

Corrosion behavior of carbon steel API 5L X65 exposed to seawater

Vitor Silva Liduino, Márcia Teresa Soares Lutterbach, Eliana Flávia Camporese Sérvulo

Abstract— Steel corrosion continues to be a topic of interest because of potential implications for the safety, economic efficiency and viability of structures. Particularly, natural deterioration of metal structure exposed to marine environmental, such as oil and gas pipelines, needs special attention. The aim of this work was to investigate the corrosion behavior of carbon steel exposed to polluted seawater from Guanabara Bay, localized in Rio de Janeiro, Brazil. Corrosion tests were performed using working electrodes of API 5L X65 steel immersed in static reactor filled with 800 mL of fresh seawater for 15 days. Concurrently, control tests with autoclaved seawater were carried out under same conditions. At the end of tests, the electrodes exposed to fresh seawater showed a visibly greater degradation than those in sterile condition. Uniform and pitting corrosion were observed on all coupons exposed to both tests; however, the number of pits was more severe and the corrosion rate was nearly twice as much in working electrodes immersed in fresh seawater. This study evidences that native microorganisms of seawater close industrial zone are able to colonize steel surfaces within a short time and intensify dangerously the corrosion process.

Index Terms— Microbiologically Influenced Corrosion, Pitting, Carbon steel X65, Seawater.

I. INTRODUCTION

Corrosion is a significant potential threat to new and existing infrastructure. In several industrial systems, this type of deterioration is the main cause of the rise in economic costs, due to maintenance, repair and protection of metal structures as well as to the occurrence of failures that can result in serious environmental accidents [1]. In the United States of America alone costs related to all forms of corrosion have been estimated at around 4% of GNP per annum [2]. This problem is especially critical in the oil industry because of the large amount of metallic materials in the exploration, transportation and storage of oil and its derivatives.

The American Petroleum Institute (API) designates different grades of carbon steel (X52, X65, X70, X80) to pipelines, tanks and oil rig construction because of its good corrosion resistance, high strength and toughness to withstand high operating pressures, field weldability and low cost [3]. These properties can be achieved by varying chemical composition and microstructure of steels. Example of API steel application is the Bolivia-Brazil pipeline, built by PETROBRAS S.A., travels through five Brazilian States, for over than 2500 km in Brazilian territory, which presents

different characteristics in terms of topography, vegetation, geology and geotechnical conditions. It should also consider that inside pipelines, petroleum and liquid petroleum products contain large numbers of various types of microorganisms that either directly or indirectly enhance corrosion [4].

Microorganisms are widely distributed in many environments, both natural and industrial due to their ability to grow using different nutritional sources and often under different and not favorable environmental conditions. In these environments, microorganisms often migrate from the planktonic lifestyle (free cells in suspension) to the sessile lifestyle (cells attached to solid surfaces) due to increased availability of nutrients. As a result, microbial communities are formed degrees of complexity, comprising dynamic biostructures, called biofilms [5].

Bacterial attachment and subsequent biofilm formation on material surfaces can influence the deterioration of those surfaces by changing the physical or chemical properties [6]. In the biofilm may co-exist several microbial species, whose activity can result in the production of enzymes, EPS, organic and inorganic acids, which in turn may affect the cathodic and/or anodic reactions on metals, increasing the speed of electrochemical processes in biofilm/metal interface. Changes in the corrosion behaviour of materials in the presence of microorganisms is a process known as microbiologically influenced corrosion (MIC) [1]. MIC can cause significant economic losses and affects various industries including maritime, oil and gas, power generation, water distribution and nuclear waste [5]. It is important to highlight that in a marine environmental, besides presence of microorganisms, steel corrosion depends on many factors including seawater chemistry (pH, dissolved oxygen content, salinity, temperature), galvanic interactions and material hydrophobicity [7].

Lots of studies have reported correlation between corrosion of carbon steel and marine environmental [8],[9], nevertheless none of them used a seawater collected from an industrial zone neither performed control assays employing autoclaved seawater. Therefore, this study investigated the influence of industrially polluted seawater and its indigenous microorganisms on corrosion behaviour of carbon steel API 5L grade X65.

II. MATERIALS AND METHODS

A. Working electrode

Carbon steel API 5L X65 plates were cut with dimensions of 30 mm x 20 mm x 2 mm using an automatic cutting machine. Then, all electrodes were polished sequentially up to 1200 grit silicon carbide paper to obtain a smooth surface, cleaned by detergent solution and kept in sterile Petri dishes and remained at room temperature ($20 \pm 2^\circ\text{C}$) until the time of testing.

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Chemical composition analysis of the steel was conducted by Centro de Tecnologia Mineral (CETEM) and the microstructure of carbon steel X65 was examined at typical cross-sections by Serviço Nacional de Aprendizagem Industrial (SENAI).

B. Sampling of seawater

The tests were performed using polluted seawater collected from the Guanabara Bay (Rio de Janeiro – Brazil), which is near petrochemical industries and contains a high number of microorganisms and organic matter. Sampling was realized 2 days before starting the tests and preserved in refrigerator ($4 \pm 1^\circ\text{C}$). For control assays, three liters of seawater were autoclaved subjecting them to high-pressure saturated steam at 121°C for 20 minutes.

C. Experimental design

The working electrodes were placed in glass reactors having a capacity of 1 liter, containing 800 mL of polluted seawater. The electrodes were fixed to the lid by a nylon thread and ports in the flask lid were stuffed with hydrophobic cotton to prevent contamination of the tests performed, mainly tests with autoclaved water. The glass reactors were kept on a stir plate under constant stirring by magnetic rod, just to keep the planktonic cells in suspension and thus promote the colonization of electrode surfaces.

Two groups of reactors were set up: (1) reactor filled with fresh seawater, in triplicate, and (2) reactor filled with autoclaved seawater, also in triplicate. In total, six reactors (three biotics and three abiotics) were performed; each one contained 3 working electrodes. All reactors were saved in a cooled room at $20 \pm 2^\circ\text{C}$ during 15 days of testing. Before and after each assay, the reactors were disinfected by immersion in a 5g/L solution of sodium metabisulphite for 24 h and then washed five times with sterile distilled water for complete removal of the disinfectant. Figure 1 illustrates the corrosion reactors performed, biotic reactor with fresh seawater and abiotic reactor with autoclaved seawater.

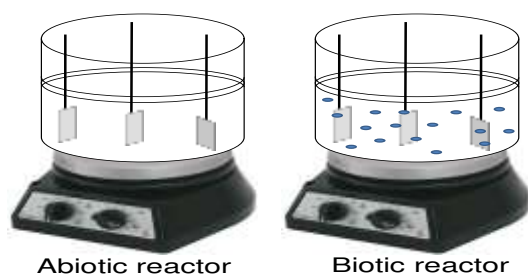


Fig. 1 Illustration of reactors employed to corrosion assays.

Open circuit potential of the working electrodes was measured with the aid of a multimeter benchtop, while saturated calomel electrode was used as reference electrode. Measurements were realized in each 24-hour interval for the entire testing period, in order to observe possible variations relevant to the formation of biofilms and abiotic deposits (metal oxides). The behavior of the measured potential represents the average of determinations acquired for 3 electrodes.

D. Microbial enumeration

Quantitative determinations of aerobic heterotrophic bacteria (AHB), iron-bacteria (IB) and sulphate reducing bacteria (SRB) populations were made by cultivation in appropriate culture media applying the most probable number technique (MPN) [10], at the beginning and the end of assays. AHB, IB and SRB of planktonic lifestyle were cultivated in nutrient broth (5.0 g meat peptone, 3.0 g yeast extract, 10.0 g d-glucose, 35.0 g NaCl [g/L]), ferric ammonium citrate medium (0.5 g $(\text{NH}_4)_2\text{SO}_4$, 0.5 g NaNO_3 , 0.5 g K_2HPO_4 , 0.5 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 0.13 g $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$, 10.0 g ferric ammonium citrate, 35.0 g NaCl [g/L]) and Postgate E semi-solid medium (0.5 g KH_2PO_4 , 1.0 g NH_4Cl , 1.0 g Na_2SO_4 , 0.67 g $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, 1.83 g $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, 7.0 mL sodium lactate (50 % m/v), 2.0 g sodium acetate, 1.0 g yeast extract, 0.1 g ascorbic acid, 0.5 g $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 1.9 g agar, 4.0 mL resazurin, 35.0 g NaCl [g/L]), respectively. The incubation for the bacterial growth was assessed according to the requirements of each population.

E. Corrosion rate

Corrosion rate was determined according ASTM standard G1-03. The cleaned electrodes were weighed before carrying out the assays and then immersed in the seawater. At the end of assays, all electrodes were picked up from the reactors, washed under running water and corrosion products were removed by immersion in Clarke's solution (1000 ml of hydrochloric acid, 20 g of antimony trioxide (Sb_2O_3) and 50 g of stannous chloride (SnCl_2)) for 40 s, rinsed with distilled water, cleaned with acetone, dried in hot air and finally weighed to mass loss measurements using a mass balance with 0.0001g accuracy. The mean of weight loss values was calculated considering three working electrodes and corrosion rates were determined using the equation below:

$$\text{Corrosion rate (mpy)} = 87.6 \times \frac{W}{\rho A t}$$

where, 'W' is the weight loss (g), ' ρ ' the density of the carbon steel (g cm³), 'A' the area of specimen (cm²) and 't' the time of exposure (h).

F. Surface analysis

After complete corrosion products removal, whole working electrode surface was scanned by electron microscope (SEM) to take an overview of surface features. The number of pits was counted on each electrode for the determination of pitting density.

III. RESULTS AND DISCUSSION

A. Electrode characteristics

Table 1 shows the chemical composition of the steel used in this study. According to American Petroleum Institute, steels are classified due their chemical composition, mechanical strength and application. This result is consistent with material classified as low carbon steel. Moreover, low-alloy steels may contain small amounts of chromium, niobium, molybdenum and aluminum, among other metals in amounts lower than 0.1%

Table 1 Chemical composition of carbon steel API 5L X65 used (weight %)

C	Mn	P	Si	Cr	V	Mo	Al	Ca	Nb	Mg	Cu	Fe
0	1.4	0.5	1	0.1	0.1	<0.01	0	0.1	0	0.1	0	Balance

The steel microstructure was examined by optical microscopy at 200x of magnification. Figure 2 gives the micrograph of

X65 in the longitudinal orientation taken from the quarter thickness position and shows coarse-grained banded ferrite

and pearlite microstructures. Lighter-colored grains are ferrite and dark areas are pearlite.

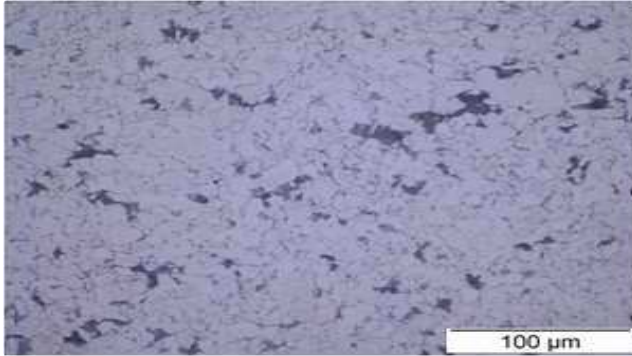


Fig. 2 Microstructure of carbon steel API X65.

This steel was produced at high load capacity rolling mills and therefore may have experienced multiple recrystallisation stages and then significant deformation below the recrystallisation stop temperature and has been subjected to accelerated cooling schedules. These factors result in a refined grain structure. The steel contains different bainitic and ferrite structures, which are also consistent for accelerated cooling of steels with low carbon content [11].

A. Seawater characteristics

The characteristics of the fluids have direct and / or indirect influence on the corrosive attack of the materials exposed to them. Therefore, for the implementation of efficient measures of prevention and corrosion control is necessary to know the material specifications and the working conditions, such as salinity, pH, dissolved oxygen, others. For instance, the salinity was about 32 ppt (32.640 mg/L); generally, salinity level in seawater is fairly constant at range 30 to 35 ppt [12]. Salinity is a strong contributor to electrical conductivity, in seawater the salt content (predominantly sodium (Na^+) and chloride (Cl^-) ions) causes rapidly increased conductivity promoting the development of corrosion [13]. Besides, seawater may contain a wide variety of microbial species from different groups, including bacteria, algae and fungi. In particular, evaluation of the sulfate reducing bacteria and iron bacteria concentrations in the fluid is fundamental to predict the prevention strategies to be adopted. They are considered the main microorganisms responsible for microbiologically influenced corrosion (MIC) [6].

Table 2 Main characteristics of the seawater collected

Parameters	Value
Salinity	32.640 mg/L
Conductivity	53.8 mS/cm
pH	7.6
Chemical oxygen demand	853 mg/L
Dissolved oxygen	7.9 mg/L
Aerobic heterotrophic bacteria	3.5×10^5 NMP/mL
Iron bacteria	7.0×10^2 NMP/mL
Sulfate reducing bacteria	1.5×10^2 NMP/mL

Table 2 shows the chemical, physicochemical and microbiological parameters of the seawater sample from Guanabara Bay.

Seawater analyzed showed low number of planktonic sulfate reducing bacteria (1.5×10^2 NMP/mL) and iron bacteria (7.0×10^2 NMP/mL), while aerobic heterotrophic bacteria count was moderate (3.5×10^5 NMP/mL). Corrosion

studies conducted with seawater collected at the same site and during different seasons had similar cell concentrations for the same bacterial groups [14],[15]. Several articles in the Literature have demonstrated that these groups of microorganisms are easily found in oil pipelines and fuel storage tanks, and they are a cause of serious damage to metallic structures [16],[17],[18]. The seawater from Guanabara Bay contains sources of carbon, sulfate and other elements required for microbial metabolic activity as well as the pH of 7.6 and dissolved oxygen value of 7.9 mg/L are ideals for the development of most bacteria. According to Jong and Parry [19], the optimal pH for SRB and IB growth is in the range of 7.0 to 7.6. Therefore, it can be suggested that the seawater used in the experimental design has corrosive characteristic, principally for MIC.

At the end of the tests, the seawater from biotic reactor showed a pH decreased to 6.8 probably due to production of some organic acids as microbial metabolites, while the pH of the autoclaved seawater from abiotic reactor had not changed significantly. Even so, the pH decrease was not enough to inhibit the bacterial growth associated to corrosion. The final microbiological counts showed that aerobic heterotrophic bacteria and iron bacteria increased 2 (up to 6.1×10^7 NMP/mL) and 3 (up to 8.4×10^4 NMP/mL) orders of magnitude, respectively. However, there was no difference in the abundance of sulphate reducing bacteria. These results indicate that the working conditions applied were satisfactory to ensure the microbial requirements, even without nutritional supplementation of the seawater.

B. Pitting investigation

Figure 3 shows the working electrode surfaces exposed to autoclaved seawater (Figure 3.A) and fresh seawater (Figure 3.B) for 15 days. After removal of corrosion products, localized corrosion can be observed especially in electrodes immersed in biotic reactor, where the overall pit density was estimated at around $3,8 \times 10^5$ pits/ m^2 (Figure 3.B). On the other hand, pitting was less frequent in electrodes immersed in abiotic reactor. In this case, the number of pits that could be seen was estimated at 2-3 in whole surface (Figure 3.A). These findings confirm that environments with microorganisms can result in harmful corrosive processes.

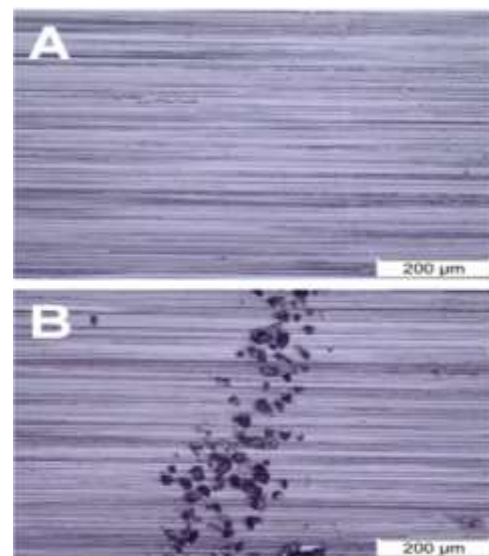


Fig. 3 SEM micrographs of carbon steel exposed to autoclaved seawater (A) and fresh seawater (B) after 15 days of assays.

A great number of investigations have demonstrated that chloride ions remarkably influence the corrosion of metal surfaces in saline environments, like marine areas [20],[21],[22]. Chloride ions, minor in radius, may be adsorbed or penetrate easily through the passive film even the oxide layer thus damaging metal integrity and accelerating the electrochemical reactions (anodic and cathodic). In contrast, the effects of chloride ions on the

Table 3 Corrosion rates of carbon steel X65

Condition	Value
Autoclaved seawater	0,0623 mpy
Fresh seawater	0,1048 mpy

corrosion kinetics are also controversial. Allam and collaborators [23], focusing on atmospheric situations, revealed that chloride ion only functioned during the corrosion initiation and failed to penetrate through the thick rust layer at later stages. Thus, the synergistic action of chloride ions and microorganisms (e.g. sulfate reducing bacteria and iron bacteria) may increase the susceptibility of the material to localized corrosion.

SEM of biofilm formed on electrode surface exposed to fresh seawater showed that rod-shaped cells were predominant, while in the control assay (autoclaved seawater) only corrosion products with cracks but no cells were observed on the electrode surface (Figure not shown). The rod-shaped bacteria found in environments with MIC occurrence are associated with some sulfate reducing bacteria belonging to the genera *Desulfotomaculum* and *Desulfovibrio*, as well as primary colonizing species such as those of the *Pseudomonas* genus [24],[25]. *Pseudomonas* species are known to produce exopolysaccharide (EPS) material and are usually isolated from environments where the biocorrosion phenomenon occurs [26],[27]. EPS favors cell adhesion to inert or non-inert surfaces, facilitating colonization by non-EPS producing successor species, and creating microenvironments with low concentration or absence of oxygen. These microenvironments allow the growth of the facultative aerobic bacteria and anaerobic bacteria (e. g. SRB) in the biofilm.

C. Electrochemical evaluation

The electrochemical experiment, consisting of open circuit potential measurements (E_{OC}), is given in Figure 4. On the first day, the open circuit potential of the carbon steel was measured after 15 minutes of stabilization in seawater.

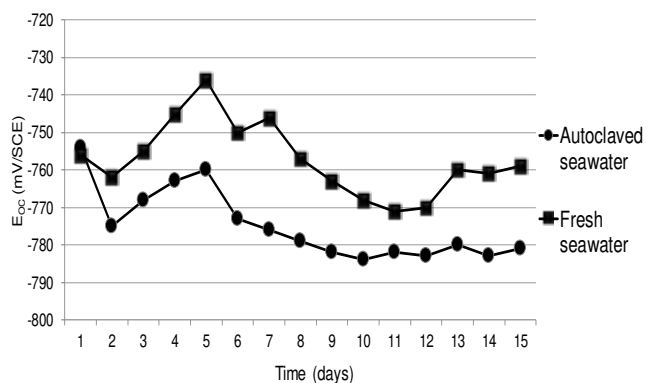


Fig. 4 Open-circuit potential (E_{OC}) monitoring.

It can be observed that there were significant changes in the E_{OC} values with the exposure time, independent of seawater employed. All potentials decreased in the first 24 hours of exposure. The decrease was even more pronounced for coupons immersed in autoclaved seawater, reaching values of -775 mV_{SCE}. The concentration of the chloride ion available in seawater may have caused this decrease. Then, the potentials increased until the fifth day of assay probably due to the corrosion products formation, which worked like a barrier on metal surface. From the sixth day onwards, all coupons showed an increasing tendency to oxidation until the end of the test. The presence of microorganisms in the fresh seawater shifted the E_{OC} values in the positive direction by approximately 15-30 mV, causing the so-called ennoblement of the steel due to the biofilm formation [28]. Therefore, the E_{OC} was affected by the microbial activity produced by the SRB, IB and EPS-producing bacteria which were adhered to the coupon surface to form a biofilm. However, as already shown in Figure 3B, the carbon steel surface was covered by many pits, which means that this biotic layer may not be considered as protective.

Table 3 shows the corrosion rates of the working electrodes exposed to different fluid for 15 days. The weight loss of steel electrodes immersed in fresh seawater was larger than that observed when electrodes were exposed to autoclaved seawater. Thus, the corrosion rate of steel X65 was nearly twice as much in presence of microorganisms. Paula and collaborators [29] also found a high corrosion rate (approximately 1.2 mpy) for carbon steel after 35 days of exposure in artificial seawater in the presence of mixed sulfate-reducing bacteria (SRB) culture isolated from the rust of a pipeline. It is highlighted that weight loss is not ordinarily recommended for use as a measure of the extent of pitting unless general corrosion is slight and pitting is fairly severe.

IV. CONCLUSION

The carbon steel API X65 showed quickly corrosion trend once exposed to autoclaved seawater. This fact was more dangerous when native microorganisms of seawater were free to adhere to steel surface, intensifying pitting corrosion. Therefore, we strongly recommend the use of antifouling coatings for the protection of carbon steel structures exposed to marine environments. The presented data showed that through the study on the bacteria growth combined with electrochemical and microscopy methods, it is possible to understand how microorganisms affect the corrosion process.

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