



Corrosion Behavior of API 5L X80 Steel in the Produced Water of Onshore Oil Recovery Facilities

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High-strength low-alloy steel is used in pipelines, which transport oil/gas and also produced water in the process of the secondary recovery of oil. This paper investigates the corrosion of X80 steel after immersion in static systems (biotic and abiotic). The corrosion rate, the corrosion morphology and the hardness of the coupons were investigated. The corrosion products were determined by SEM/EDS and XRD. The corrosion rates of the coupons in the biotic system were moderate and had an aggressive pit morphology, while those of the coupons in the abiotic system were low with less aggressive pit. The hardness of the coupons after exposure to the systems of produced water was not changed compared with that of the control coupons. The corrosion products formed in the biotic system were Fe(OH)₃, Fe(OH)₂, FeOOH, Fe₃O₄, FeS₂, FeCl₃, and in the abiotic system, NaCl, CaCl₂ and SrSO₄. BaSO₄ and CaCO₃ were identified in both systems.

Keywords: API 5L X80, biocorrosion, produced water, SEM/EDS, XRD.

1. Introduction

The development of high strength steel pipelines is of great economic importance to the oil industry. The technology of these pipelines has allowed for greater transmission of oil and gas due to increased internal pressure while decreasing the wall thickness of the pipe¹. API 5L X80 steel has excellent mechanical properties such as high strength, good toughness, good fatigue resistance and excellent corrosion resistance²⁻⁴. During the process of secondary oil recovery a large amount of produced water is generated which contains dissolved salts, inorganic and organic constituents, solids, oil, dissolved gases (CO₂ e H₂S) and microorganisms^{5,6}. The activity of microorganisms can change the nature of the aqueous environment which leads to hydrocarbon being degraded, clogging, souring and microbiologically-induced corrosion (MIC), and more specifically the localized corrosion of pipeline steel⁷⁻⁹. Sulfate reducing bacteria (SRB) are the bacteria most investigated in causing MIC in the oil industry¹⁰⁻¹². However there are other groups of bacteria that are prejudicial to pipelines such as iron-reducing and oxidizing bacteria, CO₂ reducing bacteria and manganese oxidizing bacteria^{1,7}. In the literature there are very few reports about the corrosion behavior of API 5L X80 steel in produced water with mixed cultures under static conditions. The exposure conditions are of great importance for characterizing corrosion products formed on

the surface of steel. The corrosion process, in some cases, causes the formation of corrosion products that increase the hardness on the steel's surface¹³. Recent studies in the oil and gas industry show the influence of microorganisms that cause corrosion^{8,14-16}. This paper sets out to investigate the corrosion of high-strength, low-alloy (API 5L X80) steel in produced water systems with and without microorganisms for 60 days. The material was characterized by its chemical composition and metallographic analyses. In this study, the microbiological composition of produced water was quantitatively characterized. The corrosion rate and the morphology of the corroded surface were determined by the weight loss technique and scanning electron microscopic images, respectively. The hardness of the coupons after immersion in the systems was investigated using the Vickers hardness test. The corrosion products were characterized by scanning electron microscopy coupled with energy dispersive spectroscopy (SEM/EDS) and X-ray diffraction (XRD).

2. Experimental Procedures

2.1 Material

The coupons were cut from API 5L X80 pipeline steel, which had a chemical composition (wt.%) of 0.08 C, 0.3 Si, 1.82 Mn, 0.009 P, 0.001 S, 0.17 Cr, 0.01 Ni, 0.20 Mo, 0.01 Cu, 0.037 Al, 0.024 V, 0.003 W, 0.021 Ti, 0.081 Nb and balance Fe. The size of the coupons used was 30 x 10 x 5

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mm. The surface of the coupons was abraded with sandpapers (220-1200 mesh) and polished with diamond paste (3 and 1 μm). After polishing, the coupons were cleaned in acetone and alcohol, and air dried. The characterization of the microstructure of coupons was obtained after etching them with 2% nital reagent for 10 seconds, followed by analysis in an inverted Axio Observer-Z1 Zeiss optical microscope. Figure 1 shows there was a polygonal ferrite microstructure with iron carbides at the grain boundaries.

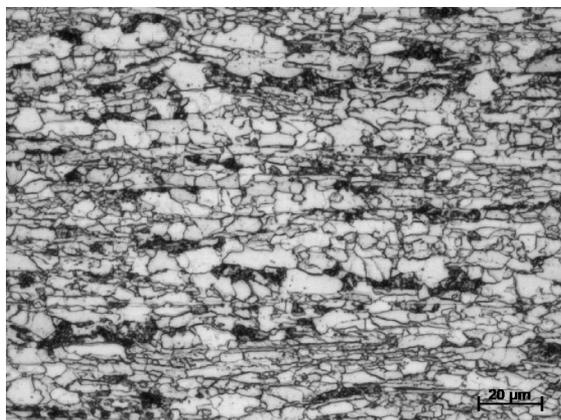


Figure 1. Microstructure of API 5L X80 steel (magnification 500x).

2.2 Water sample

Oilfield produced water was collected from a Brazilian oil production plant. The biotic system consisted of oilfield-produced water samples which were microbiologically characterized. The Most Probable Number (MPN) method was used to quantify the following groups of planktonic microorganisms: total anaerobic, acid-producing aerobic and anaerobic, and sulfate-reducing bacteria (SRB)¹⁷. Total aerobic and precipitating iron bacteria were quantified by the Colony Forming Unit (CFU) method¹⁸. The abiotic system (without microorganisms) consisted of the produced water samples from the oilfield which were filtered through membranes with 0.22 μm pores. After filtration, 1 mg/L sodium hypochlorite was added daily to remove microbial contaminants. Both static immersion systems were evaluated for 60 days at room temperature.

2.3 Cultivation of microorganisms

The group of planktonic microorganisms was quantified by inoculating 1 mL of the produced water in the reducing solutions, followed by reinoculation in different culture medium. The composition in (g/L) of the total anaerobic bacteria culture medium, which was prepared under nitrogen purges, was as follows: 30.0 NaCl and 30.0 thioglycolate (pH 7.0). The composition in (g/L) of the acid-producing aerobic and anaerobic bacteria culture medium was: 30.0 NaCl, 10.0 sucrose, 10.0 tryptone, 1.0 beef extract and

0.018 phenol red (pH 7.2). The culture medium used for acid-producing anaerobic bacteria was purged with nitrogen. The composition in (g/L) of the SRB culture medium prepared under nitrogen purges was as follows: 30.0 NaCl, 0.5 KH_2PO_4 , 1.0 NH_4Cl , 1.0 Na_2SO_4 , 0.67 $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 1.68 $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, 7.0 mL sodium lactate (50%), 1.0 yeast extract, 0.1 ascorbic acid, 1.9 agar-agar, 4.0 mL resazurin (0.025%) and 0.5 $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ (pH 7.6)¹⁹. The composition in (g/L) of the total aerobic bacteria culture medium was: 30.0 NaCl and 22.5 plate count agar (PCA) medium (pH 7.0). The composition in (g/L) of the precipitating iron bacteria culture medium was: 30.0 NaCl, 0.5 $(\text{NH}_4)_2\text{SO}_4$, 0.2 $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 0.5 $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 0.5 NaNO_3 , 10.0 ferric ammonium citrate and 0.5 K_2HPO_4 (pH 7.0)²⁰. The reducing solution contained the following constituents in (g/L): 30.0 NaCl, 0.124 sodium thioglycolate, 0.1 ascorbic acid and 4.0 mL resazurin (0.025%) (pH 7.6). All components were per liter of distilled water. All groups of microorganisms were incubated at 35°C. All culture media and solutions were sterilized in autoclave at 1 atm and 121°C for 20 minutes.

2.4 Weight loss, corrosion rate and analysis of the morphology of corrosion

The experiments were conducted on open glass static test cell. The coupons were suspended by nylon threads and totally immersed in the produced water. Coupons were weighed before being placed within the biotic and abiotic systems. The coupons were removed from the systems after 15, 30, 45 and 60 days, and the biofilms and corrosion products were removed from the surface of coupons with a non-metallic rod. The coupons were immersed in Clark solution, followed by ultrasonic cleaning in distilled water. Afterwards they were cleaned in isopropyl alcohol and acetone, dried and weighed. Subsequently, the corrosion rate was evaluated using the ASTM G1-03 standard²¹. The corrosion rate of the coupons was determined as shown below: Corrosion rate (mm/y): $\left(\frac{K \times W}{A \times T \times D}\right)$. Where K = a constant = 8.76×10^4 , W = weight loss (g), A = area (cm^2), T = time of exposure (h) and D = density of carbon steel (7.86 g/cm^3). The NACE RP 0775 Standard was used to evaluate the corrosivity of the coupons²². After establishing the corrosion rate, the corrosion morphology on the coupons was investigated using a scanning electron microscope (SEM) TM 3000 Hitachi.

2.5 Hardness test

The Vickers hardness (VH) test was carried out on the coupons before (control) and after immersion in the biotic and abiotic systems. The mean value of 6 readings of the indentation length was taken along the direction of the thickness. The 10 kg load applied was maintained for 15 s for each reading. Hardness tests were conducted on a

durometer DuraScan 70 EMCO TEST using a pyramidal diamond indenter, as per the ASTM E384-11 Standard²³.

2.6 Characterization of corrosion products

After the immersion test, the coupons were removed from the systems, and dried in a desiccator. Subsequently, the surface of the coupon was analyzed. The elements and chemical compounds of corrosion products on the coupon were analyzed by SEM coupled with energy dispersive spectroscopy (EDS) and X-ray diffraction (XRD), respectively. SEM analysis was carried out using a TM 3000 Hitachi microscope operating at 15 kV. EDS analysis was semi-quantitative. XRD analysis was carried out using a 7000 Maxima Shimadzu X-ray diffractometer operating with a Cu source ($\lambda = 0.154060 \text{ \AA}$), at the voltage of 40 kV, current of 30 mA and scan speed of $1^\circ/\text{min}$.

3. Results and Discussion

3.1 Quantification of planktonic microorganisms

Table 1 presents the cell concentration of planktonic microorganisms in the produced water sample. The total aerobic bacteria were in higher concentrations in the produced water sample. Aerobic bacteria consume dissolved oxygen in the fluid which provides the anaerobic environment with the propitious growth of anaerobic bacteria^{15,24}. SRB, total anaerobic, acid-producing aerobic and anaerobic bacteria were also present in the produced water. SRB are the main causes of microbiologically-induced corrosion in the operating systems of the oil industry^{12,25}. Iron-precipitating bacteria were not detected in the produced water, probably due to the low cell concentration or because the counting method was limited.

3.2 Corrosion test analysis

The corrosion rate for weight loss of API 5L X80 steel coupons after 15, 30, 45 and 60 days of immersion into biotic and abiotic systems is shown in Figure 2.

A significant difference between the corrosion rates was observed. The biotic system showed a higher corrosion rate, between 0.029 and 0.057 mm/year, with moderate corrosivity. In the abiotic system, the corrosion rate varied between 0.002 and 0.010 mm/year, this being considered low corrosivity. The addition of sodium hypochlorite biocide did not increase the corrosion rate of the coupons exposed to the abiotic system. It is likely that metabolic products of

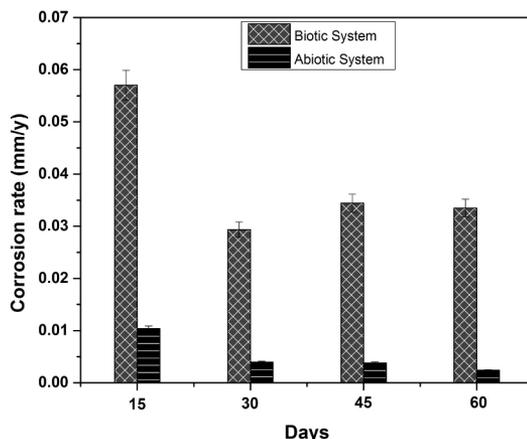


Figure 2. Corrosion rate by weight loss of API 5L X80 steel coupons.

microorganisms and production of exopolymeric substances (EPS) contributed to the increase in the corrosion rate, and thus promoted biodegradation of the coupons in the biotic system²⁶. These microbial metabolites can be organic acids, hydrogen sulfide or other sulfur species¹². The deterioration of metal surfaces because of bacteria occurs due to the initial formation and subsequent adhesion of biofilms, which as a result of physical and chemical reactions modify this interface²⁷. Major changes in this interface are observed after biofilm forms, such as changes in pH, the redox potential, the types of ions in electrochemical corrosion and even the variables used to evaluate the corrosion rate²⁸. Cetin and Aksu²⁹ analyzed after 30 days the corrosion rates by weight loss of the low alloy steel in culture medium with and without *Desulfotomaculum* sp. The researchers observed that the corrosion rate of the biotic system (0.000012 mm/year) was four times higher than the abiotic system (0.000002 mm/year). AlAbbas et al.¹ observed higher values of corrosion rates of API 5L X80 steel in the biotic system (values above 1.5234 mm/year) after 200 hours, while the abiotic system presented a rate of 0.25391 mm/year throughout the test. Mishra⁷ evaluated the corrosion rates of API 5L X80 steel in produced water with iron reducing bacteria. The iron-reducing bacteria consortium inhibited the corrosion of the steel in the biotic system, reflected by low values of corrosion rate (0.3022 mm/year). In the abiotic system, the corrosion rates were higher with a maximum value of 5.0782 mm/year after 200 hours of testing. Oliveira et al.³⁰ investigated the corrosion rate by weight loss of carbon steel SAE 1010 after 28 days of immersion in seawater with and without sodium hypochlorite. The results showed high corrosion

Table 1. Planktonic microorganisms in produced water.

Group of microorganisms	Total aerobic	Total anaerobic	Acid-producing aerobic	Acid-producing anaerobic	Iron-precipitating bacteria	SRB
Cell concentration	1.2×10^4 CFU/mL	1.1×10^3 MPN/mL	7.0×10 MPN/mL	2.5×10^2 MPN/mL	Undetected	4.5×10^3 MPN/mL

rate in the seawater system (control), while in the seawater with sodium hypochlorite system there was a reduction of the corrosion rate and the existing microorganisms. Figure 3 displays the SEM images of the coupons after cleaning and removing corrosion products.

Micrographs of the coupons immersed in both biotic and abiotic systems (Figure 3) exhibited localized corrosion, probably by pitting. However, the images of the steel immersed in the biotic system (Figure 3 (a-d)) showed more severe localized corrosion (indicated by white arrows) as compared to the abiotic system (Figure 3 (e-h)). After biofilm has formed on the metal surface because of the microbial consortia, the biofilm produces high concentrations of sulfide and other compounds that potentially prompt an increase in the corrosion process. Sulfate-reducing bacteria have an influence on the corrosion of ferrous alloys, and generally promote pitting corrosion. This is a more severe type of corrosion than uniform corrosion due to its rapid penetration into the structure of the material and may cause failures^{1,27}. The morphologies shown in Figure 3 correspond to corrosion rates, by weight loss of the system as shown in Figure 4. Wu et al.³¹ analyzed an X80 steel surface using SEM, after removing the films formed on systems containing SRB under yield stress. Small pittings were found in the micrographs of the system with SRB, when compared to those of the sterile system, both without load. Perpendicular micro-cracks were identified in the micrographs of SRB and sterile systems, both with load. In the SRB system, the number, density, width and length of the cracks were higher than in the sterile system, thus highlighting the synergistic effect between bacteria and yield stress.

3.3 Measurement of hardness

Hardness tests were performed on API 5L X80 steel coupons before immersion and after 8, 10, 15, 30 and 60 days of immersion in biotic and abiotic systems, and the test results are presented in Figure 4. Figure 5 shows the microstructure of the steel after the immersion test.

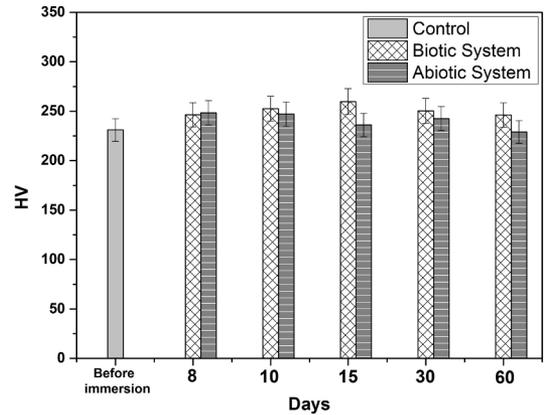


Figure 4. Hardness measurement of the API 5L X80 steel coupons.

Similar micrographs were observed in the other periods investigated. No significant difference in hardness values was found in the systems studied. The consortium of microorganisms and their aggressive metabolites did not cause changes in the microstructure (Figure 5), and consequently did not change the hardness of the API 5L X80 steel coupons (Figure 4), although an increase was observed in corrosion rates and localized corrosion on the surface of steel after immersion tests with microorganisms. Studies show that changes in the microstructure and hardness of API 5L X80 steel usually occur after heat treatment or welding processes^{32,33}. According to Walsh et al.³⁴ the weld regions have microstructural heterogeneities that are attractive to micro-organisms, which are responsible for causing the microbiologically-induced corrosion.

3.4 Analysis of corrosion products

Figure 6 shows the SEM-EDS analysis of the corrosion products formed on the surface of API 5L X80 steel coupons after 60 days. The areas analyzed in the SEM images by EDS were marked with black circles.

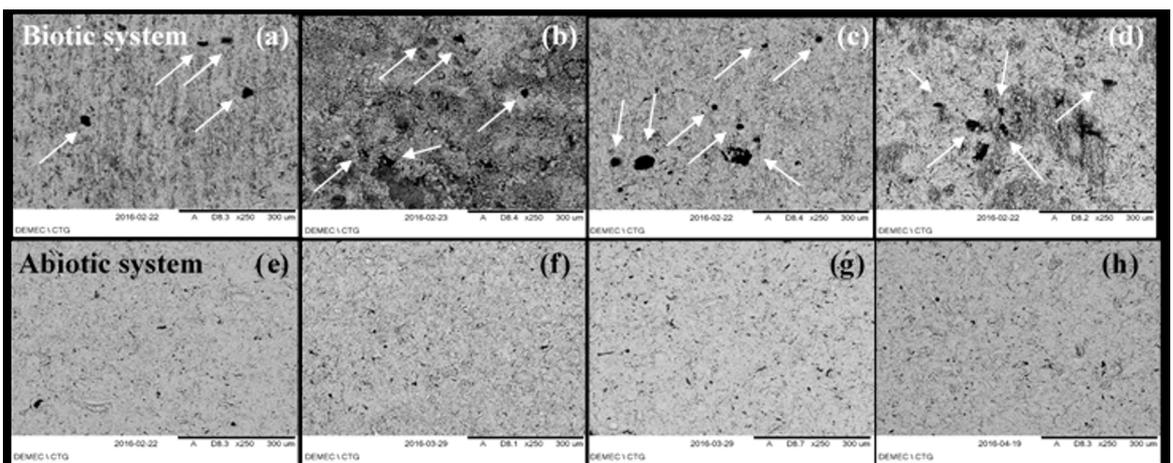


Figure 3. SEM analysis of cleaned coupon surface after: (a, e) 15, (b, f) 30, (c, g) 45 and (d, h) 60 days of exposure in biotic and abiotic systems.

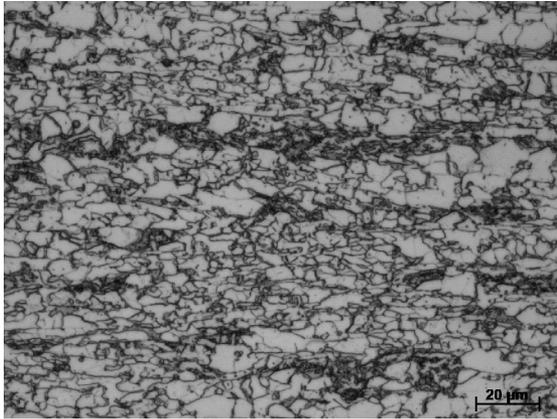
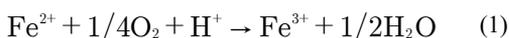


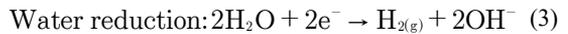
Figure 5. Microstructure of the API 5L X80 steel coupons after 10 days of immersion in the biotic system (magnification 500X).

The micrographs in Figure 6 (a and b) show corrosion products which are dense and porous with different morphologies on the coupon surface after immersion in the biotic system. The EDS analysis in Figure 6 (a1 and b1) suggests that the main corrosion products consist of the elements: C, O, Fe, S, Ba, Si, Cl and Ca. The micrographs in Figure 6 (c and d) show salt crystals deposit on the surface of coupons after immersion in the abiotic system. The EDS analysis in Figure 6 (c1 and d1) suggests that the main corrosion products consist of the elements: O, Cl, Na, Ca, Mg and Si. The produced water typically has a high concentration of dissolved salts that account for the increase of corrosion in pipes. Chloride anion (Cl^-) is the salt that is in the greatest quantity. However, sulfate (SO_4^{2-}), carbonate (CO_3^{2-}) and bicarbonate (HCO_3^-) are found in small quantities in this produced water. The cations most commonly found are sodium (Na^+) and potassium (K^+), calcium (Ca^{2+}) and magnesium (Mg^{2+})³⁵⁻³⁷. Figure 7 presents the analysis by X-ray diffraction of the corrosion products formed on the surface of API 5L X80 steel coupons from the biotic and abiotic systems.

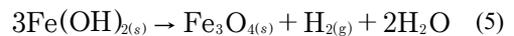
The crystalline phases identified in peaks belonging to the corrosion products which had formed on the coupon immersed in the biotic system were the following (Figure 7 (a)): cubic ferric hydroxide ($\text{Fe}(\text{OH})_3$), hexagonal ferrous hydroxide ($\text{Fe}(\text{OH})_2$), orthorhombic FeOOH , cubic face-centered (CFC) (Fe_3O_4) magnetite, orthorhombic barium sulfate (BaSO_4), orthorhombic calcium carbonate (CaCO_3), cubic pyrite (FeS_2), and hexagonal ferric chloride (FeCl_3). In aerobic environments, iron-precipitating bacteria oxidize ferrous ions to ferric ions, and these ferric ions precipitate as ferric hydroxide due to the following reactions^{15,38}:



The formation of ferrous hydroxide occurs from the iron oxidation reaction, as shown in Equations 3 and 4³⁹:



According to Liu et al.¹⁵ ferrous hydroxide can be oxidized to form compounds such as FeOOH , Fe_2O_3 and Fe_3O_4 . FeOOH is the hydrated form of Fe_2O_3 which may have sub-forms: goethite (α - FeOOH), akaganeite (β - FeOOH) and lepidocrocite (γ - FeOOH)⁴⁰. Magnetite is a ferric oxide derived from the transformation of ferrous hydroxide in the absence of oxygen, as a result of the following reaction^{39,40}:



The barium sulfate and calcium carbonate are precipitated and cause fouling in oil fields because of their low solubility in water. Generally, barium sulfate is formed when the high concentration of sulfate ions in injection water (e.g. seawater) mixes with barium ions from formation water (e.g. from an oil well) as shown in the following reaction^{41,42}:



When barium sulfate is associated with sulfate-reducing bacteria, this further enhances the corrosion process in secondary oil recovery systems⁴³. Calcium carbonate precipitates after increasing the temperature and/or a reduction in pressure as shown in the following reaction⁴¹:



Pyrite is the most stable form of iron sulfide, and is derived from biological reactions caused by sulfate-reducing bacteria⁴⁴. This formation of iron sulfide, depending on the case, may increase the corrosion or even protect the metal¹². Iron chloride can be formed by iron-precipitating bacteria in media with a high chloride concentration⁴⁴. The crystalline phases identified in peaks belonging to the corrosion products formed on the coupon immersed in abiotic system were the following (Figure 5 (b)): orthorhombic barium sulfate, orthorhombic and monoclinic strontium sulfate (SrSO_4), orthorhombic, rhombohedral and hexagonal calcium carbonate (CaCO_3), CFC sodium chloride (NaCl), orthorhombic calcium chloride (CaCl_2) and cubic body-centered (CCC) α - Fe . Strontium sulfate, barium sulfate and calcium carbonate are low water solubility salts, which cause fouling in pipes in the oil industry⁴¹. Produced water contains sodium and calcium chlorides as dissolved salts. According to Neff et al.³⁶ the concentration of dissolved salts in produced water

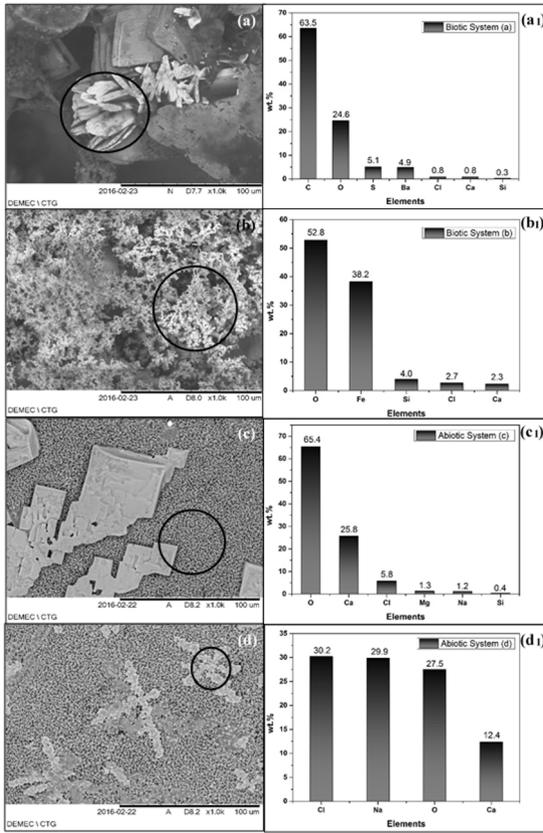


Figure 6. SEM-EDS of the API 5L X80 steel coupons after immersion test: (a, a₁, b, b₁) biotic system and (c, c₁, d, d₁) abiotic system.

may be greater than in seawater. According to Munirasu et al.⁴⁵ sodium and calcium ions are among the cations most commonly found in produced water. The α -Fe identified in the XRD analysis is the naked coupon surface, on which there was no salt deposit. Chemical elements, suggested in the analysis by EDS (Figure 6), were found in the crystalline phases identified in the XRD patterns of biotic and abiotic systems (Figure 7). Liu et al.³⁵ used EDS and XRD to identify the chemical elements and corrosion products formed on

Q235 steel pipes in produced water. FeOOH was identified at the bottom of the pipe, and the compounds CaCO_3 and $\text{Mg}_6\text{Al}_2\text{CO}_3\cdot(\text{OH})_{16}\cdot 4\text{H}_2\text{O}$ in the corrosion layers. Xu et al.⁴⁶ used XRD to analyse the corrosion products formed on the surface of low-alloy steel coupons after 365 days. The researchers indicated the presence of Fe_3O_4 on both coupon positions in the system (upward and downward surfaces). In addition to γ -FeOOH on the coupon with an upward surface, CaCO_3 and α -FeOOH were on the coupon with a downward surface. Shi et al.⁴⁷ used XRD, SEM and transmission electron microscopy (TEM) to find the corrosion products formed on API X60 steel coupons in acidic solutions containing H_2S and NaCl 5%. The researchers varied the temperature (60 - 150 °C), the pressure of H_2S (0.15 to 5 MPa) and the exposure time (72 - 216 h). The corrosion products found were: tetragonal crystal mackinawite, cubic FeS, (intermediate between mackinawite and pyrrhotite) troilite, hexagonal crystal pyrrhotite and cubic pyrite. Yaro et al.¹³ used XRD to evaluate the corrosion products formed on API X65 steel coupons after 3 hours of exposure in produced water and CO_2 , with and without acetic acid. X-ray diffractogram showed cementite (Fe_3C) and siderite (FeCO_3) in the coupons in contact with acetic acid, and siderite in the coupons without contact with the acetic acid.

4. Conclusions

The planktonic microorganisms present in produced water (from the biotic system) increased corrosion in API 5L X80 steel. The corrosion rate and pitting corrosion morphology is more severe in the steel coupons of the biotic system (moderate corrosivity) compared with those of the abiotic system (low corrosivity). In the biotic system, the microorganisms released aggressive metabolites that increased deterioration of the metal surface. The hardness of steel did not change significantly after exposure to microorganisms due to the test temperature and the formation of corrosion products that did not alter the mechanical properties of the

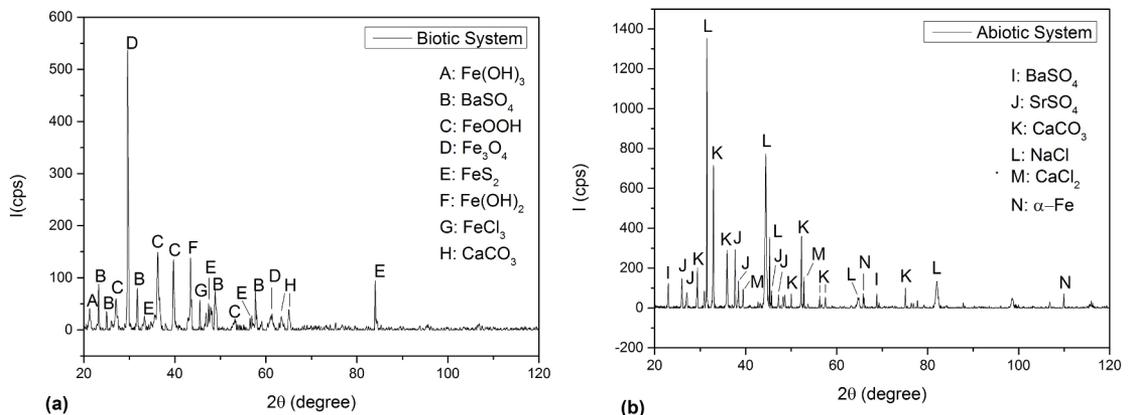


Figure 7. X-ray diffractogram of corrosion products formed on API 5L X80 steel coupons immersed in (a) biotic and (b) abiotic systems.

steel coupons. XRD revealed the formation of iron oxides and hydroxides, iron sulfide, iron chloride, barium sulfate and calcium carbonate on the steel surface immersed in the biotic system. Chlorides of sodium and calcium, strontium sulfate, barium sulfate, and calcium carbonate were deposited on the steel immersed in the abiotic system. Further studies should be conducted to investigate the behavior of this steel under dynamic flow conditions and temperature variation to test how these affect the use of this steel in the petroleum industry.

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