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Impact and evaluation of corrosion in the oil industry environment (H_2S) on HSLA X65 steel

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ABSTRACT

The oil and natural gas industry is important to guarantee energy resources; however, the concentrated H_2S environment generates an acidic environment. This generates acidic conditions for exposed metal structures, especially carbon steel pipes that are widely used, in this case, HSLA steels meet the industry's expectations regarding mechanical efficiency and corrosion resistance. In particular, the X65 grade is already being used in the automotive industry, due to its excellent properties, it is important to evaluate it in terms of corrosion study. The present study shows the behavior against corrosion of X65 steel in the sour gas medium under normal conditions using electrochemical techniques exposed in a saturated H_2S environment, the results show a considerable increase in corrosion, and intensification in surface corrosion associated with MnS inclusions, but the corrosion rate is still within safe limits for use in the oil industry.

Keywords: Steel carbon, EIS, corrosion, H_2S, petroleum industry.

I. INTRODUCTION

Oil and natural gas are the main sources of electricity generation worldwide, and there is a high demand for their extraction and transportation [1-2]. The most economical and at the same time efficient way to transport hydrocarbons is through carbon steel pipelines due to their low cost, ease of welding, and reduced maintenance costs. For this reason, there is a great demand for high-strength carbon steel pipes - HSLA, a material that has high mechanical strength, toughness, and withstands high operating pressures and is resistant to corrosion in acidic environments of oil refineries [2-4]. This type of steel guarantees low-cost structures when compared to other carbon steels, they are easily welded and cut, in addition to their low weight, which means considerable savings during transportation [4-6]. HSLA steels are manufactured through controlled thermomechanical processing (TMCP), which guarantees ideal properties by controlling the microalloying elements (V, Ti and Nb) and controlling the amount of carbon and nitrogen. Although there is a resistance to corrosion in certain media, specifically in extraction wells where the concentration of hydrogen sulfide (H_2S) is high and generates an aggressive environment in terms of acidity which generates significant damage to some metallic structures, causing corrosion problems and hydrogen damage. Even studies on the mechanism and real causes of how hydrogen damage is generated is a relevant issue for the oil industry since new alloys and materials enter operations and the response to corrosion is necessary for quality control. Likewise, the presence of H_2S decreases the pH of the medium, facilitating the corrosive process, compromising the mechanical properties [6-10]. Research in this field is of utmost importance since, during transport, due to industrial conditions, fractures can occur causing spills at sea, fires due to flammable material, direct and indirect economic losses, damage to private property, workers and the environment itself [6-10]. Electrochemical techniques are used to determine corrosion resistance safely and effectively. In recent years, they have demonstrated real, efficient, and rapid results for corrosion detection and control. These techniques can help deepen corrosion studies, which are essential for accident prevention, and help find new quality control alternatives. For this reason, we present an investigation on the impact and evaluation of corrosion in H_2S environments for HSLA X65 steel, using electrochemical impedance spectroscopy (EIS). A set of electrochemical tests and surface characterization by SEM were carried out in solution A of the NACE TM0284-16 standard (5% NaCl-acetic acid and 5% sodium chloride NaCl) [10]. It was examined under three different conditions: aerated state, with and without H_2S saturation. In this environment, it is important to focus on a condition that increases corrosion: the presence of oxygen accelerates oxidation reactions, favoring the dissociation of H_2S. In the presence of free water, it would generate H_2SO_4, increasing the risk to even more aggressive conditions. Further studies on the corrosion resistance of this cost-effective steel could enable applications in industries with controlled environments, treatment with

H_2S-resistant coatings, and even improved design conditions. This is why this research focuses special attention on experimental development, including this condition, to demonstrate this condition.

II. MATERIALS AND METHODS

For this research, samples of high-strength commercial steel X65 were used, characterized by the following chemical composition: 0.038% C, 0.39% Mn, 3.3% Nb, V, and Ti.

1. *Microstructure analysis:*

Microstructural analysis shows metallurgical characteristics such as the phases present in the matrix. For which 3 samples were extracted from the region perpendicular to the rolling direction, according to the recommendations of the ASTM E112-2021 standard [11]. Once obtained, these were sanded and polished to be subsequently observed by optical microscope (OM) and scanning electron microscope (SEM), for the identification of phases on the surface, it was attacked with 2% Nital.

2. *Analysis of inclusions:*

To verify the type and shape of inclusions present in the material, these were cut from the section parallel to the rolling, following the recommendations of the E45 standard (2017) [12]. Surface sanding up to #1200 and diamond paste polishing at 1 µm, then examined by OM and SEM. EDS analysis to verify the chemical composition of inclusions.

3. *Corrosion tests*:

3.1 *ELECTROCHEMICAL TEST*

Electrochemical tests were performed using electrochemical impedance techniques to verify corrosion behavior. A reading of OCP was previously performed for 1500 s and EIS for 45 min on samples sectioned perpendicular to the rolling direction, analyzed using a Gamry 3000 series potentiostat/galvanostat and its respective data analysis software. Test medium: solution A of NACE TM0284 standard, composed of 5% NaCl + 0.5% CH3COOH. Electrochemical cells using a three-electrode system with working electrode (WE): API X65 steel, carbon rod as a counter electrode (CE): ECS and reference electrode (RE): platinum wire. Frequency ranges varied from 10^{-2} Hz to 10^{5} Hz, with 10 data points taken per logarithmic decade and a disturbance amplitude set at 10 mV. All data were acquired in triplicate, and corrosion testing followed the recommendations of ASTM G106-89(2015) [13].

3.2 *CORROSION RATE TEST*

Mass loss tests were performed using samples of HSLA X65 steel with regular dimensions (3 mm x 1.5 mm x 3 mm) to further study the corrosion studies. A total of three samples for each condition were placed in the three solutions evaluated: 1 (steel in solution A); 2 (steel in solution A with H_2S); and 3 (solution A with H_2S and O_2). Before testing, the samples were sanded to #600 with SiC paper, washed, and dried. They were then placed vertically and immersed in small containers. The samples were exposed for a total of 120 hours. Afterwards, they were removed, dried, and the corrosion products were cleaned for mass loss determination, according to the recommendations of ASTM G102-89 (2015) [14].

III. RESULTS AND DISCUSSION

1. Microstructure and inclusions analysis of HSLA X65 steel

The results of the microstructure analysis by scanning electron microscopy (SEM) of the inclusions identified in API X65 pipeline steel are presented in Figure 1. The results of the microstructural analysis show the formation of a ferrite matrix with a low presence of pearlite islands in the matrix. This is expected since it is a continuously rolled steel that follows controlled mechanical processing (TMCP)to provide excellent mechanical properties.

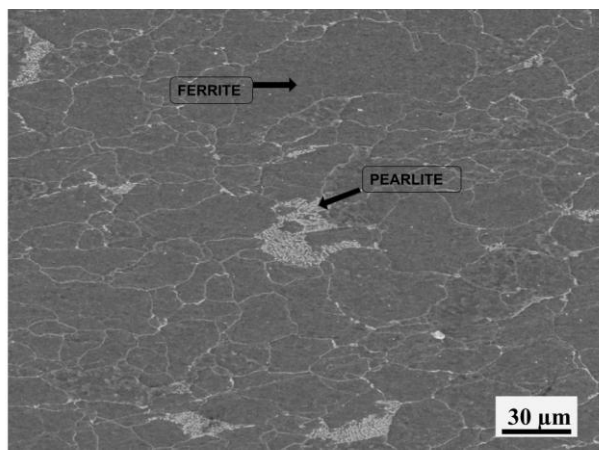

Fig. 1. Analysis of Microstructural analysis in the HSLA X65 steel using scanning electron microscopy with 2% Nital etchant.

The water-cooled thermomechanical control process (TMCP) allows to control the main cooling and rolling processes in order to guarantee adequate microstructural quality in steels, and also allows the addition of elements that reinforce the mechanical and chemical properties of the steel [15].Grain refinement in HSLA steels can be achieved by adding elements of: Nb, V, Ti A. Elements such as niobium or vanadium can be added to steels, allowing higher production yields, thus reducing manufacturing cost (Skobir, 2011). Among these elements, niobium is the most favorable, since its solubility in austenite is lower, consequently favoring the formation of carbides and nitrides, so considerable grain refinement can be obtained with minor additions of this element [14-15]. Refinement of ferritic grain into ferritic-pearlitic microstructures is obtained by restricting austenitic grain growth during rolling or by inhibiting recrystallization of austenite during hot rolling, so that the transformation of austenite to ferrite is produced from uncrystallized austenite [14-15]. High-strength low-alloy (HSLA) steels are widely used in the construction of high-pressure gas pipelines for long distance land transportation, which is why it is important to evaluate the metallurgical and corrosion characteristics. The API 5L standard regulates the limits that correspond to guarantee its safety in operation, manufacturing recommendations, among others. The results of X65 steel are within the recommended limits [15-16]. The mechanical properties of steels depend largely on the microstructure that develops during the solidification process and the weld quality of cooling and both depend on the chemical composition. The chemical composition is the basis of the material and so the conditions of steelmaking are important [15-16]. The composition of the steel can help to obtain a steel with excellent mechanical strength and alloying additions are kept to a minimum to avoid the formation of brittle phases or metallurgical defects [15-16]. The results of the examinations of the inclusions were examined according to ASTM E45 (2015), after polishing the sample, indicating that HSLA steel presents round inclusions, uniformly distributed throughout the matrix. Chemical analysis by EDS-SEM indicates that its composition is based on Al, Ca mainly with a low presence of Mn, the Fe element is inherent to the steel matrix. There is no presence of S in the matrix before performing the tests, which is adequate to ensure that no MnS inclusions have formed.

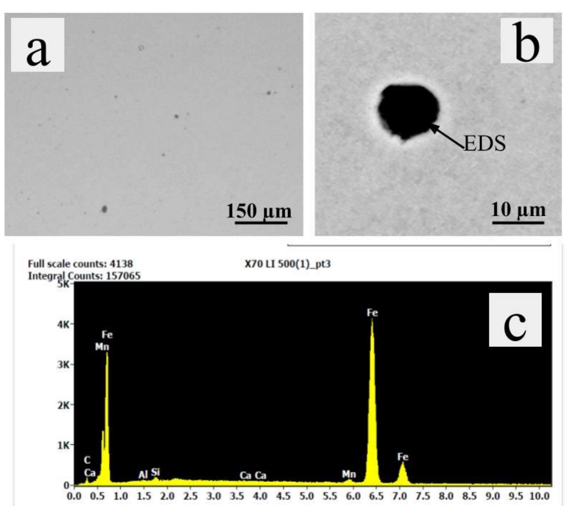

Fig. 2 Distribution for optical microscopy-MO(a); analysis of inclusions in the HSLA X65 steel (b), EDS analysis (c) polished to 1µm without etching.

In particular, irregular inclusions with high concentrations of MnS are detrimental as they act as sites for hydrogen trapping and promote localized corrosion [16-20]. Inclusions are important because they compromise the quality of steels. They are considered metallurgical impurities, affecting quality and durability. Toughness can lead to failure. This is because the presence of inclusions formed by oxides and sulfates can be very sensitive and directly affect fatigue performance, impact resistance, and plasticity, in addition to reducing corrosion resistance. In the case of the studied HSLA X65 steel, the inclusions present a unique and round morphology which is very good, in addition they are rich in Ca, Al, Ti, elements that favor the formation of round inclusions and reduce the presence of MnS inclusions and 7 deformed elongated ones. The results of the microstructure are important to ensure the efficiency in the mechanical properties of the steel. Excessively hard phases such as martensite can make the steel brittle and difficult to weld, while too soft phases can decrease its tensile strength and not serve structurally. The presence of pearlite is associated with the percentage of carbon and the cooling rate during the manufacturing processes. For HSLA X65 steel in particular, both the microstructure and grain size are in line with expectations according to API 5L [20-21].

2. Corrosion tests

2.1 Electrochemical test

To evaluate the impact against corrosion, electrochemical impedance techniques were used, considered non-destructive techniques, the selected medium was solution A of the NACE TM 0284-2016 standard, under normal conditions, the study material were samples of HSLA X65 steel. A 3-electrode system was used, and the results show an OCP (figure 3) that stabilizes correctly at 1500 s with potentials within the limit of the material, the condition in the presence of H_2S shows a higher potential, similar when the solution has the presence of O_2 and the condition with lower potential, that is, nobler or less active potentials when the desaerated solution A does not have the activity of H_2S or participation of oxygen. These results can be verified in Table 1.The OCP results show nobler potentials in the condition without the presence of H_2S, which is consistent with the literature that indicates lower activity, but in the presence of H_2S and O_2 the potentials become more active, indicating that these media are more susceptible to corrosion [21-23]. The measurement of the open circuit potential known by its acronym in English Open Circuit Potential (OCP), this is one of the main corrosion parameters and allows to know the steady-state potential of the sample under open circuit [21-23]. This parameter is important because it allows us to know the protective capacity of the coating on the substrate, the OCP potential is a full potential of the electrochemical cell when the metal is in direct contact (immersed) with the electrolyte, this potential obtained ultimately defines its ability to oxidize or reduce [21-23].Open circuit potential (OCP) is graphically represented as the potential (Y axis) vs exposure time (X axis), for the graphical determination the most active potential (most negative potential) indicates that the material exposed to the electrolyte will dissolve more quickly in an electrolyte than another metal that has a higher circuit potential [21-23].

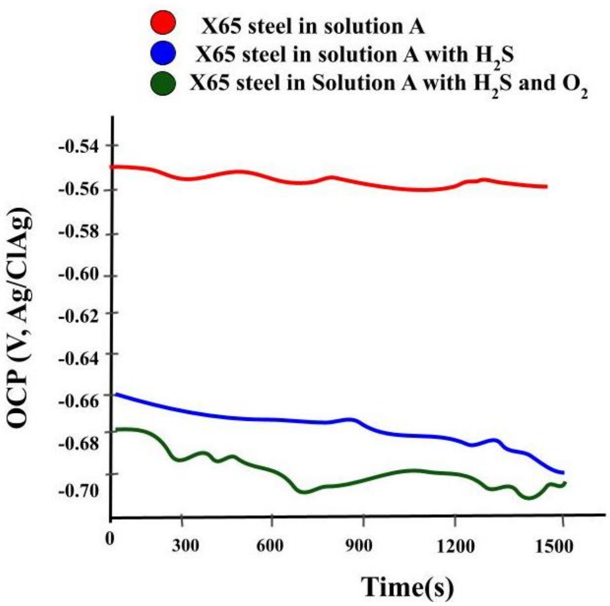

Fig. 3. Open Potential Circuit (OCP) for HSLA X65 steel in deaserated Solution A, H_2S and O_2 media (NACE TM0284, 2016).

Table 1. Values Open Potential Circuit (OCP) for HSLA X65 steel in desaerated Solution A, H_2S and O_2 media (NACE TM0284, 2016). Obtain in fig. 3

Condition Solution	Potential OCP E (V vs Ag/AgCl)	
	E (mV)	Value
X65 steel in solution A	´- 0.56	Non-susceptible
X65 steel in solution A + H_2S	-0.66	susceptible
X65 steel in solution A + H_2S+O_2	-0.68	susceptible

The EIS results (Figure 4) show a similar behavior where the desaerated condition without H_2S shows the highest corrosion resistance, followed by the presence of H_2S and with the presence of oxygen showing that in this medium the corrosion resistance decreases considerably. There is greater susceptibility to corrosion in this medium, there are two-time constants reflecting the onset of the corrosive process and surface dissolution.

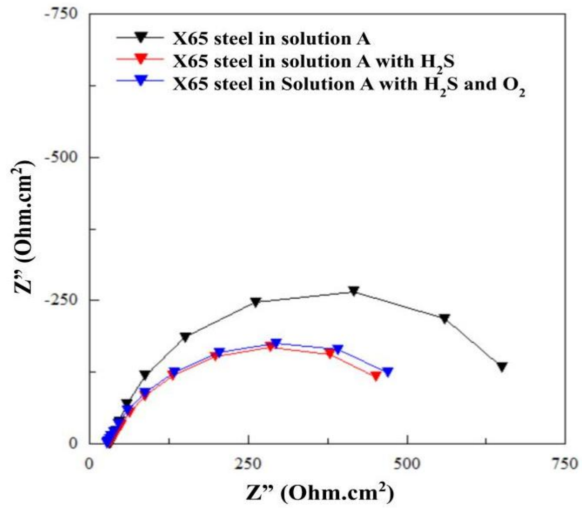

Fig. 4. results of Nyquist diagrams for HSLA X65 steel in desaerated Solution A (NACE TM0284, 2016), both without and with H_2S saturation.

Fig. 5 shows the surface characterization of the samples of the HSLA X65 steel surfaces, the samples exposed to H_2S and O_2 present a higher intensity of surface attack observed by Optical Microscopy -OM with the presence of localized attack in some regions. The SEM characterization (Figure 6) shows spots with significant concentrations of S, Mn and Fe. These elements indicate the formation of sulfates and the dissolution of iron. Surface comparison shows greater localized attack in the presence of H_2S and O_2 as shown in corrosion tests. The corrosion behavior of a material is important to verify whether or not the material is suitable for certain applications, evaluating its resistance to the same industrial conditions guarantees to optimize performance, there are various techniques, however, non-destructive techniques such as EIS allow to evaluate in a short period of time, ease of work in the laboratory, data performance in a short time, obtaining information on the real corrosion resistance of the material [24-29]. Environments rich in H_2S according to the literature decrease the pH of the medium, which promotes corrosion in acidic media. As expected, the presence of hydrogen and oxygen within the material can directly affect its properties and weaken it. Therefore, in the oil and natural gas industry, the materials used must comply with specific controls, be resistant to acidic media, low cost due to high demand, easy to weld [24-34]

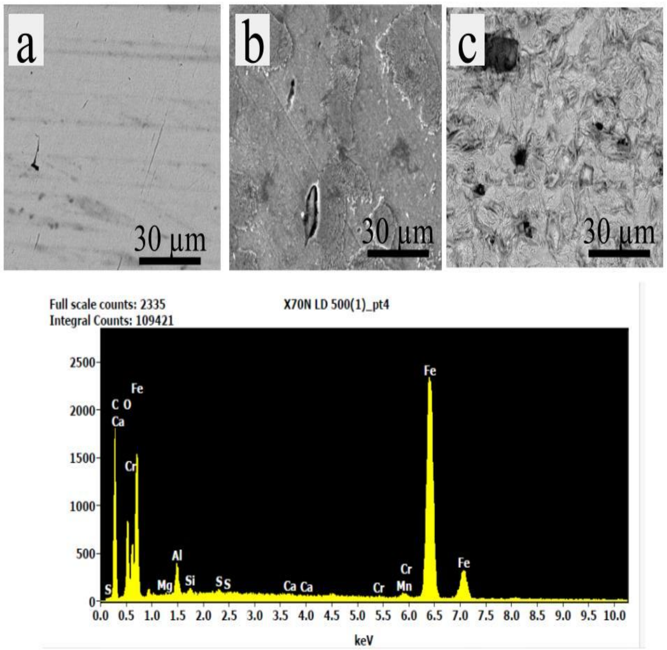

Fig. 5. Surface Analysis- SEM of HSLA X65 samples submitted to electrochemical testing, in solution A(a), solution A with H_2S (b) and solution A with H_2S and O_2 (c). Cross-sectioned perpendicular to the rolling direction.

2.2 Corrosion rate test:

The mass loss results in Figure 6 and table 2 confirm greater material loss when exposed to solution in the presence of H_2S and O_2, and a lower mass loss in solution A without these agents. This behavior supports the evidence of the electrochemical tests, confirming the greater corrosion kinetics due to the presence of H_2S and O_2. The results are consistent with the literature. Similar research confirms a greater increase in the corrosion rate in the presence of H_2S, which facilitates surface corrosion. This is due to the acidity of the medium. The presence of O_2 favors a reduction, facilitating the formation of oxides on the steel surface. This is due to the iron dissolution process [28-29].

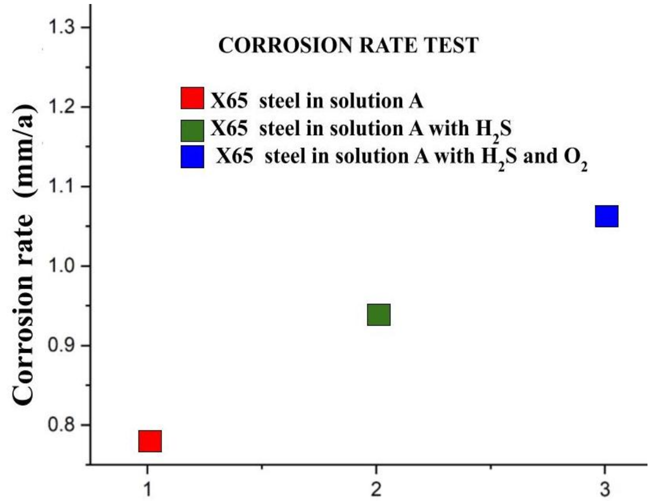

Fig. 6. Result of corrosion rate of HSLA X65 exposure in solution A(a), solution A with H_2S (b) and solution A with H_2S and O_2 (c). Cross-sectioned perpendicular to the rolling direction.

Table 2. Values corrosion rate test for HSLA X65 steel in desaerated Solution A, H_2S and O_2 media (ASTM G102-89, 2015). Obtain in fig. 6

Condition Solution	Corrosion rate (mm/a)
X65 steel in solution A	0.65± 0.01
X65 steel in solution A + H_2S	0.97± 0.01
X65 steel in solution A + H_2S+O_2	1.0 ± 0.01

Studies on pipe corrosion in the presence of H_2S indicate that high concentrations can facilitate surface corrosion, compromising the performance of the pipes. This is why studies are important for early detection, finding suitable solutions or selecting materials. On the other hand, the presence of oxygen acts as a cathodic catalyst of the reaction, which is associated with increasing corrosion kinetics due to the effect of oxidation in the medium [30- 34]. The influence of environmental conditions is important for the guarantee of pipe structures, that is why the presence of sulphate, oxygen can trigger corrosion problems associated with the loss of their properties, corrosion studies are important to improve the performance of high strength low alloy steels (HSLA) due to metallurgical changes and stresses in full operation can bring terrible consequences [28-37].

IV. CONCLUSIONS

The following conclusions can be drawn from the present investigation on the corrosion of HSLA X65 steel:

1. The microstructural characterization showed a ferrite matrix and the presence of pearlite in the matrix, which is suitable for this type of steel.
2. Inclusion analysis showed round-shaped inclusions with the presence of Ca, Al mainly, and Mn without the presence of S.
3. Corrosion tests using electrochemical and mass loss tests showed greater susceptibility to corrosion and greater material loss in the presence of H_2S and O_2, while in a deaerated medium the corrosion resistance is greater.
4. In sour gas environments, the surface of HSLA steel is more intense.
5. MnS inclusions are associated with localized corrosion in sour gas conditions, the presence of H_2S and O_2 can generate more corrosion.
6.

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