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The Green Inhibitive Effect of *Ptychostomum schleicheri* (Bryophyta) Extract on Mild Steel Corrosion

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Abstract
In present study, the inhibitive impact of *Ptychostomum schleicheri* extract, a moss species of Bryaceae family, on the mild steel corrosion in 1 M HCl at ambient temperature was firstly analysed by Tafel extrapolation method, electrochemical impedance spectroscopy (EIS) and linear polarization (LPR) techniques. Electrochemical tests were conducted by 1 h immersing mild steels in HCl solutions containing four various concentrations of moss extract. At a concentration of 0.200% (w/v), the moss extract displayed around 90 percent inhibition efficiency by three electrochemical methods, and it was also observed that its protective effect increased depending on the upsurge in the concentration of *Ptychostomum schleicheri* extract. The morphologies of mild steels surface in 1 M HCl with and without *P. schleicheri* extract at the end of 1 h immersion time were visualized by a metal microscope. Finally, according to the electrochemical data and surface morphology results, it can be said that the extract molecules adsorbed on the mild steel surface to achieve almost maximal coverage and contact.
Keywords: Bryaceae, *Ptychostomum schleicheri*, Eco-friendly inhibitor, Corrosion, Metal microscope.

Ptychostomum schleicheri (Bryophyta) Ekstraktının Yumuşak Çeliğin Korozyonu Üzerine Yeşil Önleyici Etkisi

Öz
Bryaceae familyasının bir yosun türü olan *Ptychostomum schleicheri* ekstraktının 1 M HCl içerisindeki yumuşak çeliğin korozyonu üzerine önleyici etkisi, oda sıcaklığında Tafel ekstrapolasyon yöntemi, lineer polarizasyon (LPR) ve elektrokimyasal impedans spektroskopisi (EIS) yöntemleri ile ilk kez, mevcut çalışmada analiz edilmiştir. Elektrokimyasal deneyler, dört farklı derişimde yosun ekstraktı içeren HCl çözeltilerine, yumuşak çeliklerin 1 saat daldırılması ile gerçekleştirilmiştir. % 0,200 (w/v) derişiminde yosun ekstraktı, üç elektrokimyasal yöntemle yüzde 90 civarında inhibisyon etkinliği göstermiş ve ayrıca *Ptychostomum schleicheri* ekstraktının derişiminin artmasına bağlı olarak, koruyucu etkisinin de arttığı gözlemlenmiştir. Yosun ekstraktı içeren ve içermeyen 1 M HCl içerisindeki yumuşak çeliklerin yüzey morfolojileri, 1 saatlik daldırma süresinin sonunda metal mikroskobu yardımıyla görüntülenmiştir. Son olarak, elektrokimyasal veriler ve yüzey morfolojisi sonuçlarına göre, ekstrakt moleküllerinin yumuşak çelik yüzeyine neredeyse maksimum kaplama ve temas elde etmek için adsorplandığı söylenebilir.
Anahtar kelimeler: Bryaceae, *Ptychostomum schleicheri*, Çevre-dostu inhibitör, Korozyon, Metal mikroskopu.

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1. Introduction

Metallic corrosion stems from the chemical and electrochemical interaction between the metal surface and the environments surrounding the metal, as well as the gradual deterioration of the metal's structural integrity (Fang et al., 2019; Allaoui et al., 2017). When the cost of corrosion is considered worldwide, corrosion is a worrisome problem as it contributes greatly to the gross product (Abdul Aziz et al., 2022). Research on corrosion and its protection, which is related to energy conservation, conservation of resources and the continuation of sustainable development, is therefore of great importance (Du et al., 2022). In last years, small amounts of corrosion inhibitors have been incorporated to the aggressive solution, which can remarkably diminish the corrosion rate of metallic materials, especially in industry. With increasing inhibitor applications, interest in the concept of global green development has increased. However, many conventionally toxic inhibitors were also shelved (Ramezanzadeh et al., 2018; Umoren et al., 2019). On the contrary, interest in natural plant extracts as inhibitors has increased and has become a hot topic. It has been reported that various plant extracts such as Hyperoside (Huang et al., 2022a), *Uncaria laevigata* (Huang et al., 2022b), *Pterolobium hexapetalum* and *Celosia argentea* (Kumar and Mohana, 2014), *Thymus vulgaris* (Ehsani et al., 2017), *Mikania micrantha* (Du et al., 2022), *Saussurea obvallatta* leaves (Kalkhambkar et al., 2022), *Reineckia carnea* leaves (Wang et al., 2022) are used as effective inhibitors in inorganic acids recently.

Since plant extracts generally contain aromatic rings, conjugated double bonds, as well as polar functional groups with heteroatoms such as N, S and O in their molecular structures, these structures serve as adsorption centers (Mobin et al., 2019). The main point to draw attention to in this study is that *Ptychostomum schleicheri* (DC.) J.R. Spence ex D. Bell & Holyoak, whose inhibitor effect was examined, is a moss and therefore contains a large amount of secondary metabolites called phytochemicals in its structure. Such substances are called bryophytes, that is, they are the ancestors of land plants. The term bryophyte is derived from the words "Bryon" and "Phyton". The bryon means mosses and the phyton means plants (URL1). Secondary metabolites called phytochemicals, which are abundant in bryophytes, interact strongly with metallic surfaces. Because they have groups that provide strong adsorption centers such as flavonoids, terpenoids, amino acids and alkaloids in their structures (Özkır and Ezer, 2020; Özkır, 2021).

In this study, in order to inhibit the mild steel corrosion in HCl solution, it was aimed to take the extract of *Ptychostomum schleicheri* moss in water and to exert its inhibitor effect with three electrochemical methods at four different concentrations. The use of bryophytes as inhibitors in corrosion studies was investigated for the first time in Turkey with the study published by Özkır and Ezer in 2020 (Özkır and Ezer, 2020). For this reason, these types of plants have started to attract intense interest in recent days. In conclusion, it has been argued that the non-poisonous and environmental friendly *Ptychostomum schleicheri* extract is an essential inhibitor for corrosion duration with this study.

Mosses absorb water by holding moisture in their environment (Shaw et al., 2011). In this way, they also contribute to the prevention of erosion. The water holding property of *Ptychostomum schleicheri* moss, whose inhibitor property were examined, was displayed in Figure 1 with the help of digital camera integrated OLYMPUS (SZX7) a stereo microscope.

2. Materials and Methods

2.1. Plant material

The *P. schleicheri* mosses were collected from Aladağlar, Niğde, Kaleboynu place, 2250 m, 37°53'966" N, 35°10'305" E, in 22 June 2013.

2.2. *Ptychostomum schleicheri* extract preparation

Moss samples were already dry because they had been collected earlier. It was cleaned from the dirty parts remaining in the roots and the moss samples weighed 6 g were ground into powder. Weighed samples were placed in a 250 mL reaction flask and refluxed for approximately 13 h by adding sufficient distilled water. The resulting solution was filtered and a yellow extract was obtained. Figure 2 shows that the images before and after the moss extract was taken. The actual concentration of the stock *Ptychostomum schleicheri* extract was calculated as 0.321% (w/v) by weighing the residue left after evaporation of 10 mL of the extract. All concentrations studied were prepared by diluting from stock moss solution in 1 M HCl.

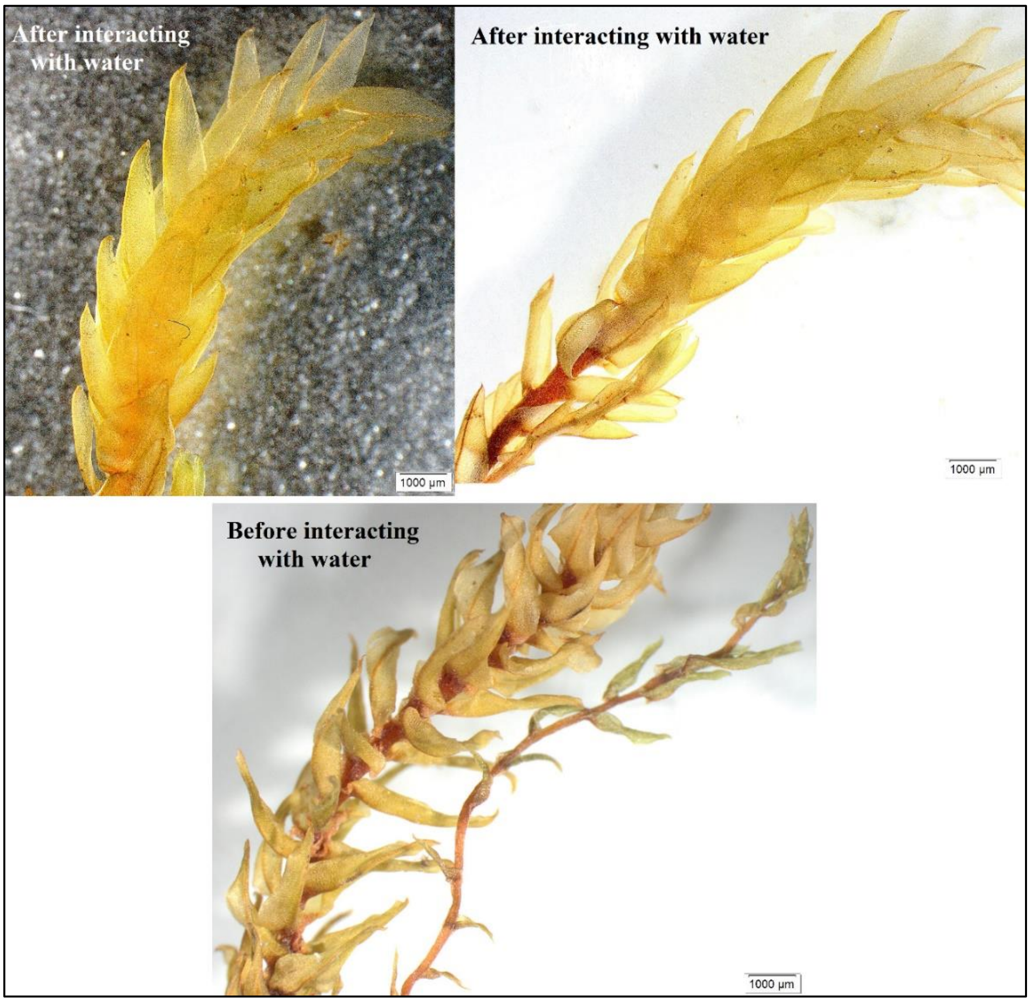

Figure 1. Stereo microscope images of *P. schleicheri* moss in un-watered and watered


Figure 2. The photographs of *Ptychostomum schleicheri* moss and extract solution

2.3. Experimental reagents and measurements
Corrosion experiments were performed using the three electrode technique. The first electrode is the working electrode and mild steel, whose composition is indicated in Table 1, was used. The surface area of each mild steel electrode is 0.5024 cm^2.

Table 1. The composition of the mild steel electrodes (wt.%)

Element	%	Element	%	Element	%
(C)	0.08400	**(Mn)**	0.40900	**(Si)**	0.10200
(Mo)	0.01040	**(S)**	0.01900	**(Cr)**	0.06030
(P)	0.01100	**(Ni)**	0.07890	**(Nb)**	0.00222
(Co)	0.00198	**(Cu)**	0.21700	**(Al)**	Trace
(Ti)	Trace	**(V)**	0.01100	**(W)**	Trace
(Pb)	Trace	**(Sn)**	0.01620	**(Sb)**	Trace
(Fe)	Remain				

The second electrode used in the experiments is the platinum plate called the counter electrode. The surface area of this plate is 2 cm^2. The third electrode used last is the reference Ag/AgCl electrode. All working electrodes, a mixture containing a certain amount of polyester, accelerator and hardener were prepared and poured into a cylindrical mould and used as such. The surfaces of mild steels were polished with alumina solution by sanding with different numbered emery papers just before each measurement. Afterwards, it was cleaned with acetone and distilled water and immersed in test solutions containing aggressive and inhibitor solutions.

CHI 660B electrochemical analyser device was used for the three electrochemical methods applied in the experiments. Before each measurement, the working electrodes were immersed in the test solutions containing and without inhibitor for one hour, and the system balance required for the corrosion process was provided at the open circuit potential (E_{corr}). Firstly, EIS measurements were performed under 5 mV amplitude in the frequency range of 10^5 Hz to $5x10^{-3}$ Hz, secondly, LPR measurements were performed at a scanning rate of 0.1 mV s^{-1} and by applying a potential in the range of ±10 mV (Ag/AgCl) from the open circuit potential. The polarization resistance ($^*R_p$) values given by the LPR method, as a result of the values given by the CHI device, a curve was drawn with the potential against the current values and calculated by taking the inverse of the slope obtained from this curve. The third and last applied method is the Tafel extrapolation method. Also known as potentiodynamic polarization curves. With this method, at a scanning rate of 1.0 mV s^{-1}, firstly -0.350 V from the open circuit potential (corrosion potential) to the cathodic potential, and then +0.350 V from the open circuit potential to the anodic side was applied. Corrosion current density (i_{corr}) values of the process were calculated from the curves obtained by this extrapolation method.

2.4. Surface characterizations of mild steels

The images of mild steel surfaces were evaluated by metal microscope (a digital camera integrated OLYMPUS (BX-51), Centre Valley, PA, USA) in HCl solutions with and without 0.2% (w/v) *Ptychostomum schleihceri* extract after 1 hour of immersion at 298 K.

3. Results and Discussion

3.1. Corrosion inhibition by electrochemical methods

In order to measure corrosion inhibition, firstly, four different concentrations of extract solutions {0.200%; 0.100%; 0.050% and 0.025% (w/v)} were prepared in 1 M HCl, and all of these experiments were conducted at these four concentrations. The first stage of corrosion inhibition is EIS experiments. EIS is among the first preferred methods with the application of alternating current in corrosion measurements. That is, it is preferred because it does not distort the metallic surface with the current applied during the evaluation phase of the corrosion process that exists between the metal and the inhibitor. Moreover, this method is quite essential in terms of both time and cost and practicality. After the experimental EIS measurements were taken from the CHI, the equivalent circuits of the corrosion process for both the aggressive solution and the moss extract solutions were proposed separately in Figure 3 using Zview2 software.

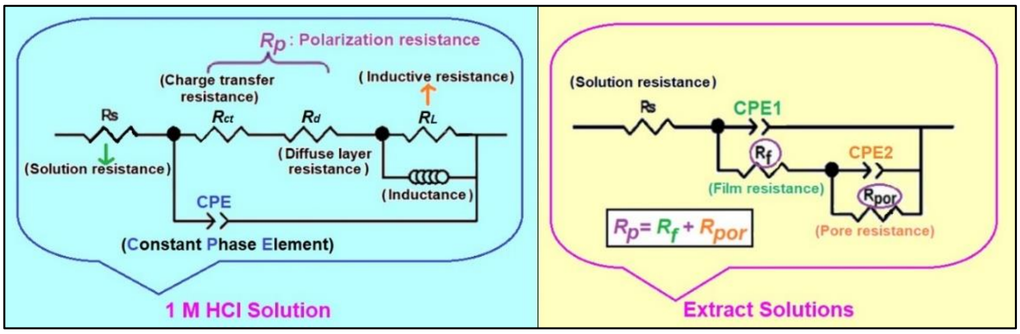

Figure 3. Proposed equivalent circuits for 1 M HCl solution and all *Ptychostomum schleicheri* extract solutions

The first thing to notice between two equivalent circuits is the film resistance (R_f) when there are moss extracts in the solution. The film resistance is due to the fact that the secondary metabolites in the moss are directed on the mild steel in the acidic solution, that is, they are adsorbed. Another difference is the inductance (L) and inductive resistance (R_L) value found in the equivalent circuit model valid for the hydrochloric acid solution. So, there is an interaction with the charge transfer resistance (R_{ct}) during the corrosion process. The polarization resistance (R_p) in the equivalent circuit of a 1 M HCl solution includes both the diffuse layer (R_d) on the surface and the resistances responsible for charge transfer (R_{ct}). While the Rp in the equivalent circuit created for all moss extract solutions is both the surface film (R_f) layer and It is responsible for the sum of the pore (R_{por}) resistances, which includes all the species accumulated on the surface together with the corrosion products. The data fitted as a result of the experiment are presented in Figure 4 as EIS diagram, containing both blank (1 M HCl) and solutions with inhibitor.

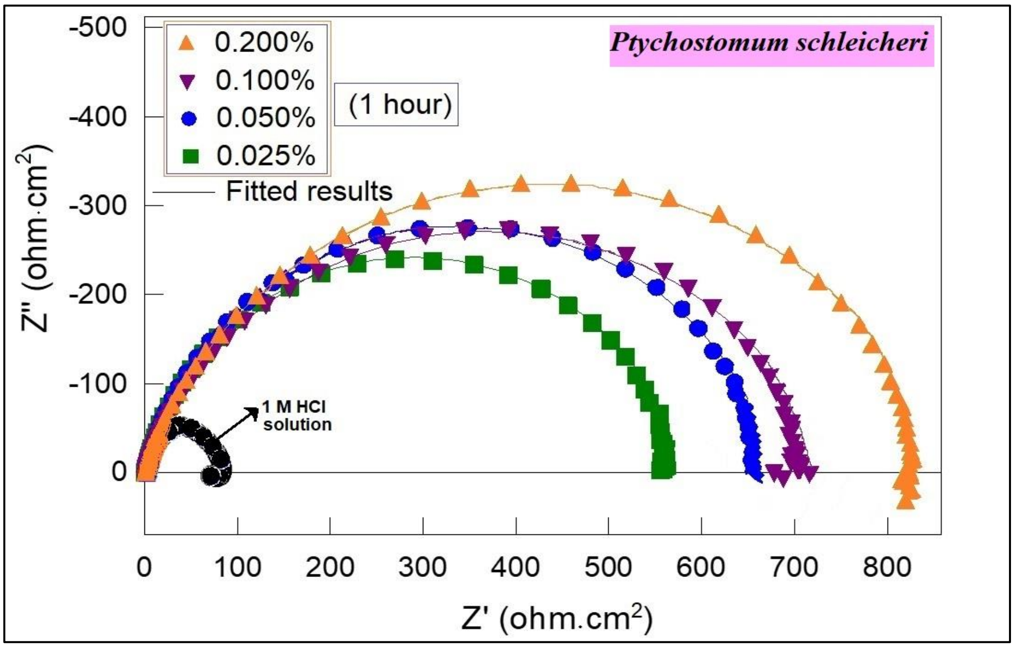

Figure 4. EIS diagram of *P. schleicheri* extract in 1 M HCl solution for 1h exposure at 298 K

In the absence of *Ptychostomum schleicheri* extract, metallic dissolution appears in mild steel. As the moss extract is added to the medium, the diameter of the capacitive loops increases as it is clearly seen in Figure 4. In other words, R_p values raised with the addition of *Ptychostomum schleicheri* extract in different concentrations into the aggressive solution and so, the rate of dissolution of the metal slowed down. All solutions in the EIS diagram are depressed semicircles. Even more are the species located in the high and low frequency regions. *LPR and potentiodynamic polarization measurements were also taken consecutively after the EIS measurement. Parameters calculated by EIS and LPR are given in Table 1.

Table 2. The calculated EIS and LPR data at 298 K

C (w/v %)	***CPE*** (µF cm^{-2})	***n***	R_s (Ω cm^2)	R_L (Ω cm^2)	***L*** (H)	R_p (Ω cm^2)	***η*** (%)	$^*R_p$ (Ω cm^2)	$^*\eta$ (%)
P.* s*chleicheri									
1 M HCl	110	0.94	1.2	8	4	72	-	71	-
0.025	80	0.89	1.4	-	-	580	**87.6**	628	**88.7**
0.050	75	0.86	1.3	-	-	690	**89.6**	682	**89.6**
0.100	60	0.82	1.1	-	-	750	**90.4**	723	**90.2**
0.200	52	0.81	1.2	-	-	855	**91.6**	855	**91.7**

It is possible to say from Table 2 that the highest inhibition was achieved with the solution containing 0.200% (w/v) *P. schleicheri* extract. Because while the R_p value in the solution containing 1 M HCl was 72 Ω cm^2, the R_p value in the solution containing 0.200% (w/v) moss extract was found to be 855 Ω cm^2 by both methods. As a result, a protection of 92% (η%) was achieved at the highest concentration after 1 hour of immersion. This result is a very high inhibition. In this study, the effect of the aqueous extract of *Ptychostomum schleicheri* moss on the corrosion inhibition of mild steel in HCl solution is presented by calculating the percent inhibition efficiency values (η% and $^*\eta$%) at each concentration by both methods as follows (Deyab et al., 2022):

$$\eta(\%) = \left(\frac{R'_p - R_p}{R'_p} \right) \times 100 \qquad (1)$$

Where R_p and R'_p and are in 1 M HCl solution and moss extract solution values, respectively.

The CPE values in Table 1 represent the double layer formed between the metal and the inhibitor solution. CPE is also known as double layer capacitance. The lower the double layer thickness between the metal and the inhibitor, the better the inhibitor will protect the metal (Haldhar et al., 2019). As the concentration of moss increases, the Rp values upsurge and accordingly the CPE values decrease (Popoola, 2019). CPE values attenuated from 110 µF cm^{-2} to 52 µF cm^{-2} with raising concentration. In addition, the "*n*" values, called the surface inhomogeneity coefficient, decreased as the moss extract was added to the acidic environment. In other words, it can be concluded that the oxide layer formed during the corrosion process slows down as the inhibitor is added to the solution. R_p and $^*R_p$ values at each concentration and η% and $^*\eta$% values were calculated based on these values with both EIS and LPR methods. According to Table 2, the findings obtained by both methods are highly compatible with each other. As a result of EIS and LPR measurements, it can be said that the *Ptychostomum schleicheri* extract prevents the corrosion of mild steel in 1 M HCl at a very high rate. In particular, 90% and more than 90% inhibition was achieved in three of the four concentrations studied. This can be interpreted as the organic molecules in the *Ptychostomum schleicheri* extract delay the charge transfers by forming a hard film layer on the metal surface. Since *P. schleicheri* is a bryophyte species, aromatic ringed phenolic, glycoside, alkaloid compounds in its basic structure and π-electrons in these compounds, as well as unshared electron pairs in heteroatoms act as basic adsorption centres. For this reason, at this stage of the study, it would be more accurate to talk about collective inhibition in protecting the metal surface of such compounds. A very high inhibition was demonstrated at the 1-hour immersion time by green inhibitor.

The third and last electrochemical method, "potentiodynamic polarization" curves, is actually a kind of extrapolation application. In Figure 5, Semi-logarithmic cathodic and anodic current-potential curves, which are overlaid at each concentration as a result of the experimental data, are shown.

In Figure 5, both cathodic and anodic Tafel curves are seen together. As can be clearly seemed from this curve, when *P. schleicheri* extract is added to 1 M HCl solution at different concentrations, a diminish is observed in both the cathodic and the anodic side compared to the curve in the acidic solution. This indicates that moss acts as a mixed natured inhibitor on mild steel in hydrochloric acid solution. According to the Table 3, no remarkable shift was observed in the corrosion potentials either. The -β_c values, which represent the cathodic slope, did not cause a significant change on mild steel in 1 M HCl with and without *Ptychostomum schleicheri* moss extract, and it was also concluded that it was not affected by the hydrogen evolution mechanism (Tripathy et al., 2019). This confirms that the corrosion mechanism of mild steel remains the same in the presence and absence of moss, thus acting as a mixed-type inhibitor to the mild steel (Mehta et al., 2022).

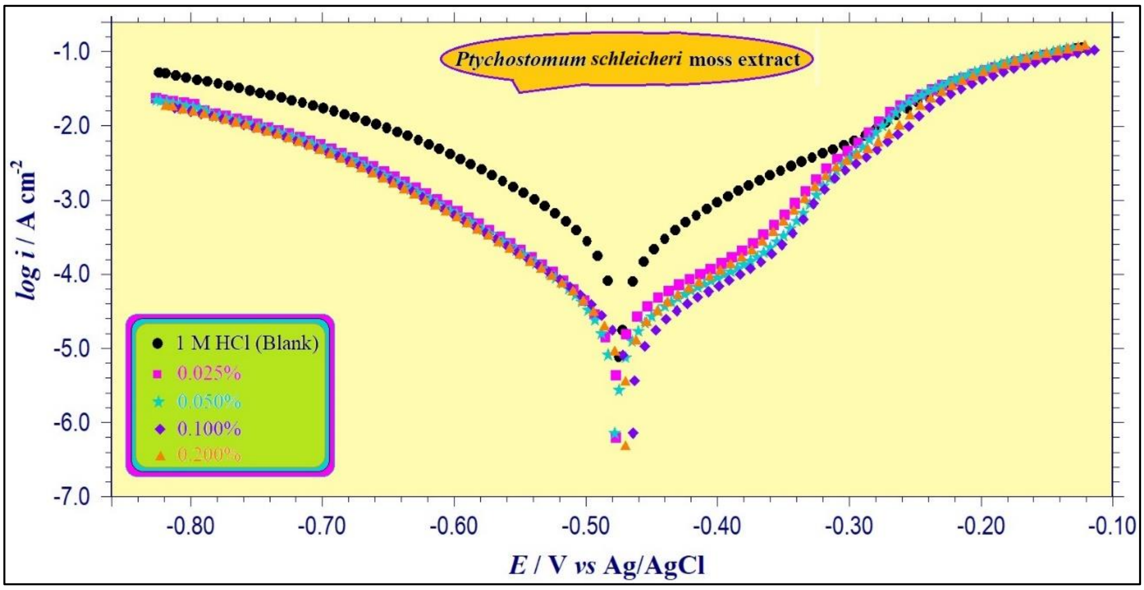

Figure 5. Semi-logarithmic current-potential curves at 298 K by Tafel extrapolation

Table 3. Tafel parameters of mild steel in 1 M HCl with and without *Ptychostomum schleicheri* extract at 298 K

***P. schleicheri* extract concentration (w/v %)**	**E_{corr} (V/Ag/AgCl)**	**$-\beta_c$ (mV dec^{-1})**	**i_{corr} (μA cm^{-2})**	**η (%)**
1 M HCl	-0.475	108	265	-
0.025	-0.477	91	34	**87.2**
0.050	-0.478	93	32	**87.9**
0.100	-0.470	93	24	**90.9**
0.200	-0.464	92	21	**92.1**

The percent inhibition efficiencies (η%) found from the Tafel curves are given by the equation below (Kumar et al., 2022):

$$\eta(\%) = \left(\frac{i_{corr} - i'_{corr}}{i_{corr}}\right) \times 100 \qquad (2)$$

Where i_{corr} and i'_{corr} are the corrosion current densities in 1 M HCl and *Ptychostomum schleicheri* extract, respectively.

The gradual diminish in the corrosion current density values with the increase in the concentration of moss indicates the constitution of an inhibitor defensive barrier on the surface of the mild steel and resists its dissolution. With the Tafel method, 92.1% protection was revealed at the highest concentration (0.200% {w/v}). These results are also in good agreement with those in Table 2. To summarize the inhibition efficiencies calculated by the EIS, LPR and Tafel extrapolation methods, they were alined in the range of 87.6%-91.6%, 88.7-91.7% and 87.2%-92.1%, respectively.

3.2. Surface characterization of mild steels by metal microscope

Figures 6a and 6b show the images of mild steel electrodes immersed for 1 hour in solutions containing 1 M HCl and 0.200% (w/v) *Ptychostomum schleicheri* extract, examined with a metal microscope (Abderrahim et al., 2022; Özkır and Kayakırılmaz, 2020). As can be seen in Figure 6a in the absence of moss, the mild steel surface is severely damaged from the acidic solution. After the interaction of the aggressive solution with the *Ptychostomum schleicheri* extract, the roughness of the surface was significantly reduced (Fig. 6b). In this case, it was shown that the moss extract of *P. schleicheri* deposited on the surface of the mild steel and formed a strong and protective film.

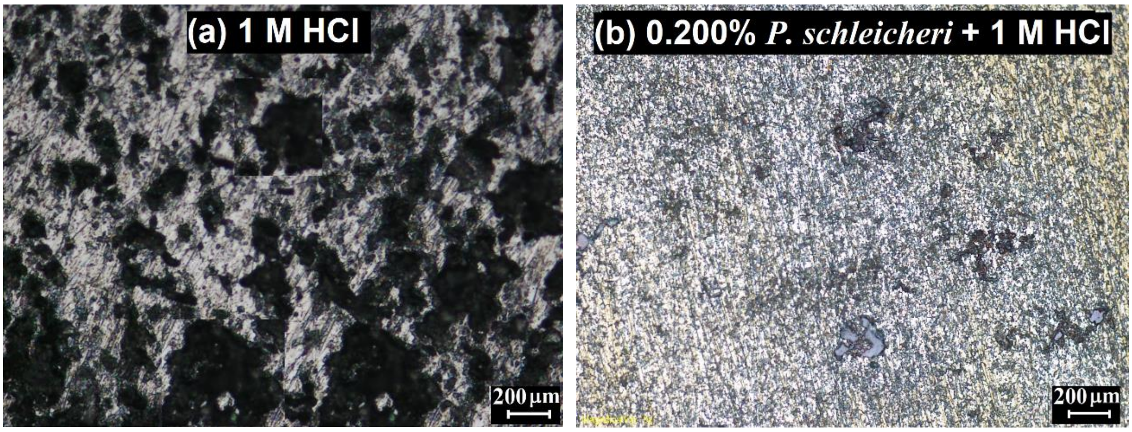

Figure 6. Metal microscope views of the mild steels for 1 h of immersion at 298 K

At this stage of this research, the findings obtained by electrochemical experiments approve each other with metal microscope images.

4. Conclusions

Ptychostomum schleicheri extract, whose inhibitory effect was examined, can be a remarkable answer to the corrosion phenomenon, which is of great importance for many industries. It is of great importance that the mild steel in 1 M HCl of *P. schleicheri*, which is a species of the Bryaceae family, is both environmentally friendly and green, and is applied for the first time in the literature.

The experimental findings clearly indicated that the moss extract was adsorbed very well on the mild steel surface after 1 h of immersion time, with an inhibition of 90 percent. According to the EIS and LPR findings, the increment in polarization resistance values with enhancing concentration values is also an evidence of an increase in the number of organic molecules attached to the metal surface and an effective inhibition.

According to the Tafel extrapolation findings, as *P. schleicheri* extract was inset to the hydrochloric acid solution, the corrosion current density values decreased and accordingly the inhibition efficiencies raised. Since the cathodic Tafel slope ($-\beta_c$) values did not change much, it was interpreted that the hydrogen evolution mechanism was not affected by the inhibitor. At the same time, *P. schleicheri* extract behaved as a mixed-type inhibitor, as the concentration of moss extract increased in 1 M HCl solution, reducing the slope of both the cathodic and anodic side curves at the same rate.

When the images taken with a metal microscope for the surface characterization of the *P. schleicheri* extract are examined, the surface containing the moss appears to be extremely smooth and homogeneous compared to the image of the surface in the aggressive solution. The other surface was heavily affected by the hydrochloride acid solution and it was concluded that there were deep pits.

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