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Study of the inhibiting efficiency of the corrosion inhibitor (prop-2-yn-1-ol, methyloxirane) of mild steel in the chemical pickling (18.5% HCl)

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Abstract: The study of the inhibitory efficacy of prop-2-yn-1-ol, methyloxirane for corrosion control of mild steel in concentrated hydrochloric acid (18.5%) at temperature 80 °C (chemical pickling conditions of steel) was investigated using electrochemical and gravimetric methods for different concentrations. Using weight loss, and potentiodynamic polarization techniques and the morphological structure, the results shows that inhibitor had an excellent protection. The substance has demonstrated remarkable inhibition efficiency, and its inhibitory characteristics are stronger as concentrations increase (Inhibitory efficacy of up to 91 % for 1.2g/L). Based on the polarisation curves, the addition of the inhibitor in the HCl solution induces the decrease of the anodic current corresponding to the attack of the metal. The morphological structure was examined by scanning electron microscopy, which clearly shows this inhibitor's good protection, which delays the acid attack of bare steel with a uniform and bright pickling appearance.

Keywords: Corrosion, inhibitor, hydrochloric acid, chemical pickling, mild steel.

1. Introduction

Many industries prefer the use of mild steel. Because of its superior mechanical qualities and inexpensive price, it is frequently used as a piping and oil and gas production building material for conduit tubes, downspouts, and transport tubes (Aiad *et al.* 2018; Abdallah *et al.* 2018; Abd El-Lateef *et al.* 2018; A. Zouitini and Zouitini 2018; Al-Azawi et al. 2016; Adam et al. 2018; Wang et al. 2012;. Corrosion is a major problem in the industrial sector, causing significant economic losses every year. The use of corrosion inhibitors is a way to prevent or minimize corrosion. In particular, the use of inhibitors in the chemical pickling of mild steels in hydrochloric acid (HCl) is a common practice in many industries. Prop-2-yn-1-ol, methyloxirane is one type of corrosion inhibitor that has shown

promise in reducing corrosion in this context. However, the effectiveness of this inhibitor needs to be studied in depth to determine its effectiveness and potential limitations (Hegazy 2018).

However, mild steel has experienced corrosion assault in industrial settings, particularly from attacks caused by hydrochloric acid, which is employed in the sector for acid cleaning, descaling, and pickling (Elouafi *et al.* 2002; Bouklah *et al.* 2006). The usage of it is still significantly limited by its sensitivity to corrosion, though (A. Zouitini *et al.* 2018; Al-Taweel *et al.* 2019; Hegazy 2018; Mohamad *et al.* 2014; Salman *et al.* 2019). The most popular strategy for preventing metal corrosion is the application of corrosion inhibitors because it doesn't call for any specialized tools is cheap and is simple to implement (El-Lateef *et al.* 2018; Kadhim *et al.* 2017; Kharbach *et al.* 2017; Zhang *et al.* 2015).

Most of the inhibitors intended for this application are organic compounds containing coordination sites for joining with metals. These coordination sites may include many linkages as well as heteroatoms including nitrogen, sulfur, and oxygen. These inhibitors minimize corrosion by obstructing the active corrosion sites, and by the complexion on the metal surface, they create a protective layer (Cavani *et al.* 1991; Mousty *et al.* 1994; Playle *et al.* 1974). Due to the interaction between the molecular structures of the inhibitor and the active sites found on the metal's surface, they primarily function through adsorption processes (chemisorption and/or physisorption) (Abdel-Karim and El-Shamy 2022; Ahamad *et al.* 2010; Ouali *et al.* 2010). The density and availability of free pi electrons on inhibitor drugs' functional groups often regulate the interaction's strength. Overall, due to their toxicity, many corrosion inhibitors generate issues for the environment. The environment should not be harmed by new corrosion inhibitors, and they should be non-toxic (Khaldi *et al.* 1998; Legrouri *et al.* 1999).

The search for novel organic anti-corrosive chemicals has been intensive in recent years, and N-heterocyclic compounds have emerged as potent corrosion inhibitors.

Pickling inhibitors are chemical compounds added to the pickling solution to suppress the aggressive action of acid on the metal surface. They form a protective film or barrier on the metal, reducing the rate of acid attack and preventing excessive dissolution of the metal. Inhibitors greatly improve the pickling process, ensuring controlled and efficient removal of impurities while minimizing metal loss and corrosion.

Prop-2-yn-1-ol, methyloxirane, also known as propargyl alcohol epoxide, is a versatile organic compound with excellent acid inhibiting properties. Its chemical structure includes a triple bond and an epoxide ring, giving it unique properties and making it an ideal candidate for pickling inhibition.

The inhibiting action of prop-2-yn-1-ol, methyloxirane can be attributed to its ability to adsorb onto the metal surface and form a protective film. The triple bond and epoxy ring contribute to the formation of a stable complex with metal ions, hindering acid attack and reducing the rate of metal dissolution. This film acts as a barrier, preventing direct contact between acid and metal, thus reducing the corrosion process. There are several advantages to using prop-2-yn-1-ol, methyloxirane as a pickling inhibitor. Firstly, it effectively minimizes metal loss during the pickling process, thus preserving the structural integrity of mild steel. Secondly, the inhibitor reduces the risk of surface pitting and corrosion, thereby improving surface quality. In addition, prop-2-yn-1-ol, methyloxirane is compatible with the HCl medium, facilitating its application and integration into existing pickling processes. The a pplication of prop-2-yn-1-ol, methyloxirane as a pickling inhibitor in HCl medium (18.5%) for mild steels shows promising potential for various industrial sectors. The use of this inhibitor can improve the efficiency

and reliability of pickling processes, thus contributing to the production of high-quality steel products. Further research and development are required to optimize its dosage, its compatibility with other pickling acids and its performance under different processing conditions.

Continuing to search for the progress of heterocyclic compounds as efficient inhibitors for the mild corrosion steel in HCl solution, the present investigation discusses the inhibitory efficiency, namely, prop-2-yn-1-ol, methyloxirane on corrosion of mild steel in (18.5%) HCl by using weight loss measurements and scanning electron microscopy (SEM). The purpose of this research was to investigate mild steel's resistance to corrosion in a (18.5%) HCl media. Utilizing weight loss, and potentiodynamic polarization techniques, the surfactant was thoroughly studied. Additionally evaluated was the mild steel adsorption mode of the inhibitor chemical and the mechanism of corrosion inhibition.

2. Experimental procedure

2.1 Materials

The substance used in this study are analytically trustworthy, therefore they haven't been previously purified. Moreover, the prop-2-yn-1-ol, methyloxirane molecules (**Figure1**) (commercial name: NP INIBIT) was supplied by (Quaker company, French). The hydrochloric acid at 37% was obtained from VWR INTERNATIONAL SAS. The mild steel samples used in this study were pre-treated prior to the experiments by grinding with various emery papers of grades 220, 400, 800, 1000, and 1200, rinsing with bi-distilled water, degreasing with acetone, and then washing again with double-distilled water. Finally, the samples were washed again with air drying before use. The molar HCl solutions used for all electrochemical and gravimetric studies were made by diluting the analytical solution, HCl 37%, with bidistilled water.

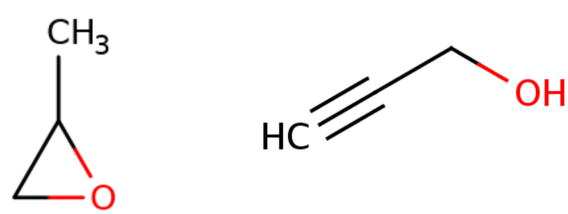

Figure 1: Structural molecular "prop-2-yn-1-ol, methyloxirane

2.2 Gravimetric measurement

Using the ASTM approach, the weight loss measures were refined. A double glass cell with a thermostat-cooling condenser was used for the gravimetric testing. A (18.5%) HCl solution was used to immerse rectangular samples that measured 3 cm x 3 cm x 2.7 cm in both the absence and presence of various concentrations of a prop-2-yn-1-ol, methyl oxirane, for 5 min at temperature 80 °C. After The steel samples were then withdrawn, rinsed with bi-distilled water, cleaned with acetone, and dried before being weighed. The efficiency of the inhibitor is calculated by the following equation:

$$EI = \frac{\Delta m_0 - \Delta m_i}{\Delta m_0} \times 100 \qquad (1)$$

Δm_0, represents the mass loss without inhibitor and Δm_i, the mass loss in the presence of the inhibitor.

2.3 Electrochemical measurement

The three electrodes are connected to a potentiostat / Galvanostat (PGP201) controlled by a computer via a "Volta Master4" software. The scanning speed applied is 2 mV/s in the area of the anodic and cathodic branches in a potential range encompassing the corrosion potential from -700 mV to -300 mV. The electrolyte solution is a hydrochloric acid solution (18.5%). The temperature is fixed at T= 80 °C. The latter is equipped with a lid with five openings: three for the three electrodes, one for the thermometer, and possibly one to control the atmosphere above the solution. Before each series of experiments, the electrode is immersed in the solution for 10 min under agitation (Saouti et al. 2021).

2.4 SEM analysis

The microstructure and the morphology were analyzed by scanning electron microscopy (SEM) using Quattro S, FEG from FEI Company at 20 KV. The observation allows seeing in detail the texture and the morphological structure of the inhibitor used in this study (shape, size, and arrangement of its organics constituents on the surface).

3. Results and discussion

3.1 Gravimetric measurement

The analysis of the obtained results showed that the mass loss Δm is equal to 0.548 g after 6h of immersion in the acid solution in the absence of inhibitor. While it is equal to 0.051 g in the presence of the inhibitor with a concentration of C = 0.6 g/L. This result is justified that the corrosion rate decreased with the increase in the concentration of prop-2-yn-1-ol, methyloxirane, and the inhibition was enhanced, as displayed in **Table 1** and consequently an increase in the effectiveness of the inhibitor which reaches a maximum of 92.70% (Bouchtart *et al.* 2020; Ji *et al.* 2015; Zarrok *et al.* 2012). This confirms that prop-2-yn-1-ol, methyloxirane molecules are relatively well adsorbed on the mild steel surface.

Table 1: Changes in the mass of the steel sample during the gravimetric tests.

C(g/L)	mi (g)	m_f (g)0.3	Δm (g)	EI (%)
0	112.285	111.737	0.548	0
0.3	111.602	111.467	0.135	75.36
0.6	111.602	111.551	0.051	90.69
0.9	111.551	111.500	0.051	90.69
1.2	111.500	111.460	0.04	92.70

In addition, the mass loss decreases to 0.040 g with a positive effect of adding the inhibitor up to a concentration of 0.6 g/L. For a concentration above this value, the mass loss remains almost constant. For a double concentration (1.2 g/L) the mass loss is 0.04 (a decrease of 0.01 g). We also note that the efficiency reaches a threshold value of 90% for a concentration of 0.6 g/L (figure 2).

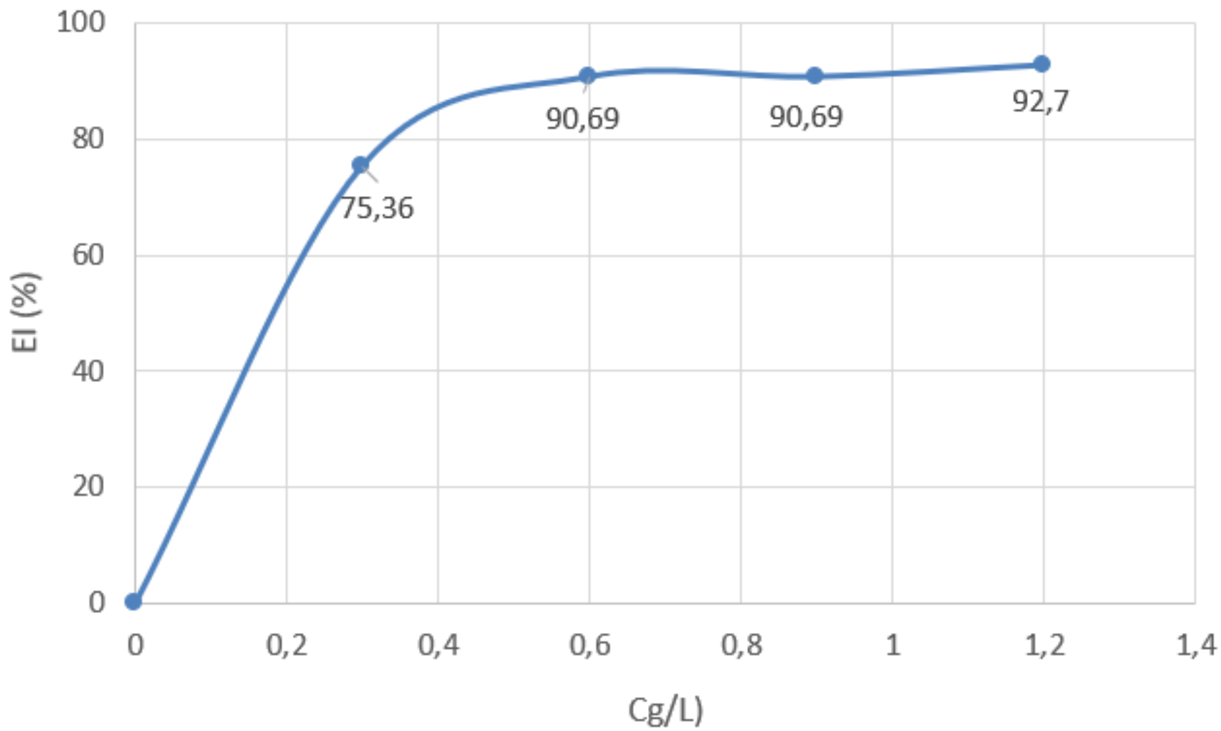

Figure 2: curve of variation of the inhibitory efficacy of the inhibitor with prop-2-yn-1-ol, methyloxirane as a function of the concentration

3.2 Electrochemical measurement

The anodic and cathodic potentiodynamic polarization curves of mild steel in the absence and presence of different inhibitor concentrations in (18.5%) HCl acid medium are shown in Figure 3. The first analysis of these curves shows that the anodic and cathodic reactions are affected by the addition of the inhibitor. Indeed, the presence of the inhibitor even at a very low concentration (0.3g/L) causes a shift of the corrosion potential towards positive values (from -550 mV to -480 mV) (Bentiss *et al.* 2012; Tourabi *et al.* 2013). The addition of the inhibitor in the HCl solution induces the decrease of the anodic current corresponding to the attack of the metal and the decrease of the cathodic current corresponding to the reduction of oxygen (Table 2) (Ech-chihbi *et al.* 2019; Yıldız 2019). Moreover, the effectiveness of the electrochemical inhibitor (%) is defined as follows:

$$EI\ (\%) = \frac{j_0^{Corr} - j^{Corr}}{j_0^{Corr}} \times 100 \qquad (2)$$

where j_0^{Corr} and j^{Corr} are the corrosion current density values of the steel in the absence and presence of the inhibitor, respectively.

The electrochemical inhibitory efficiency of the inhibitor shows several advantages based on the concentration studied. The lowest concentration examined, which is 0.3 g/L equivalent to 0.00262 mol/L, exhibits a relatively low inhibitory efficiency. However, as the concentration of the inhibitor increases, the inhibitory efficiency improves significantly. At a concentration of 1.2 g/L, the inhibitory efficiency reaches a maximum value of 91%. Moreover, through the analysis, it is found that the optimal concentration of the inhibitor is 0.6 g/L equivalent of 0.00525 mol/L. This concentration provides a satisfactory inhibitory efficiency, as doubling the concentration to 1.2 g/L equivalent of 0.01050 mol/L

only results in a marginal 4% increase in electrochemical efficiency. Therefore, the additional amount of inhibitor added to double the concentration does not provide a significant improvement in inhibitory performance, considering the potential costs and practical considerations associated with higher inhibitor dosages.

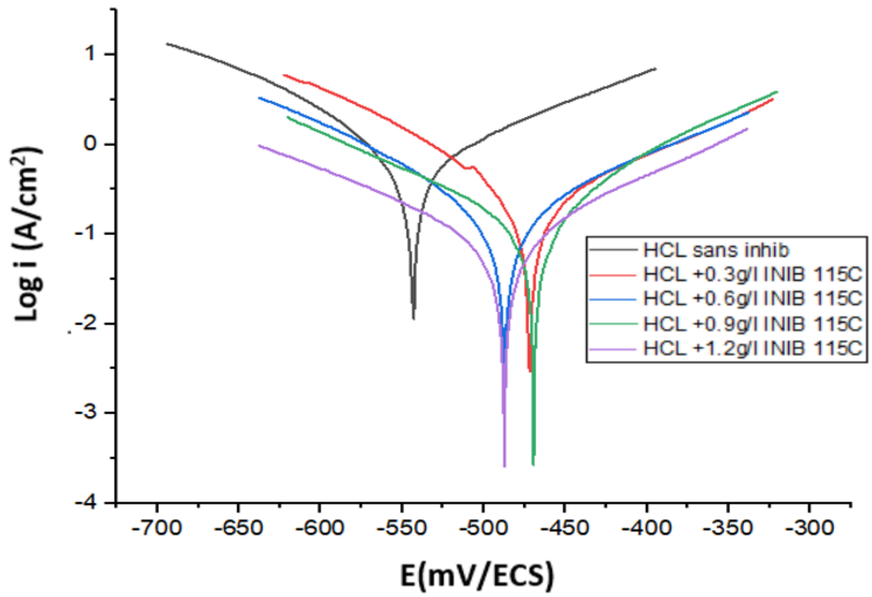

Figure 3: Polarization curves of steel in HCl medium without and with prop-2-yn-1-ol, methyloxirane. log i (E)

Table 2: electrochemical parameters obtained from polarization curves for different inhibitor concentrations

C(g/L)	E(mV)	j_{cor} ($\mu A/cm^2$)	R_p ($\Omega.cm^2$)	β_a (mV)	β_c (mV)	EI/j_{cor} (%)
0	-543.4	1.1432	30.26	192.9	-136.2	--
0.3	-471.9	0.2241	89.89	135.1	-93.0	80
0.6	-487.3	0.1388	139.82	123.2	-99.7	87
0.9	-469.4	0.1149	144.68	80.3	-121.4	89
1.2	-487.6	0.0975	273.22	131.2	-150.8	91

The presence of the prop-2-yn-1-ol, methyloxirane molecules play simultaneous effect to create a barrier at the metal surface against the arrival of both H^+ and dissolved oxygen in aerated acid solution. We can introduce the synergistic intermolecular effect to protect steel in the chemical pickling (18.5% HCl) via the presence of oxygen and triple bond as well as hydroxyl group (Zarrok *et al.* 2013, Loto & Tobilola (2018); Toghan *et al.* 2023).

3.3 Examination of the steel surface

Fig.5 shows photos of these different samples, each strip in an HCl solution (18.5%) containing a different value of the inhibitor concentration. From this image, we notice by examination, a remarkable improvement of the samples aspect by comparing the pickled piece in the absence (dark aspect) to those pickled in presence of the inhibitor (clear and bright aspect).

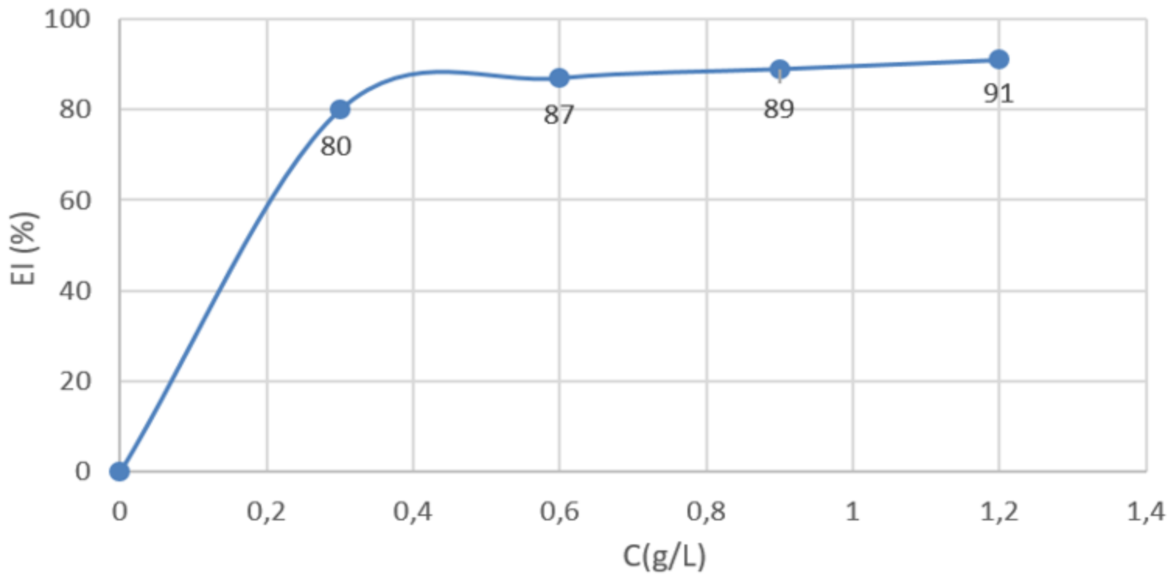

Figure 4: curve of variation of the inhibitory efficacy of the inhibitor with prop-2-yn-1-ol, methyl oxirane as a function of the concentration

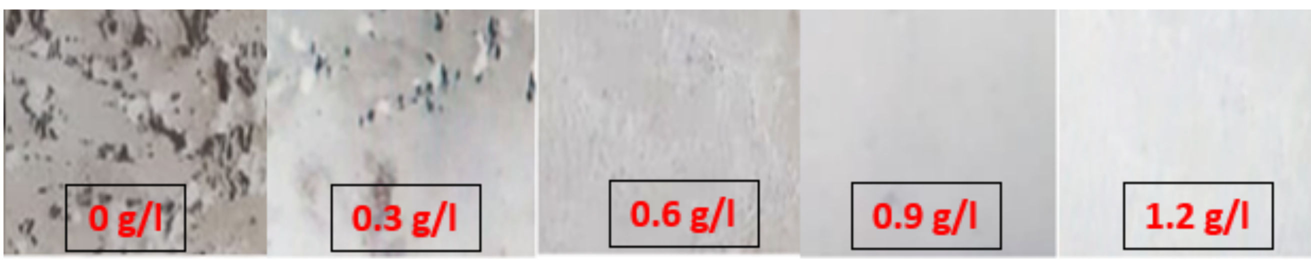

Figure 5: Samples immersed in HCl solutions (18.5%) at different concentrations of the inhibitor.

3.4 SEM analysis

Figure 6 shows the SEM images of the corroded and uncorroded mild steel surfaces without and with the prop-2-yn-1-ol, methyloxirane. The results obtained by SEM for the samples of the steel after chemical pickling in HCL medium in the absence of inhibitor, allow identifying the type of corrosion, indicating pitting corrosion and a damaged state due to some cracks scanned on the surface (Fig.6b). On the other hand, in the presence of the inhibitor, no attack on the surface of the steel was observed, which shows good protection of the steel against corrosion due most probably to the formation of a protective layer on the surface (Fig.6a) (Eddy et al. 2014; Hanoon et al. 2020;). The analysis of two SEM (Scanning Electron Microscopy) images showing steel attacked by hydrochloric acid and another that is not attacked in the presence of an inhibitor can help to understand the effects of inhibitors on steel corrosion. The image of the steel attacked by hydrochloric acid shows a rough and irregular metal surface, indicating that corrosion has occurred. One can also observe the formation of cavities, cracks, and deposits on the metal surface, indicating that corrosion has altered the metal structure.

On the other hand, the image of the steel in the presence of an inhibitor shows a smoother and less altered surface, indicating that corrosion has been slowed down or inhibited by the action of the inhibitor. One

can also observe a reduction in the formation of cavities, cracks, and deposits on the metal surface, suggesting that the inhibitor effectively protected the metal structure.

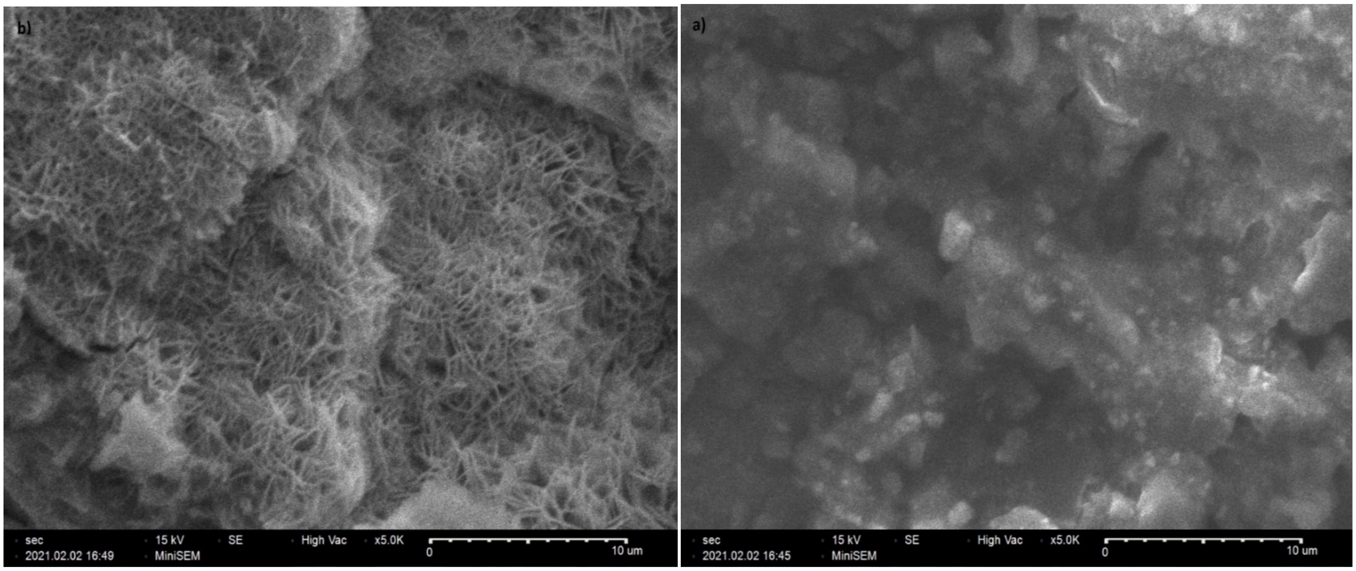

Figure 6: Scanning electron microscope (SEM) photos of the mild steel surface after 5 min immersion in HCl medium, (a) in the presence of 0.6 g/l of the inhibitor, (b) in the absence of the inhibitor.

The SEM micrograph also shows the development of a thick layer of corrosion products, relating to the iron oxides formed. This layer shows a porous structure, in fact, the presence of pits and crevices in the iron oxide layer leads to the formation of channels that allow the diffusion mainly of H^+ ions as well as Cl^- anions and water molecules from the HCl solution (corrosive medium). We are therefore faced with a degradation of the surface state of the alloy in question (steel) through the intense dissolution of iron by the attack of H^+ ions and the consequent release of Fe^{2+} ions. SEM micrographs reveal an improvement in surface condition, i.e. the absence of heterogeneities (pitting) in the morphology of the steel's metallic surface in the presence of corrosion inhibitors. This is due to the formation of a protective layer on the metal surface as a result of physiosorption and essentially chemisorption of the inhibitor molecules on the steel's metal surface. Corrosion inhibitors are substances that reduce or prevent corrosion by modifying the chemical environment around the metal surface. They can act by forming a protective layer on the metal surface, neutralizing corrosive acids, or regulating electrochemical reactions at the metal surface.

In conclusion, the SEM images clearly demonstrate the protective effect of corrosion inhibitors on steel and can help to evaluate the effectiveness of different inhibitors for industrial applications.

Conclusion

In (18.5%) hydrochloric acid solution, the produced inhibitor (Prop-2-yn-1-ol, methyloxirane) showed excellent inhibitive activity for mild steel corrosion. The substance has demonstrated remarkable inhibition efficiency, and its inhibitory characteristics are stronger as concentrations increase. In a hydrochloric acid solution, it works as an inhibitor with anodic predominance for mild steel. The polarization curves indicate that Prop-2-yn-1-ol, methyloxirane acts as a anodic-type inhibitor.

According to SEM examination, metal is successfully shielded from the HCl aggressive solution by a protective coating, which is the cause of Prop-2-yn-1-ol, methyloxirane inhibitory mechanism.

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