosion of Q235 Steel in EPS-Containing Solution

3.2.1 Characterization of EPS

In this work, UV and ATR-IR measurements were used to confirm the adsorption of EPS on carbon steel and the direct interaction between the EPS macromolecules and ferrous cations. The UV spectrum of the EPS is shown in Fig. 6a, in which the significant absorbance between 260 and 280 nm was correspond to the presence of the DNA

and proteins. Figure 6b illustrates the Fourier-transformed infrared (FT-IR) and ATR-IR spectra of EPS adsorbed on carbon steel surface. Figure 6c shows the corresponding second-derivative transformation of the ATR-IR spectra in the range of 1700–1000 cm^{-1} . The distinct peaks at 1640 and 1564 cm^{-1} were assigned to the amide I (C=O stretching) and amide II groups (N–H bending and C–N stretching). Owing to the presence of carboxylic groups in proteins and carbohydrates, the peaks positioned at 1467 and 1418 cm^{-1} were dominated. The characteristic peak at 1300 cm^{-1} was indicative of amide III region of proteins (N–H in-plane bending coupled to the C–N stretching; C–H and N–H deformation) [40].

The second-derivative transformation revealed that the peak at 1564 cm^{-1} for the EPS was fragmented into two signal peaks at 1560 and 1536 cm^{-1} for the adsorbed EPS on carbon steel surface (the red arrow). Generally, the appearance of new chemical bonds due to the change of functional groups can be responsible for the splitting and shift of the peaks. It could be concluded from the results that EPS could easily adsorb on the steel surface, thus influenced the corrosion process of steel [41]. And, the organic macromolecules in EPS played the key roles.

3.2.2 Electrochemical Measurements

The cyclic double-polarization curves of Q235 carbon steel under different corrosion conditions are shown in Fig. 7, which are performed to explore the effect of EPS and MF on the electrochemical behaviour of Q235 carbon steel.

Table 2 Fitted results of polarization curves in Fig. 3 with SRB in the absence and presence of SRB after 8 days of incubation in 3 wt% NaCl solution (E_{corr} : free corrosion potential; b_a and b_c correspond to the anodic and cathodic Tafel slopes, respectively)

Static magnetic field (mT)	E_{corr} (mV)	i_{corr} (mA cm^{-2})	b_a (mV dec^{-1})	b_c (mV dec^{-1})
0	– 600	0.16	110	– 393
150	– 598	0.20	111	– 204

Fig. 4 Nyquist plots of coupons with SRB in the absence and presence of MF after 8 days of incubation in 3 wt% NaCl solution (Z' and Z'' are the real and imaginary parts, respectively)

Fig. 5 Equivalent circuit used to fit EIS data (R_s is the solution resistance, R_f and C_f the resistance and the capacitance of the biofilm, respectively. R_{ct} and C_{dl} correspond to a charge transfer resistance and a double layer capacitance, respectively)

Table 3 Fitted results of EIS data corresponding to Figs. 4 and 8 with SRB and EPS in the absence and presence of MF after 8 days of incubation in 3 wt% NaCl solution

	SRB + MF		EPS + MF			
	0 mT	150 mT	Blank	EPS	MF	EPS + MF
R_{ct} ($\Omega \text{ cm}^{-2}$)	852	673	1200	1780	2050	430

According to previous study, the shift in the corrosion potential revealed the concentration change of iron ions in the electrolyte due to the fractional oxidation of iron through anodic polarization [42]. In Fig. 7, coupon with EPS (0.42 V) exhibited larger ΔE than that of the control group (0.37 V). The enhancement of ΔE value was possibly associated with the complexation of the EPS with iron, which could cause enrichment of iron ions on the electrode surface. By the implementation of MF, the ΔE was further enhanced, signifying that the application of both MF and

EPS accelerated the formation of metal oxides. Iron with positive charge is difficult to leave electrode surface in the presence of MF, because magnetic induction lines are perpendicular to electrode surface. And, the iron ions with positive charge in bulk solution can also migrate to electrode surface. These will promote the formation of corrosion products. More importantly, the plateau in the anodic region in a potential range of -0.9 to -0.45 V versus SCE could also be attributed to the increased dissolution rate of iron from the surface, which in turn assisted the formation of a corrosion product film [42].

In addition, the corrosion current density (i_{corr}) was also obtained using Tafel extrapolation method, and the corresponding results are shown in Table 4. Either MF or EPS substantially inhibited steel corrosion in a 3 wt% NaCl solution. However, the corrosion current density was 0.36 mA cm^{-2} in the presence of both MF and EPS, which was significantly larger compared with the other groups. These imply that the synergistic effect of MF and EPS tremendously accelerated steel corrosion in a 3 wt% NaCl solution. Moreover, when the potential was < -0.94 V versus SCE, in the magnified zone as shown in Fig. 7, the current density in the group with both MF and EPS continued to increase and was significantly higher than the others. These results also indicated that the interaction between MF and EPS could accelerate steel corrosion.

Furthermore, EIS results under different corrosion conditions are provided as shown in Fig. 8. All EIS data were fitted using the same equivalent circuit as shown in Fig. 5, where the parallel R_f and C_f indicated the resistance of the film. The corresponding fitted results of EIS data are listed in Table 3. The addition of EPS resulted in the increase of impedance and R_{ct} values, demonstrating that the EPS adsorbed on the electrode surface showed slightly protective effect against the steel corrosion. The application of MF without SRB also led to the increase of the R_{ct} values and therefore slowed down the steel corrosion. However, substantial decrease in the R_{ct} value was found in the presence of both MF and EPS, suggesting the synergistic effect of MF and EPS on the corrosion of carbon steel in a 3 wt% NaCl solution. These results were in good agreement with the analysis results of polarization curves. Moreover, the Nyquist curves bent inwards at low frequencies, which could be explained by the adsorption/desorption of EPS or the adsorbed intermediary species produced during the dissolution of iron [17].

3.2.3 Surface Analysis

SEM images of coupons after 8 days of incubation in testing solution containing EPS in the absence and presence of MF are shown in Fig. 9. Without applying MF, comparatively uniform thin corrosion product film could be

Fig. 6 UV spectrum of EPS extracted from SRB culture medium **a**, FT-IR spectra of EPS, EPS adsorbed on steel surface in the absence and presence of 150 mT MF **b**, and corresponding second-derivative transformations of IR spectra **c**

Fig. 7 Cyclic double-polarization curves of Q235 carbon steel under different corrosion conditions in 3 wt% NaCl solution (the coupon without EPS and MF is as blank)

observed on the steel surface, and some polish lines also could be clearly observed (Fig. 9a, c). The presence of polish lines suggested a slight corrosion induced by EPS in the absence of MF. However, a large amount of corrosion products with some cracks could be clearly observed on the steel surface in the presence of MF (Fig. 9b, d),

Table 4 Fitted results of cyclic double-polarization curves in 3 wt% NaCl solution after 8 days of incubation under different corrosion conditions

Situation	ΔE_{corr} (V)	i_{corr} (mA cm ⁻²)
Blank	0.37 ± 0.01	0.28
EPS	0.42 ± 0.02	0.19
SMF	0.38 ± 0.01	0.21
EPS + SMF	0.45 ± 0.03	0.36

demonstrating that the presence of MF promoted the formation of corrosion products. The more corrosion products indicated the more irons dissolved from the steel matrix. So, the SEM images indirectly indicated that MF promoted the steel corrosion induced by EPS.

Additionally, elemental mapping of selected zone from Fig. 9c, d for the analysis of corrosion products was obtained by EDS, and the corresponding quantities of elements from the selected zone in Fig. 9c, d are listed in Table 5. It was confirmed by the results that iron oxides and iron chloride were the major corrosion products. Compared with the carbon steel without MF, the

Fig. 8 Nyquist plots of coupons under different corrosion conditions after 8 days of incubation in 3 wt% NaCl solution

percentage of chloride ions was higher for the coupon in the presence of MF (Table 5), which implied that MF resulted in the enrichment of chloride on the carbon steel surface. The metal surface could be stacked by EPS which was supposed to act as a barrier to aggressive ions such as chloride ions. However, MF assisted the transfer of the aggressive chloride ions, which consequently demolished

Table 5 EDS analysis results corresponding to SEM images in Fig. 9 in the absence and presence of MF in EPS-containing testing solution

MF (mT)	Situation	O (wt%)	Cl (wt%)	Fe (wt%)
0	EPS	6.63	0.72	70.06
150	EPS + MF	26.96	7.91	44.37

the protective film and therefore boosts up the corrosion rate of the Q235 steel in the presence of EPS and MF.

Because it has been previously reported that MF is liable to influence the orientation of the membrane protein crystals [43] and the thickness of protein solutions as well [44], this synergistic acceleration between MF and EPS can be related to the possibility that the adequate intensity of MF alters the orientation of the EPS, and the newly ordered arrangement of the EPS provides the respective channels for the diffusion of chloride ions and oxygen, as shown in Fig. 10. Thus, the corrosion of Q235 steel in a NaCl solution was significantly accelerated in the presence of both MF and EPS.

Fig. 9 SEM images of coupons under different corrosion conditions after 8 days of incubation in 3 wt% NaCl solution containing EPS in the absence **a, c** and presence **b, d** of MF at low **a, b** and high **c, d** magnification

Fig. 10 Schematic diagram of EPS steel corrosion in EPS-containing testing solution in the absence **a** and presence **b** of MF

4 Conclusion

A 150 mT MF could accelerate steel corrosion in a 3 wt% NaCl solution containing SRB by 202%. Pitting corrosion was the major corrosion type of steel in this work. The analysis results of polarization curves indicated that MF influenced the diffusion of dissolved oxygen and chloride ions as well as the reduction process of oxygen. Both the adsorbed EPS on the steel surface and the direct interaction between EPS and iron ions were confirmed by ATR-IR measurements. Electrochemical analysis results indicated that either EPS or MF inhibited the corrosion of Q235 carbon steel, but the combination of MF and EPS significantly accelerated the corrosion of Q235 carbon steel.

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