Study on Copper Protection in H_2S-rich Marine Environments using SAMs from *Sargassum* Extract

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Abstract: Corrosion is a complex and progressive degradation process that occurs when an aggressive external agent attacks a metal and is known to cause significant harm to materials, the environment, and metals. One important factor in these physicochemical phenomena is the atmosphere. Factors like temperature, humidity, and salinity are relevant. For most materials, the marine environment is the ideal example of an environment that is naturally aggressive. Because few metals can resist it, they must be protected. For instance, when copper corrodes in an alkaline environment, copper oxide forms a protective film on the metal's surface, reducing its ability to attack. Nevertheless, the metal eventually deteriorates because of the instability of the surrounding environment, so this solution is only temporary. These days, plants-based inhibitors represent a real source of robust, long-lasting protection. They can function as natural inhibitors of metals and their alloys due to their very beneficial natural characteristics. Therefore, the focus of this work is on how *Sargassum fluitans III* self-assembled monolayers (SAMs) inhibit copper corrosion in coastal environments. When copper is submerged directly in the inhibiting solution, the extract successfully prevents copper corrosion, according to the findings of surface analysis and electrochemistry. In this article, for example, it was demonstrated that sargassum extract halves the loss of copper thickness, reducing it from 8.00±0.61 μm to 4.76±0.52 μm on a site heavily exposed to sea salts.

Keywords: Corrosion, Copper, *Sargassum fluitans III*, Self-assembled monolayers (SAMs).

1. INTRODUCTION

Metal and alloy corrosion is a global problem that threatens industrialized nations' economies and results in significant material losses. Due to its high electrical and thermal conductivities, copper is a noble metal that is used effectively in a variety of applications, including construction, electronic and electrical components, and communication [1]. The electrochemical process of atmospheric corrosion of copper is primarily influenced by the atmosphere's relative humidity and the presence of pollutants. In fact, the atmosphere's water vapor may condense and create a liquid layer on the surface, which will facilitate the interaction of the metal with the corrosive pollutant [2, 3]. Numerous studies have been conducted on the atmospheric corrosion of copper in various parts of the world [4-13]. According to these studies, the copper corrosion layer is initially made up of Cu_2O cuprite or a Cu_2O/CuO duplex (CuO tenorite), regardless of the type of atmosphere—rural, urban, or marine [14, 15]. $Cu_2Cl(OH)_3$, which exists in multiple crystalline forms, first forms on Cu_2O in a purely marine atmosphere that is rich in chlorides as copper chloride CuCl (nantokite) [13, 16, 17]. Copper typically corrodes at a rate of a few µm per year on average. Pollutants in the atmosphere, however, have the power to alter the corrosion layer's behavior and quicken the corrosion process [18]. Previous studies have in fact shown that the atmosphere of Martinique (French Caribbean island) is extremely corrosive to metals, mainly due to the presence of H_2S generated by the decomposition of Sargassum algae [19, 20]. This research revealed that H_2S has a considerable impact on the kinetics and morphology of copper corrosion. A majority formation of CuS was observed, as well as unprecedented copper corrosion rates in excess of 325 µm/year [21].

Many methods have been developed to combat corrosion. These include the so-called passive technique, which consists of isolating the metal from the corrosive environment by means of metallic coatings (paint, varnish, etc.), non-metallic coatings and/or protective oxides [22-25]. The other so-called active method used in this study (SAMs) involves the use of inhibitors that act directly on the corrosive medium. They are classified into two sub-categories: active methods (very energetic), which include electrochemical techniques, and passive methods (requiring a lot of raw materials), which include coating techniques and corrosion inhibitors. Another coating method involves the use of organized molecular assemblies (SAMs), obtained by adsorption of an active surfactant onto a solid surface. Langmuir and Blodgett have documented the physical adsorption of organic molecules onto metal surfaces. The simplicity of preparation and spontaneous formation of these monolayers at the interface make the SAMs process particularly attractive. Organic molecules commonly used to inhibit metal corrosion include alkaloids and phenolic compounds, among others [26-28]. These

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compounds, derived from plants, constitute an inexhaustible source of organic molecules [29].

Depending on the season, *Sargassum* can be accessed and found in large quantities. These brown algae have the potential to be a significant and promising source of organic molecules with inhibitory effects. *Sargassum fluitans III* and *Sargassum natans* are the two species of algae in the *Sargassum* genus that have been found along the coast of Martinique. Utilizing corrosion inhibitors made from *Sargassum* algae would allow for a novel method of enhancing *Sargassum*'s value while simultaneously addressing a rapidly expanding field of study that is the focus of several publications every year [30, 31]. In previous studies, the crude extract of *Sargassum fluitans III* has been shown to significantly inhibit the corrosion of carbon steel in immersion [32] and of zinc in atmospheric exposure, thanks to the application of a self-assembled layer (SAM) process [20].

This study investigated the potential protective effect of crude *Sargassum fluitans III* extracts on the copper surface, using self-assembled layers (SAMs). The extracts were tested in two distinct marine atmospheres of Martinique: the atmosphere of Diamant, rich in chloride ions, and that of Frégate-est, characterized by a high concentration of H_2S due to extensive Sargassum strandings.

Mass loss analysis was used to assess the efficacy of *Sargassum* extracts and X-ray diffraction (XRD) analyses provided a detailed description of the composition of the corrosion surface. In addition, electrochemical impedance spectroscopy (EIS) was also used to identify the elementary corrosion mechanisms acting on the copper surface.

2. MATERIALS AND METHODS

2.1. Samples Preparation

The copper samples studied are 99% pure. They measure 100 × 75 × 1 mm. Before any treatment, the samples were chemically stripped by immersion in a 10% sulfuric acid bath for a few seconds, then mechanically polished with SiC up to grade 1200, rinsed with distilled water and ethanol, dried, and weighed to the nearest 10^{-4} g using an Adam Nimbus 210-001 microbalance.

After this step, the samples were immersed in a solution of crude extract of *Sargassum fluitans III* obtained by refluxing with a mixture of 50% ethanol and distilled water. The preparation protocol for this extract was described in a previous study by Lambert *et al.* [32]. The copper samples were immersed for 72 hours in the crude extract of *Sargassum fluitans III*. The samples were then exposed at the two selected sites on inclined racks at 45°, in accordance with the EN13523-19 standard, for 3 months. A triplicate of samples was exposed at each site, with or without coating.

2.2. Selection of Sites

The locations of the two sites (Frégate Est and Diamant) selected are shown on a map of Martinique in Figure **1**. The climatic characteristics of the two sites studied are similar and have been described in previous studies [21, 33]. These sites were chosen because they illustrate two types of marine atmosphere present in Martinique:

- An atmosphere rich in Cl- ions and poor in H_2S (Diamant)
- An atmosphere rich in H_2S and low in Cl- ions (Frégate Est).

This difference can be explained by different exposure to Sargassum algae strandings, which release large quantities of H_2S during their biodegradation. Table **1** summarises the average concentrations of H_2S and Cl- measured over the three months of exposure.

Figure 1: Location of the various locations on the Martinique map.

Table 1: Average H_2S and Cl^- ion Concentrations over a Three-Month Period at Each Location

Sites	H_2S(ppb)	Cl^-deposited ($mg.m^{-2}.day^{-1}$)
Diamant	5	470
Frégate Est	2590	56

2.2. Gravimetric Analysis

To remove corrosion products, copper samples are treated chemically in accordance with the NF EN 8407 standard [34]. Prior to chemical treatment, the surfaces of the samples are gently brushed to eliminate loosely adhering corrosion products. The samples are then immersed in a sulfuric acid solution (100 mL in 900 mL of distilled water) for 1 to 3 minutes at a temperature of 20-25°C. To enhance the effectiveness of the treatment, an ultrasonic bath is utilized. After the treatments, the samples are air-dried and subsequently rinsed with distilled water and ethanol before being weighed to assess mass loss. This procedure is repeated multiple times for each sample until a consistent mass plateau is achieved. Mass loss is determined for each exposure site using a triplet of samples. The thickness loss is deduced from these measurements.

2.3. Measurements Involving Electrochemistry and XRD Analysis

The smaller 25×20 mm coupons were subjected to electrochemical testing and XRD analysis. A Bio-Logic VMP3 potentiostat was connected to a three-electrode setup for the purpose of conducting electrochemical measurements. The working electrode was the copper plate that had been previously exposed. The reference electrode in this experiment was a saturated $Hg/Hg_2Cl_2/KCl$ calomel electrode, while the counter-electrode was a platinum electrode. The open circuit potential (OCP) was measured as a function of time for every test. The value noted matched the working electrode's corrosion potential (E_{corr}) once the medium had stabilized. Signals with an amplitude of ±2.5 mV were used for electrochemical impedance spectroscopy (EIS) measurements, with a scanning frequency ranging from 100 kHz to 10 mHz. The scanning speed used to record the polarization curves (LPR) was 10 mV/min. The experiments were conducted in duplicate for each concentration that was tested, and EC-Lab V11.43 software was used to analyze all the data that was gathered.

The diffractometer used to identify the copper corrosion products is a PANalytical Empyrean, consisting of an X-ray source, a sample holder, an X-ray detector, and an acquisition system. The sample is fixed, while the source and the detector rotate around it, each following a 2θ angle. The radiation used is that of copper ($\lambda K\alpha 1 = 1.5408$ Å), and the detector employed is a gas scintillator, as described in the previous studies [20, 21, 33].

3. RESULTS AND DISCUSSION

3.1. Evaluation of Copper Thickness Loss

The corrosion kinetics of copper exposed at the two sites studied, with and without a coating of *Sargassum fluitans III* crude extract, were assessed by mass loss measurements. In order to better visualise the damage, this mass loss was converted into thickness loss (ε) using the following equation (1).

$$\varepsilon = \frac{10\times\Delta m}{Sd} \tag{1}$$

Where ε is expressed in µm; Δm is the lost mass (in grams); d is the density of the copper (8.94 g/cm^3), and S is the surface area of the copper (in cm^2).

Table **2** presents the average thickness losses of copper samples exposed for three months at the two sites. With and without coating, the thickness losses recorded at Diamant and Frégate Est are 8.00 µm, 4.76 µm, 110.00 µm, and 57.23 µm, respectively. Notably, the thickness losses at Frégate Est are significantly higher. This is attributed to the high concentration of H_2S in the atmosphere, resulting from the degradation of *Sargassum* algae, which contributes to the corrosive and aggressive nature of the atmosphere [33, 35].

In contrast, corrosion at Diamant is primarily linked to an atmosphere rich in sea spray laden with chloride ions. Furthermore, the presence of the coating on the copper surface reduced thickness losses by half in both environments. These findings indicate that *Sargassum* extracts provide a protective effect on copper against corrosion, independent of the surrounding corrosive conditions. This suggests that the corrosion inhibition properties of *Sargassum* extracts extend beyond specific environmental contexts.

Phytochemical analyses conducted by Lambert *et al.* [32] on crude extracts of *Sargassum fluitans* III revealed the presence of several chemical families known for their corrosion-inhibiting properties. Among these compounds were coumarins, anthocyanins, quinones, flavonoids, saponins, tannins, and triterpenes. These molecules play a key role in the formation of self-assembled monolayers (SAMs) on the copper surface, thereby enhancing its corrosion resistance.

The atmospheric conditions at Frégate Est and Diamant are classified as extremely corrosive, surpassing the upper limit of category CX according to the NF EN ISO 9223 standard [34]. However, the application of *Sargassum fluitans III* extract lowered the corrosiveness level in Diamant from class CX to class C5.

A statistical analysis was carried out on the gravimetric data to evaluate the significance of the differences observed in thickness loss. The results showed that the reduction in thickness loss for the coated samples was statistically significant ($p < 0.05$) at both exposure sites, with confidence intervals of ±0.3 µm for Diamant and ±2.5 µm for Frégate Est. These findings confirm the reliability of the protective effect provided by the *Sargassum* extract.

Table 2: Copper Thickness Loss Following Two Months of Exposure. Multiple Samples were Exposed for Every Site. The Numbers Match the Means of the *Triplicate Batches that were Exposed*

Sites	Surface State	Loss of Thickness (µm)
Diamant	Without coating	8.00
	With coating	4.76
Frégate Est	Without coating	110.00
	With coating	57.23

3.2. Electrochemical Results

Gravimetry research provides only a limited amount of information about the mechanisms underlying corrosion. Figure **2** displays the Nyquist diagrams of copper samples exposed to the atmosphere at Frégate-est and Diamant for three months, both with and without SAM treatment. Because of the interfacial impedance frequency dispersion caused by the heterogeneity of the copper electrode surface, the Nyquist plots do not perfectly form semicircles. The constant-phase element (CPE) (α, Q) accounts for surface inhomogeneity through the coefficient α, which ranges from 0 to 1. The impedance associated with the CPE is provided in references [22-24]. These graphs show a single semicircular capacitive loop, indicating the presence of a single phenomenon related to the copper corrosion products formed in the case of untreated copper and to the film formed in the case of copper treated with the extract/SAM. For the copper electrodes treated with extract/SAM, the impedance of the electrode modified with the Sargassum extract is significantly higher than that of the bare, untreated electrode.

In fact, the size of the capacitive loop in electrochemical impedance spectroscopy (EIS) indicates how well the material in question protects against corrosion. Larger capacitive loops are typically indicative of better corrosion resistance. Because of the self-assembled extract/SAM layers, the copper electrodes modified by extract/SAM in this instance have larger capacitive loop sizes than the bare electrode, suggesting improved anticorrosion protection. This finding contributes significantly to the advancement of material science research by highlighting the potential of utilizing *Sargassum* extract in the SAM process to reduce atmospheric corrosion and increase the durability of copper-based materials. The experimentally obtained impedance data were modeled using the simple Randles equivalent circuit, incorporating a constant-phase element (CPE), Figure **3**. The low values of the error factor (χ^2) validate the applicability of the proposed equivalent electrical circuit (EEC). The electrochemical parameters of both bare and SAM-modified zinc were extracted through parametric fitting of the Bode and Nyquist diagrams and are summarized in Table **3**. The analysis of Table **3** shows that the resistance increases in the presence of the treatment, indicating an improvement in the copper's resistance. The parameter α increases with the inhibitor concentration, suggesting a reduction in the electrode surface heterogeneity. On the other hand,

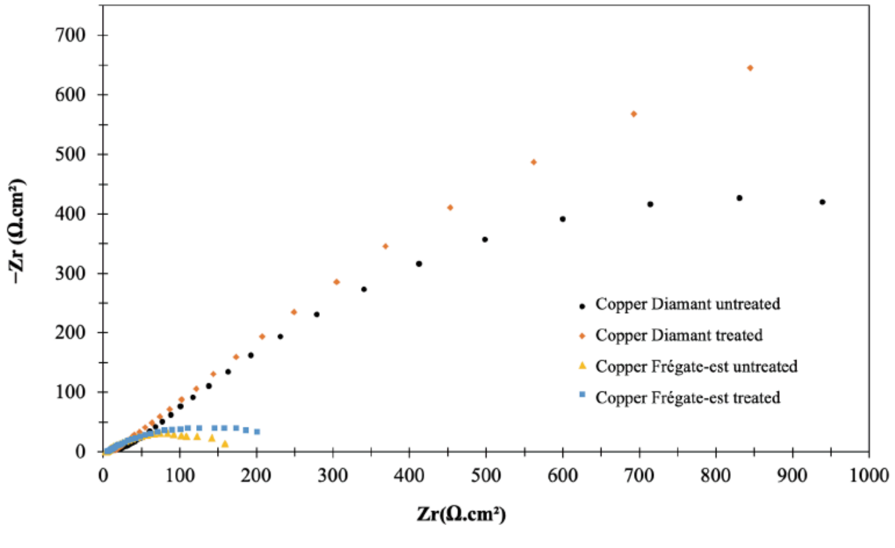

Figure 2*:* Nyquist plots in NaCl were produced for copper samples exposed in Diamant and Frégate-est for three months, both with and without Sargassum extract/SAM modification.

the values of Q decrease. These results are consistent with those obtained in the evaluation of copper thickness loss.

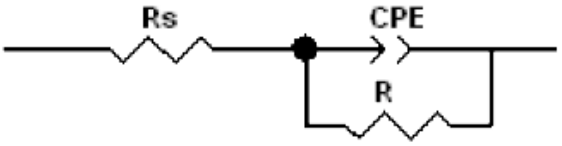

Figure 3: The equivalent circuit used to fit the impedance data.

3.3. XRD Results

To supplement the results obtained by gravimetry and to better understand the corrosion mechanisms, we performed XRD analyses. Figure **4** shows the diffractograms obtained after exposing copper with (b, d) and without (a, c) coating for 3 months in Diamant (a, b) and Frégate Est (c, d). In Diamant, the corrosion surface is mainly composed of Cu_2O and $Cu_2Cl(OH)_3$. These results can be explained by the proximity of this site to the shore, where average chloride concentrations are high. Indeed, these corrosion products are typically observed during atmospheric marine corrosion [36]. In both cases, with (Figure **3b**) or without (Figure **3a**) coating, no significant difference in the corrosion products is observed at this site. Additionally, metallic copper is detected in both cases, indicating that at certain points, the corrosion layer is thinner than the analysis depth of approximately 1 µm. However, the peaks associated with metallic copper are much more intense when the surface is coated (Figure **3b**). These results indicate that the presence of a crude extract coating from *Sargassum fluitans III* on the surface has a slowing effect on the formation of corrosion products.

These observations are consistent with the lower corrosion rates observed for the coated copper (Figure **3**). The corrosion products formed are the result of electrochemical corrosion initiated by the formation of an aqueous layer on the metal surface due to high humidity [37]. This leads to the anodic dissolution of copper and the reduction of dissolved oxygen as described by equations (2) and (3). Specifically, Cu_2O forms spontaneously through the reaction of Cu^+ and OH^- as described by equation (4).

$$Cu \rightarrow Cu^+ + e^- \quad (2)$$

$$O_2 + 2H_2O + 4e^- \rightarrow 4OH^- \quad (3)$$

$$2Cu^+ + 2OH^- \rightarrow Cu_2O + H_2O \quad (4)$$

Subsequently, Cu^+ is converted into CuCl (nantokite) and a soluble chloride complex, $CuCl_2^-$, as described by equations (5) and (6).

$$Cu^+ + Cl^- \rightarrow CuCl \quad (5)$$

$$Cu^+ + 2Cl^- \rightarrow CuCl_2^- \quad (6)$$

The oxidation of these two compounds in the presence of dissolved oxygen leads to the formation of basic copper chlorides $Cu_2Cl(OH)_3$, as shown in equations (7) and (8).

$$3\mathrm{CuCl} + 3/4\,\mathrm{O_2} + 3/2\ \mathrm{H_2O} \rightarrow \mathrm{Cu_2(OH)_3Cl} + \mathrm{Cu^{2+}} + 2\mathrm{Cl^-} \quad (7)$$

$$3\mathrm{CuCl_2^-} + 3/4\,\mathrm{O_2} + 3/2\,\mathrm{H_2O} \rightarrow \mathrm{Cu_2(OH)_3Cl} + \mathrm{Cu^{2+}} + 5\mathrm{Cl^-} \quad (8)$$

In Frégate Est, XRD analyses reveal that copper sulfide (CuS) is the major product, while Cu_2O constitutes only a minor portion, both in the presence (Figure **3d**) and absence (Figure **3c**) of coating. This observation aligns with the high concentrations of H_2S measured at this site. Indeed, the formation of CuS results from the reaction of Cu^{2+} ions with S^{2-} ions originating from the two-step dissociation of H_2S [38, 39]. The proposed process is described in equations (9) to (12).

$$Cu \rightarrow Cu^{2+} + 2e^- \quad (9)$$

$$H_2S \rightarrow H^+ + HS^- \quad (10)$$

Table 3: Parameters Deduced from EIS Data Obtained in 3% NaCl at 25 °C for Copper Surfaces without and with Modification of Sargassum Extract/SAMs

Site	Diamant		Frégate Est	
	Without SAMs	**With SAMs**	**Without SAMs**	**With SAMs**
Rs (Ω.cm^2)	3	7	5	5
R(Ω.cm^2)	1014	2115	185	264
α	0.7002	0.8632	0.7316	0.7844
$Q_2 10^{-4}$ (Ω^{-1}cm^{-2}s^{n2})	0.38	0.21	0.38	0.24

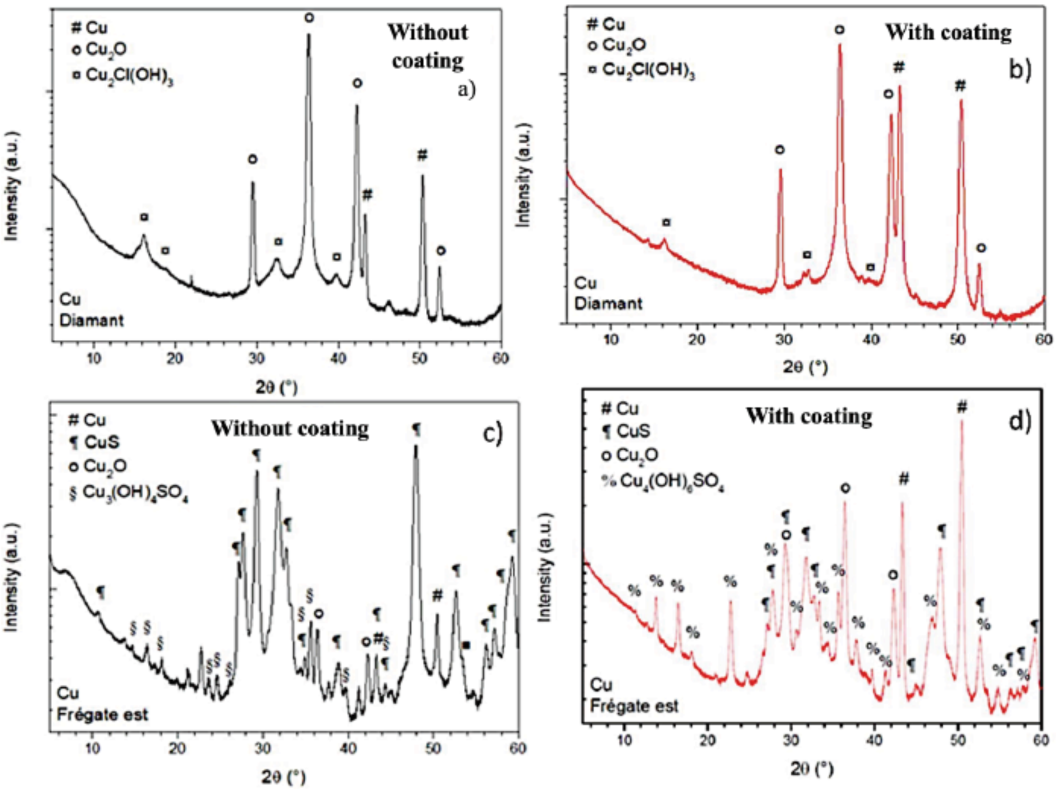

Figure 4: XRD diffractograms of copper samples exposed for 3 months in Diamant and Frégate Est, with (b,d) and without (a,c) coating based on crude extracts of *Sargassum fluitans III* algae.

$$2HS^- \rightarrow 2S^{2-} + 2H^+ \quad (11)$$

$$Cu + H_2S \rightarrow CuS + H_2 \quad (12)$$

It should be noted that the surface composition differs in the two cases. Indeed, in the absence of coating, there is a significant presence of antlerite ($Cu_3(OH)_4SO_4$) (Figure **3c**). Conversely, in the presence of coating, antlerite is replaced by brochantite ($Cu_4(OH)_6SO_4$) (Figure **3d**). These two hydrosulphates are formed over the medium term in atmospheres rich in sulphur compounds [40] and are known for their relatively similar structure. The formation of these products is favoured by the decrease in pH at the copper surface due to the formation of sulfuric acid (H_2SO_4) resulting from the oxidation of H_2S by dissolved O_2 [41], leading to the dissolution of copper as Cu^{2+} ions. These ions react with the sulphates and hydroxides present in the aqueous layer to form antlerite [16] and brochantite [17] as described in equations 13 and 14.

$$3Cu^{2+} + SO_4^{2-} + 4OH^- \leftrightarrows Cu_3(OH)_4SO_4 \quad (12)$$

$$Cu^{2+} + SO_4^{2-} + 6OH^- \rightarrow Cu_4SO_4(OH)_6 \quad (13)$$

This analysis reveals that the presence of a coating modifies the composition of the corrosion surface. Indeed, the phytochemical screenings conducted by Lambert *et al.* [32] on crude extracts of *Sargassum fluitans III* algae showed that these extracts contain various chemical families, such as coumarins, anthocyanins, quinones, flavonoids, saponins, tannins, and triterpenes, which have corrosion-inhibiting properties. The presence of these families could therefore explain why brochantite appears instead of antlerite. Indeed, the formation of brochantite is induced by the high pH of the surface [42]. In our case, the appearance of brochantite can be explained by the presence of a coating based on extracts from *Sargassum fluitans III* algae, which likely acts by limiting the dissolution of H_2S on the metal surface, thereby forming a barrier at the interface between the electrolytic environment and the metal surface. This leads to a decrease in the acidity of the environment and promotes the formation of basic corrosion products such as brochantite.

Gravimetric measurements, together with surface characterizations by SEM and XRD analyses, confirmed the protective effect of *Sargassum fluitans* III extract, supporting its efficiency as a corrosion inhibitor. The use of *Sargassum fluitans* III extract as a corrosion inhibitor represents a sustainable and economically viable alternative to conventional synthetic inhibitors. In Martinique, *Sargassum* algae are abundant and readily available, particularly during stranding seasons, which significantly reduces raw material procurement costs. The extraction process, employing a 50%

ethanol–distilled water mixture, is relatively simple and cost-effective. Compared to conventional inhibitors, the use of *Sargassum* extract not only provides environmental benefits but also reduces reliance on expensive and potentially hazardous chemicals. This makes it a promising candidate for large-scale industrial applications, especially in regions affected by *Sargassum* strandings.

4. CONCLUSIONS

The study on the formation of self-assembled monolayers (SAMs) based on *Sargassum* extract has led to the following important conclusions:

- Following treatment with the extract-based coating, there was a notable decrease in the loss of copper thickness, as indicated by the mass loss measurements that were taken three months later at two distinct locations. The protective film's significant reduction of the corrosion effects was exhibited by the formation of SAMs, enhancing copper's durability and performance.
- Electrochemistry has been used to verify these protective qualities by comparing the impedance curves of copper samples with and without coating. When compared to the uncoated material, the coated copper curves indicated increased impedance, suggesting a more effective corrosion barrier.
- The presence of compounds such as brochantite revealed by XRD shows the action of *Sargassum fluitans III* extract on the copper surface. The resulting coating modification induces the formation of a protective layer on the metal thanks to the *Sargassum* extract.

AUTHOR CONTRIBUTIONS

Conceptualization, M.L.; methodology, P.L., M.S.-a., M.L., B.L. and S.R.; software, P.L. and M.S.-a.; validation, M.L.; formal analysis, M.S.-a., M.L., B.L., S.R. and P.L.; investigation, P.L., M.S.-a., M.L., B.L. and S.R.; resources, M.L.; data curation, P.L., M.S.-a. and M.L.; writing—original draft preparation, P.L., M.S.-a. and M.L.; writing—review and editing, P.L., M.S.-a. and M.L.; visualization, M.S.-a. and M.L.; supervision, M.S.-a. and M.L.; project administration, M.L.; funding acquisition, M.L. All authors have read and agreed to the published version of the manuscript.

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DATA AVAILABILITY STATEMENT

The raw data supporting the conclusions of this article will be made available by the authors on request.

CONFLICTS OF INTEREST

The authors declare no conflicts of interest.

DISCLAIMER/PUBLISHER'S NOTE

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