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ARTICLE

Evaluation of corrosion behavior of a pipeline carbon steel (API 5L-X52) subjected to a short-term immersion test in a sour aqueous media

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Abstract

API carbon steels pipes are widely used in oil and gas industry for transporting products. They are constantly exposed to aggressive environments, most of them rich in CO2 and H2S concentrations, which increases wear by corrosion. This study investigated the corrosive behavior of API 5L-X52 carbon steel when immersed in a static solution simulating sour media for short periods of time, simulating conditions of initial contact between a sour media and API steels, which is of interest because oil production pipelines are affected by corrosion, causing embrittlement even after very short service times. Tests were performed according to ASTM G31 standard. The mass loss method was used to evaluate corrosion rates at each time point, and Scanning Electron Microscopy (SEM) with Energy Dispersive X Ray Spectroscopy (EDS) was used to characterize the microstructure and corrosion products of the samples. The results showed that the first few hours of contact with the sour media generate a peak in the corrosion rate; after this time, corrosion rate decreases and tends to stabilize. Furthermore, the presence of H2S in the media accelerates the formation of craters on the surface, causing pitting corrosion damage. Finally, the presence of Sb in the EDS analysis helps to identify corrosion products on the metal surface.

Index terms: corrosion, API 5L, sour media, H2S, immersion tests.

Resumen

Las tuberías de acero al carbono API son ampliamente utilizadas en la industria del petróleo y el gas para el transporte de productos, están constantemente expuestas a ambientes agresivos, la mayoría de ellos ricos en concentraciones de CO2 y H2S, lo que aumenta el desgaste por corrosión. En este estudio se investigó el comportamiento corrosivo del acero al carbono API 5L-X52 cuando se sumerge en una solución estática simulando un medio amargo durante cortos periodos de tiempo, simulando las condiciones del primer contacto entre un medio amargo y los aceros API, lo cual es interesante debido a que los oleoductos de producción de petróleo se ven afectados por la corrosión, causando fragilización incluso después de tiempos de servicio muy cortos. Las pruebas se realizaron según la norma ASTM G31. Se utilizó el método de pérdida de masa para evaluar las tasas de corrosión en cada periodo de tiempo, y se empleó microscopía electrónica de barrido (SEM) con espectroscopia de rayos X de energía dispersiva (EDS) para caracterizar la microestructura y los productos de corrosión de las muestras. Los resultados mostraron que las primeras horas de contacto con el medio amargo generan un pico en la tasa de corrosión, después de ese tiempo la tasa de corrosión disminuye y tiende a estabilizarse; además, que la presencia de H2S en el medio acelera la formación de cráteres en la superficie, generando daños por corrosión por picaduras. Finalmente, la presencia de Sb en el análisis EDS ayuda a identificar los productos de corrosión en la superficie del metal.

Palabras clave: corrosión, API 5L, medio amargo, pruebas de inmersión.

I. INTRODUCTION

The phenomenon of corrosion is a process of electrochemical deterioration that leads to an accelerated material degradation, specifically on metals exposed to corrosive fluids [1], [2]. Several factors affect this phenomenon such as material properties, for example: hardness, metallurgical properties and chemical resistance; and media properties as: carbon dioxide (CO_2) or hydrogen sulfide (H_2S) concentration, viscosity, pH and so on [3, 4]. But one of the most important are the environmental conditions like flow conditions, pressure, temperature, among others. There are two types of corrosion according to the chemical factors of the media, sweet and sour corrosion [5]. Sour or acid corrosion occurs when the H_2S gas in the environment is dissolved into the solution, acting as a catalyzer for anodic and cathodic reactions; this usually promotes the dissolution of the iron at the anodic site, promoting the formation of some corrosion products as FeS [4], [6], this process is expressed in the following equation (1). In some cases, the formation of these corrosion products could act as a protective layer, delayed corrosion process but this effect depends on certain special conditions [7].

$$Fe + H_2S \rightarrow FeS + 2H + H_2O \tag{1}$$

The presence of H_2S increases the electrochemical reaction in the corrosion process, which leads to different forms of corrosion as uniform and localized corrosion such as pitting [4, 8]. This is because H_2S inhibits hydrogen molecules formation, producing an accumulation of hydrogen ions near the material surface allowing these ions to permeate inside the material lattice structure, accumulating in the interstitial spaces, enhancing premature failure [1], [9].

Several industries are affected by corrosion damaging a lot of industrial equipment, one of them is the oil and gas industry, which uses pipelines to transport corrosive fluid for long distances, it uses them due to its price and ease of manufacturing [10], [11]. Commonly these pipelines are produced with API steels and have a relevant concern in this industry because of their excellent mechanical and physical properties, nevertheless, the internal exposure to harsh and corrosive environment with high levels of CO_2 and H_2S, along with high pressures and flow velocities, the corrosion is inevitable [12]. Corrosion is one of the principal root causes of failure in pipelines [7], [13], for this reason is essential to identify corrosion and wear deterioration on pipelines [14].

Some authors have studied the effect of sour media and dissolve hydrogen on pipelines, API carbon steels, some of them report how these conditions affect the mechanical properties of the material [15], [16]. Others point out that the microstructure and chemical composition of the material define how it is affected by the media [17], [18]. But, in sour corrosion, stress-corrosion cracking (SCC), uniform corrosion and pitting are the most common wear deterioration. Hidayat et al., [19] evaluates the dependency of SCC of an API in a sour media, Shabani et al. [11] exposed that pitting by corrosion is the initial point of crack in the weld sections due to these pits acting like stress concentration points. Then, Ren et al. [20] conclude that the addition of H_2S initially accelerates corrosion rate and then decreases as corrosion layers form; furthermore, sour corrosion is more likely to generated pitting.

Nowadays, there is a lack of research on the possible damage of brief exposure of API steels to corrosive environments, especially in sour media and how hydrogen absorption due to the brief exposure to this media affects this material [16]. For that reason, the aim of this study is to investigate the effect that short time immersion tests, without induce accelerated corrosion by applying a potential difference, has in corrosion rate and pitting formation.

II. EXPERIMENTAL DETAILS

A. Immersion test

The immersion tests were carried out at Escuela Superior de Ingeniería Química e Industrias Extractivas of the Instituto Politécnico Nacional based on ASTM G31-72 standard [21]. Fig. 1 shows a schematic diagram of the flask used and how the samples are supported along the tests. For this setup, four samples were used, each one submerged for different test periods. The first three samples were submerged for 72, 192 and 264 hours, respectively. The last sample was submerged for 72 hours, the same as the first one, but with the solution contaminated with the previous corrosion products from the other three samples. The immersion times were chosen based on the ASTM G31-72 [21] and NACE TM0169/G31-21 [22], where it is mentioned that the most common testing periods are from 48 to 168 hours, hence it was decided to widen the common intervals, resulting in these three different immersion times. The tests were carried out twice according to what is mentioned in the standard. All specimens were weighed before and after each test with a digital balance with an accuracy of 0.1 mg as well as cleaned before and after conducting the immersion test with the aim of removing corrosion products residues adhering to the sample to obtain more accurate mass loss measurements. The media selected for this study was a sour media rich in S, finally all tests were performed at room temperature. The test conditions are listed in Table 1.

TABLE 1.
IMMERSION TEST CONDITIONS.

Parameters	Values
Specimen dimensions	50x20x3 mm
Number of samples	4
Media	Sour media
Test temperature	Room temperature
Test duration	72 hrs., 192 hrs., and 264 hrs.

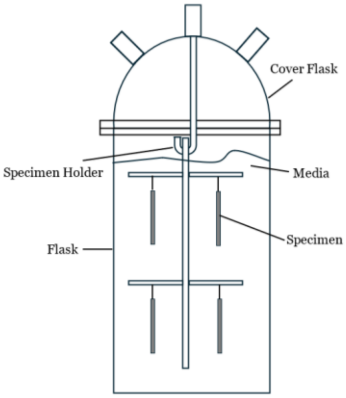

Fig. 1. Schematic diagram for immersion test.

B. Materials

1) API 5L-X52

The material tested was a carbon steel pipeline (API 5L-X52) used in oil and gas industry to transport crude oil due to its high mechanical strength and endurance to corrosive media [23]. The samples were machined into a rectangular shape with a thickness of 3 mm; they were not thicker due to the circular shape of the pipe. Samples were prepared according to ASTM G31-72 standard method [21], polished with silicon carbide sandpaper 120, ultrasonic cleaned and weighed with a digital balance. An EDS analysis was accomplished with a JEOL model JSM 7800 microscope which incorporates an EDS detector, to know elemental composition of the material along with other tests to know its chemical and physical properties, which were reported in a previous work [24]. Fig. 2 shows an SEM image of the microstructure of the material with a magnification of 5,000X; two main phases of the material can be observed here: ferrite and pearlite. In this case, polygonal ferrite is the main microconstituent appearing as equiaxed grains and relatively smooth areas separated by irregular, brightly edges. On the other hand, pearlite appears in the lower center of the SEM image with greater roughness, and a more lamellar morphology. In this case, ferrite tends to dissolve more easily than pearlite, as ferrite is more active, acting as an anode, while pearlite acts as a cathode. Furthermore, in sour environments, it generates localized corrosion and pitting, primarily where inclusions are present.

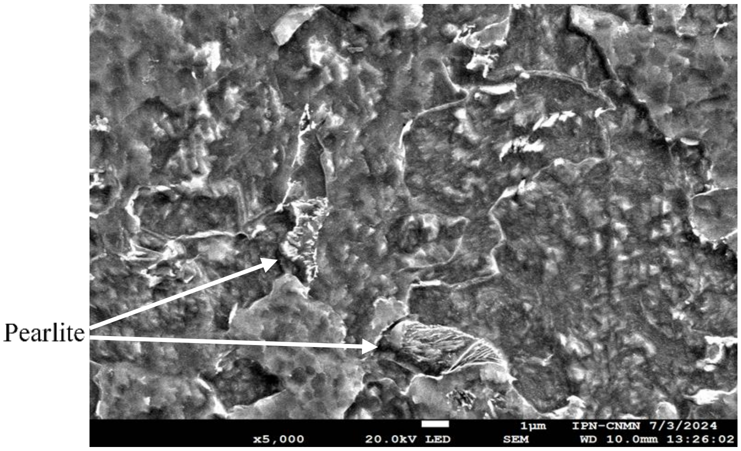

Fig. 2. API 5L-X52 SEM (5,000X magnification).

2) Sour media

Within the phenomenon of corrosion there are two types of it, sweet corrosion (high CO_2 concentration) and sour corrosion (high H_2S concentration) [5]. The media used in this investigation was a brine solution enriched with H_2S, prepared following the NACE International Publication 1D182 [25] which is used to reproduce sour tests. Initially a synthetic brine solution was prepared in a proportion of 9.62% of sodium chloride (NaCl), 0.305% of calcium chloride ($CaCl_2$), 0.186% of magnesium chloride hexahydrate ($MgCl_2 \cdot 6H_2O$) and 89.89% of distilled water. To generate the H_2S, is added to the brine 1.700 mg/L of acetic acid (CH_3COOH) and 3.530 mg/L of sodium sulfide ($Na_2S \cdot 9H_2O$) to the brine previously prepared, these quantities are valid for an amount of 500 mg/L of solution. This media was selected due to the scarcity of sweet crudes around the world and because in Mexico this type of crude has a higher production [26]. Besides, sour media with H_2S is more prone to corroded pipelines [27].

III. RESULTS AND DISCUSSON

In these tests, corrosion rate was calculated based on the mass loss of samples. During the initial immersion time, a thin corrosive layer began to form, and some corrosion products adhered to the metal surface, as shown in Fig. 3. The corrosion rate peaked during the first 72 hours, as expected, due to the lack of surface protection on the material. After the first immersion time, the corrosion rate gradually decreased until reached an average value of 2.5 mm/year approximately. This decrease in corrosion rate can be attributed to the thin protective film formed on the surface, as mentioned by Ren et al. [20], where corrosion products around the exposure area of the sample act as a protective layer. These data are shown in Fig. 4 which present the average of the test performed with their respective error bars.

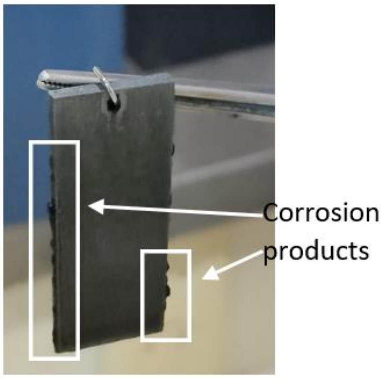

Fig. 3. First specimen with corrosion products after immersion test.

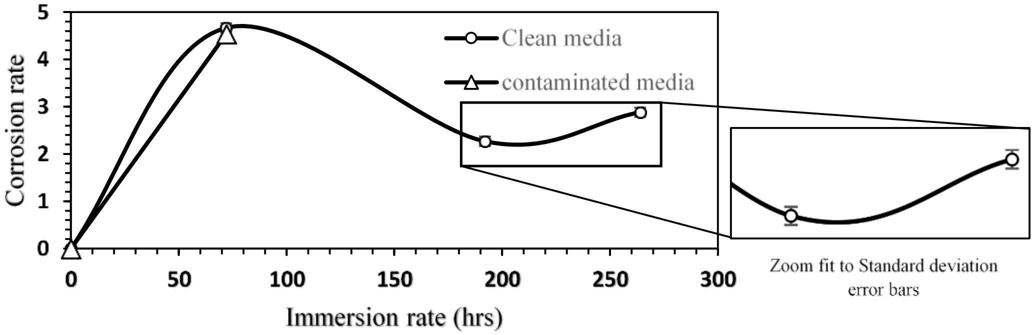

Fig. 4. Corrosion rate with immersion test corrosion.

Along with these results, it is possible to assume that the sour media does not degrade over time or with the presence of corrosion products within it, at least during the eleven days of the test. This was determined with the fourth sample; this sample was immersed in the media after the first two samples were taken out, and its corrosion rate was compared to that of the first. The corrosion rate of both samples, the fourth and first, was almost the same, indicating that the media does not tend to degrade over time under these conditions. It is necessary to evaluate this material under other conditions, such as more contaminated media or longer exposure time, to determine how these conditions affect the degradation of the media. However, corrosion products in the exposed area appear to form more rapidly than at the beginning of the test. Fig. 5 shows the third and fourth sample; the first is the sample with the longest immersion time, and the second one was immersed after the media became contaminated with the corrosion products.

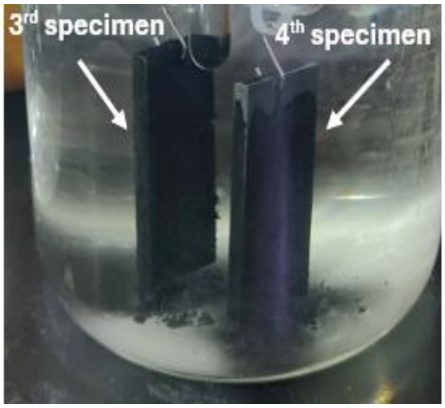

Fig. 5. Corrosion products of third and fourth specimens after immersion test.

After the immersion testing, the samples were analyzed with SEM and EDS. Pitting was found in various areas of the material. In Fig. 6 the pitting phenomenon and crater formation can be observed. In this case, the pitting is due to the gradual degradation of the different layers that make up the material. This type of pitting formation could be due to inclusions or precipitates in the sample, commonly Si or Mn [28]. The EDS punctual analysis performed on the pitted areas shows that the different crater layers contain Sb and Mn in their compositions, indicating that the degradation of the surface layer reveals some precipitates in the microstructure as a result of contact between the sour media and the metal surface. Fig. 7 shows the EDS analysis of different crater areas. The way the pits form is noteworthy, showing concentric circles with different layers.

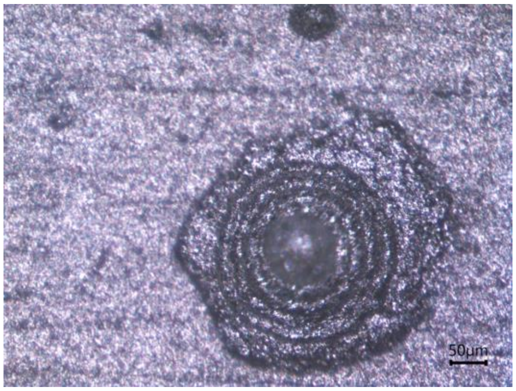

Fig. 6. Pitting corrosion.

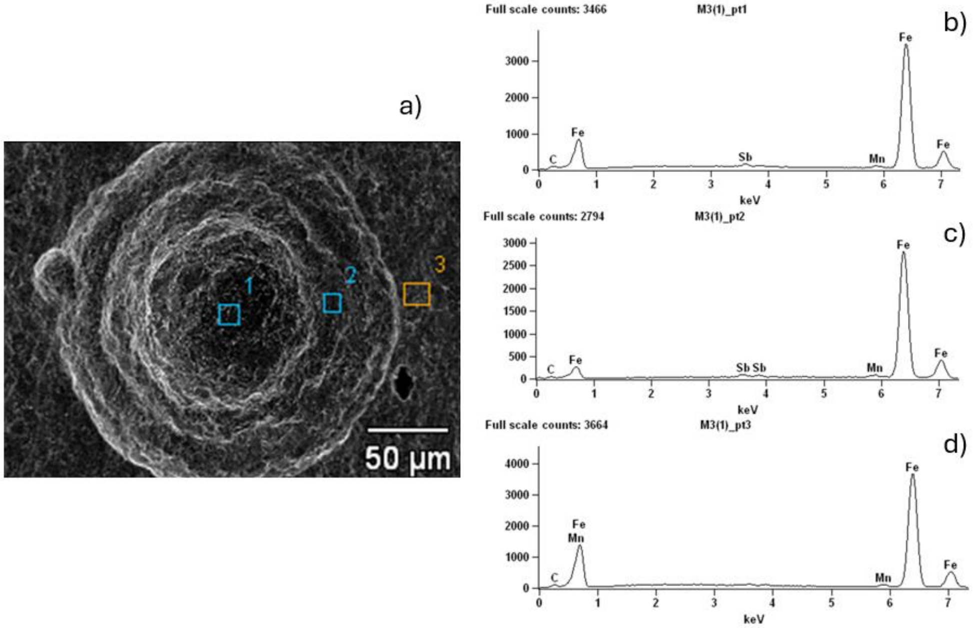

Fig. 7. EDS punctual analysis, a) SEM image of pitting area, b) 1st punctual EDS analysis, c) 2nd punctual EDS analysis, d) 3rd punctual EDS analysis.

TABLE 2.
PUNCTUAL EDS ANALYSIS OF PITTING AREA.

	Atom %			
	C-K	Mn-K	Fe-K	Sb-L
Point 1	39.41	0.85	58.97	0.77
Point 2	28.74	0.77	69.77	0.72
Point 3	46.07	0.78	53.15	

Fig. 8 shows a chemical composition map obtained from SEM analysis on the samples. The presence of antimony (Sb) is observed in the analysis; in Fig. 8c, the Sb distribution is dispersed and relatively homogeneous, indicating that the presence of this element is not due to possible metallographic contamination. Therefore, the Sb detected on the corroded surface may plausibly originate from trace contamination in the sodium sulfide nonahydrate reagent. Although Sb is not routinely reported in commercial certificates of analysis for $Na_2S{\cdot}9H_2O$, Sb is a well-known chalcophile element that naturally associates with sulfide minerals, primarily as stibnite (Sb_2S_3) [29]. Sulfide media are also known to efficiently solubilize Sb through the formation of thioantimonate complexes [30]. Furthermore, commercial Na_2S may contain metal ion contamination [31]. Hence, contamination by trace amount of Sb from industrial sulfide raw materials or reagent manufacturing cannot be ruled out, representing a reasonable source for the Sb detected by SEM-EDS analysis. Thus, Sb adsorption onto the FeS layer or Fe-S-Sb coprecipitation is likely to occur.

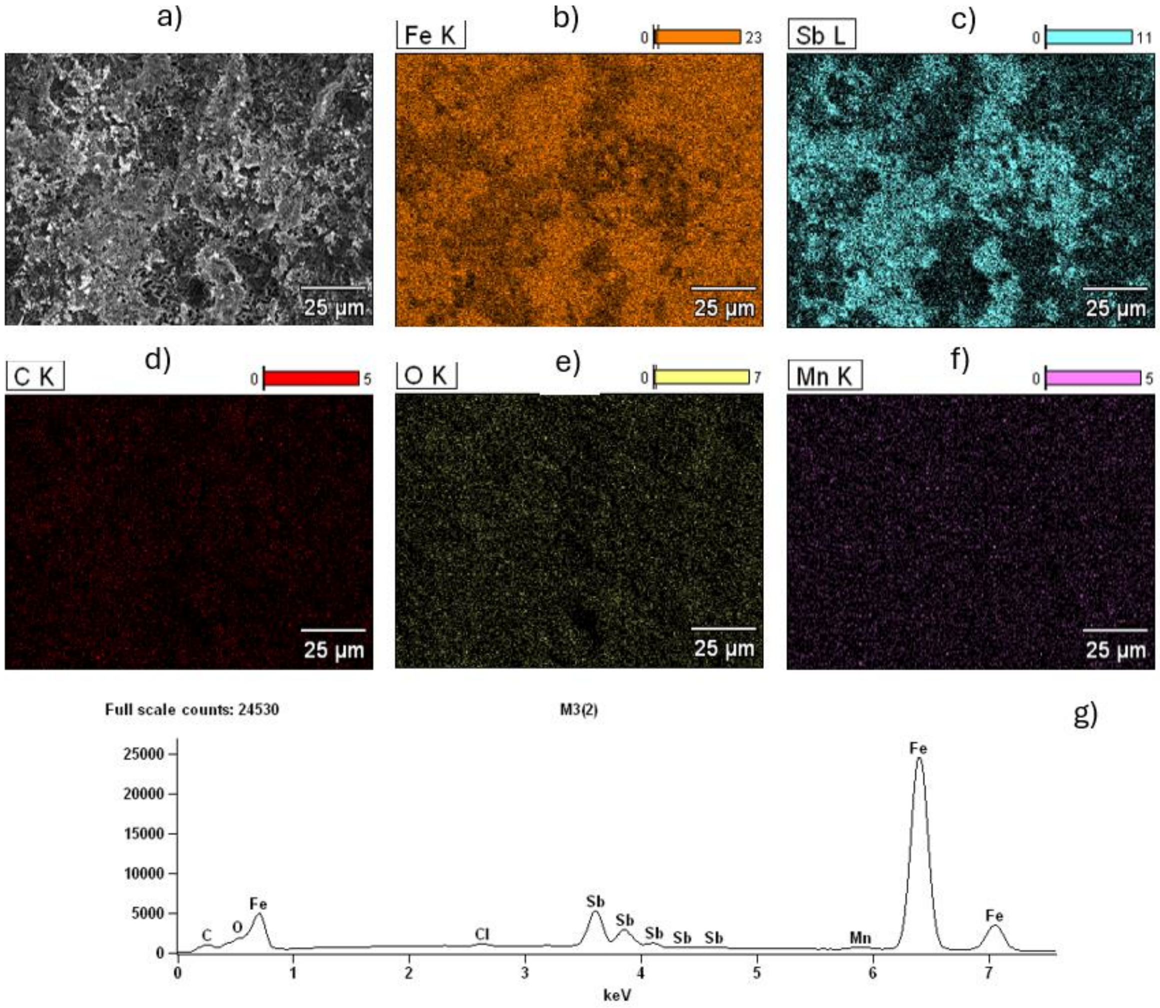

Fig. 8. Chemical composition analysis a) SEM image, b) Chemical mapping of Fe, c) Chemical mapping of Sb, d) Chemical mapping of C, e) Chemical mapping of O and f) Chemical mapping of Mn.

TABLE 3.
PUNCTUAL EDS ANALYSIS OF PITTING AREA.

Element line	Wt %	Atom %
C	11.88	39.39
O	1.74	4.32
Cl	0.41	0.46
Mn	0.93	0.67
Fe	70.81	50.50
Sb	14.24	4.66
Total	100.00	100.00

The spatial relationship between the corroded image and the Sb mapping (Fig. 8a and 8c) indicates that the Sb concentration occurs in the corrosion products, not in the base metal. These products are highlighted as a rough or shiny area in Fig. 8a. It is important to note that in sour corrosion, some elements such as Sb, As, and Sn are known as hydrogen recombination poisons, leading to increased absorption of atomic hydrogen and raising the risk of SSC or hydrogen embrittlement. Therefore, as Macedo et al. [32] mention in their experiments, the corrosion rate depends more on environmental factors than on metallurgical ones.

IV. CONCLUSION

1. The immersion corrosion test shows non-uniform pitting wear in the samples. This phenomenon exhibits a peculiar crater formation pattern, consisting of concentric circles that wear down the material in layers. These pits could be attributed to inclusion in the material matrix, as typical inclusion elements appear in the elemental analysis of the craters. This form of corrosion supports existing literature data indicating that pitting and stress corrosion cracking are the main types of corrosion in sour environments.
2. The corrosion rate obtained with this test indicates that the major damage suffered by the material is at the beginning of the test, in the first 72 hrs., that the material comes into contact with the sour media, after this point the corrosion rate decreases and tends to stabilize. This is because when the sour media meets the metal, there is no protective layer on the surface of the material, which accelerates corrosion process. Subsequently, corrosion products that begin to form act as a protective layer, inhibiting hydrogen ions from catalyzing reduction reactions.
3. Despite being an unexpected situation, the appearance of antimony (Sb), in the chemical element composition map, is a way to identify the corrosion products, since it does not concentrate in the base metal, but on the rougher (corroded) surface; however, further investigation is needed to understand the appearance of this element. It is adsorbed or deposited in the FeS layer due to its affinity with S. This element also could enhance the pitting formation due to the inhibition of hydrogen molecules formation.

CRediT (*Contributor Roles Taxonomy*)

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