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Corrosion Inhibition Performance of *Euphorbia pulcherrima* Bark Extract in 1M H_2SO_4 on Mild Steel

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Abstract

The bark of the attractive flower *Euphorbia pulcherrima* (Lalupate), which is widely accessible in Nepal, can be used to extract an alkaloid that acts as a corrosion inhibitor. The methanolic extract of *Euphorbia pulcherrima* (EP) in 1M H_2SO_4 was employed to study corrosion inhibition of mild steel (MS) by weight loss and potentiodynamic technique. The immersion time and EP extract concentration consequences on corrosion inhibition were explored. The weight loss of MS is significantly reduced in the presence of EP inhibitors caused by the adsorption of inhibitor molecules. The findings exhibited that the increasing immersion time and EP concentration resulted in an increment in inhibition efficiency (IE). The maximal IE from the weight loss method was determined to be 90.38% for 1000 ppm extract immersed for 6 hours at room temperature and 86.36% IE was obtained for 1000 ppm concentration of the EP extract after 1hr of immersion by potentiodynamic polarization. The open circuit potential (OCP) of the MS in the inhibitor solution confirmed the EP extract behaving as a mixed inhibitor. The current density decreased with the addition of the EP, which reflected the inhibitive nature of inhibitor molecules. Similarly, the corrosion current density decreased as the EP extract concentration increased. Hence, it implied that the bark extract of EP is a potent inhibitor of MS corrosion, even after just one hour of immersion. Because of the reduction of hydrogen ions and the adsorption of inhibitor molecules on the MS surface, EP extract is effective in inhibiting corrosion current on the cathodic and anodic polarization curves, forming a barrier that stops the evolution of hydrogen gas and metal dissolution. Fourier Transform Infrared (FTIR) spectroscopic analysis showed the presence of N-H and C=O functional groups in the EP attributed to the adsorption on MS and consequently reducing the dissolution of MS.

Keywords: barrier, euphorbia pulcherrima, inhibitor, mild steel, polarization

Introduction

Corrosion has evolved as a major challenge with industrialization. In the 21st century, it has remained an issue for corrosion scientists and engineers for the substantial preservation of material and economic loss. Acid solutions are utilized often in a variety of industrial operations. Pickling,

cleaning, descaling, and etching metals using hydrochloric and sulphuric acids is common practice, however these acids damage metal surfaces(Iluve and Ashwe 2023; Rani and Basu 2012; Tezeghdenti et al. 2015). Isolating the metal surface from corrosive reagents is one of the most practical techniques for avoiding corrosion (Abdelaziz et al. 2021). Corrosion inhibitors are typically the most suited approach for achieving this goal among the various options (Rani and Basu 2012). Organic, inorganic, precipitating, passivating, or volatile species might be used as inhibitors. Corrosion inhibitors are often classified into three categories: oxidizing, precipitating, and adsorption inhibitors. Organic compounds having nitrogen, sulfur, phosphorous and oxygen are often used as adsorption inhibitors (Ngouné et al. 2019; Verma et al. 2018). The molecular structure of inhibitors having pi electrons promotes adsorption on corroding metal surfaces (El-Haddad et al. 2019). The nature of the corrosive media, the metal composition, its surface charge, and the inhibitor's molecular structure all impact adsorption. Some corrosion inhibitors, which are employed in various environments significantly, reduce the metal's dissolution. Inhibitor adsorption in acid corrosion can alter the double layer's structural properties, slowing down either anodic metal dissolving cathodic hydrogen ion reduction, or both.

Some corrosion inhibitors and their derivatives are recognized to be harmful and polluting to the environment (Karki et al. 2023). As a result, novel corrosion inhibitor replacements that are nontoxic, environmentally benign, ecologically acceptable, and cheap are needed. Compounds acquired from plants have been proven as highly effective inhibitors among alternative corrosion inhibitors (Karki et al. 2023; Pourzarghan and Fazeli-Nasab 2021; Quraishi et al. 2010). Numerous studies have revealed that a wide variety of environmentally friendly plant extracts and pharmaceutical products can prevent metal corrosion in a variety of acidic situations. Environmentally friendly natural amino acids have also been studied for their ability to prevent corrosion in a variety of corrosive environments for steels, iron, aluminum, copper, nickel, and certain alloys (Khan et al. 2015). Because natural products are generally inexpensive and simple to use, the 21st century has seen a lot of research on them. The extracts derived from various parts of plants applied as inhibitors in acidic environments have been recorded in a substantial number of scientific studies.

Since the 1970s, plentiful studies on alkaloids for corrosion inhibition have been conducted, primarily in hydrochloric acid and sulfuric acid medium. Berberine, piperine, atheroline, brucine, tropane, pyrrolizidine, caulerpin, isoreserpiline, anibine, strychnine, quinine, vasicine, vasicinone, and isopelletierine, as well as lupanine, multiflorine, and indole (Li H.J., et al. 2018) are a few of the alkaloids that have been investigated against the MS corrosion in acidic media. The extracts of *Artemesia vulgaris* (Karki, et al. 2018), *Lantana camara* (Shrestha et al. 2019), *Euphorbia royleana* (Thapa, et al. 2019), and *Jatropa curcas* (Gupta, et al. 2020) have been investigated as corrosion inhibitor. Similarly, as inhibitor, *Eucalyptus globules* (Gupta, et al. 2020), *Equistem helyme* (Karki et al., 2021), *Berberis aristata* (Karki et al. 2020), and *Eletterai cardamom* (Gupta et al. 2023) extracts have also been studied, and demonstrated adequate protection of mild steel (MS) with good inhibitory efficacy. With the weight loss technique and characterization procedures in 0.5 M HCl, the viability of employing Euphorbia hirta (EH) extract as an inhibitor for MS was investigated. The findings demonstrated the rate of corrosion decreased with rising concentration of inhibitor and time. The maximal IE was attain at an optimal concentration of 15 g/L which improved within the temperature range of 30-70°C (Aliyu et al. 2022). El Bribri et al. (2013) examined Euphorbia falcata extract (EFE) as an inhibitor for carbon steel

in 1 M HCl. According to the experimental findings, EFE is a potent inhibitor of corrosion, and the effectiveness of corrosion protection increased with the concentration of EEF. According to polarization curves, EFE is a mixed inhibitor. The Langmuir adsorption isotherm illustrated the better adsorption of the E. falcata extract which demonstrated that a physisorption process is primarily responsible for the carbon steel inhibition in 1 M HCl.

High-altitude plants are abundant in Nepal, and it is quite likely that an extract from one of these plants will function as an effective corrosion protective layer on active metals like steel. Only a few numbers of natural compounds with Nepali roots have been studied as corrosion inhibitors. Phytochemical screening of *Euphorbia pulcherrima* (EP) has revealed that alkaloids, steroids, terpenoids, saponins, glycosides, reducing sugar, and amino acids are the main constituents of EP extract (Sharif et al. 2015). Sharif et al. (2015) have isolated and identified alkaloids O-(3-(tert-Butyl) cyclohexa-2,4-dien-1-yl)(6-methoxypyridin-2-yl)(methyl)carbamothioate,4-(((Tetrahydrofuran-2-yl)methyl)amino)-1-oxaspiro[4.5]dec-3-en-2-one, etc present in *Euphorbia pulcherrima*. The main objective of this investigation is to determine how well EP extract, protects MS from corroding in an acidic environment.

Materials and methods

Preparation of mild steel sample

A mild steel (MS) pieces were acquired from a trader in Kathmandu. It was cut into samples having dimensions of 3 cm x 3 cm x 0.1 cm. MS samples were abraded with #100 to #1000 grit Silicon carbide (SiC) papers in succession. Abraded specimens were cleaned with hexane and ultrasonicated in ethanol, dried, and kept in desiccators before use. Weight loss measurements and electrochemical measurements were performed with these samples.

Preparation of *Euphorbia pulcherrima* extract and its solution

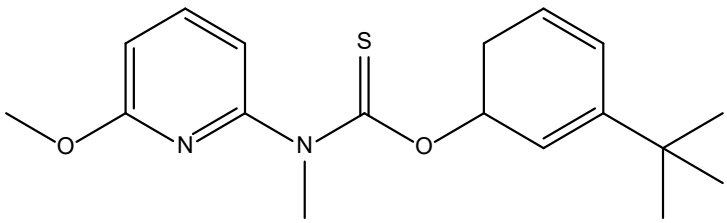
Stems of *Euphorbia pulcherrima* were gathered from the Ramghat-10, Pokhara (Latitude: 28°29 '14.7156" N and Longitude 83°44 '31.6824" E). The barks of stems were then peeled off and allowed to dry for roughly a month in a shaded place. It was subjected to grinding into fine powder in the grinding mill at Tribhuvan University.

The pulverized samples were mixed with hexane left for 2 days and filtered. Using hexane, the residue was washed and filtered. Methanol was poured in the residue left for a week and filtered. The filtrate was acidified with 3% tartaric acid and the pH was maintained at 10 with the help of NH_4OH. Alkaloids were separated by a solvent extraction process into a separating funnel mixed with chloroform. The organic layer contained primary, secondary, and tertiary alkaloids while the aqueous layer contained highly polar alkaloids (4°- alkaloid).

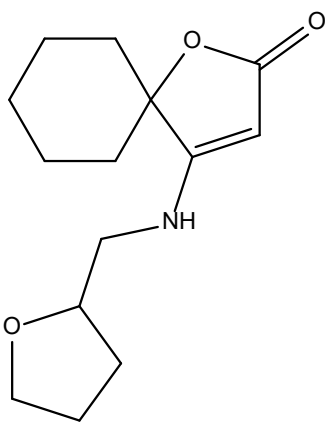
The organic layer was subjected to evaporation in a rotator evaporator. The extract was kept in a water bath for further evaporation at 35°C until it was dried.

1g of alkaloid extract was dissolved in 1M H_2SO_4 and 1000 ppm of 1L alkaloid solution was prepared. Similarly, the solutions of required concentrations ranging from 800 ppm to 200 ppm were prepared by serial dilution.

Figure 1: *Euphorbia pulcherrima* plant



O-(3-(tert-Butyl)cyclohexa-2,4-dien-1-yl) (6-methoxypyridin-2-yl)(methyl)carbamothioate



4-(((Tetrahydrofuran-2-yl)methyl)amino)-1-oxaspiro[4.5]dec-3-en-2-one

Figure 2: chief alkaloids molecules found in *Euphorbia pulcherrima*

Corrosion study by weight loss method

The difference between the MS sample weights before and after immersion in the acid and inhibitor solutions was used to evaluate the weight loss. Weight loss analysis was applied to investigate the effect of immersion time and inhibitor concentration on the corrosion rate. To investigate the impact of the immersion period, the MS samples were immersed in 1 M H_2SO_4 and 1000 ppm EP inhibitor solutions for 0.5, 1, 2, 3, and 6 h. Likewise, samples were immersed in 1 M H_2SO_4 and different solutions of inhibitor concentrations (200 to 1000 ppm) for 3 hours to examine the impact of inhibitor concentrations. The following relationships were used to calculate the corrosion rate (CR), surface coverage (θ), and inhibition efficiency (IE%):

$$\text{Corrosion Rate (CR)} = \frac{\text{Weight loss}(\square)}{\text{Area}(\square)\times\text{time}(\square)\times\text{density}(\square)} \times 8.76 \times 10^4 \quad (1)$$

W stands for weight loss of the MS in gram t is the immersion time, *in* hours, A is the surface area of the MS (cm^2), and d is the density of the MS (g cm^{-3}).

$$\text{Surface Coverage } (\theta) = \frac{Wo-Wi}{Wo} \quad (2)$$

$$\text{Inhibition Efficiency } (IE)\ \% = \frac{Wo-Wi}{Wo} \times 100 \quad (3)$$

W_o and W_i represent the weight loss of the MS in the absence and presence of the inhibitor.

Corrosion study by electrochemical measurement

Three electrodes system consisting of a saturated calomel electrode (SCE) as a reference, a graphite electrode as a counter electrode, and the MS sample as a working electrode was used for electrochemical measurements. The open circuit potential (OCP) of the MS was recorded for 30 minutes at intervals of 2 minutes. To achieve a steady-state condition, the OCP of the MS was measured prior to anodic and cathodic polarization of the MS in acid and EP solution. Potentiodynamic polarization was performed using a Hokuto Denko potentiostat (HA-151) away from OCP ± 350 mV with 60 mv/S. The corrosion current (Icorr) and corrosion potential (Ecorr) were estimated using the Tafel plot. The corrosion *IE* was determined by applying the following relation:

$$IE\% = \frac{Icorr(uninhibited) - Icorr(inhibited)}{Icorr(Uninhibited)} \times 100 \quad (4)$$

Characterization by Fourier Transform Infrared (FTIR)

An IR prestige 21-FTIR device (Shimadzu, Japan) was used to examine Fourier transmission infrared (FTIR) spectra. IR spectra were taken by dissolving EP extract in H_2SO_4 solution, and its functional groups were identified

Results and discussion

Alkaloids test

The *Euphorbia pulcherrima* extract exhibited the occurrence of alkaloids.

Table 1

Results of phytochemical screening test for alkaloid.

Mayers Test	Wagners Test	Dragendroffs Test
+	+	+

Effect of immersion time

Table 2 shows the corrosion rate, surface coverage, and inhibitory efficacy of EP extract resulted from weight loss measurements at various time intervals.

Figure 3 represents the corrosion rate as a function of immersion time with and without inhibitor. The results reveal that the EP extract has a substantial effect on the MS corrosion rate. During the immersion period, the EP reduced the corrosion rate of the MS sample by around 6 to 11 times. The adsorption of phytochemicals of EP extract is responsible for the reduction in corrosion rate. Figure 4 depicts the inhibitory efficiency of 1000 ppm plant extract for periods of 0.5, 1, 2, 3, and 6 hours. After 6 hours of immersion, the inhibitory efficacy of EP extract was found to be around 92.0%. It depicts the MS surface covering.

Table 2

Corrosion rate, surface coverage, and inhibition efficiency of EP at various immersion times

Time (hours)	Corrosion Rate (mm/year)		Surface Coverage (θ)	Inhibition Efficiency%
	For acid	**For inhibitor solution**		
0.5	212.77	37.94	0.819	81.91
1	186.36	27.15	0.854	85.42
2	124.55	16.55	0.867	86.7
3	124.16	14.88	0.88	88.01
6	78.73	7.56	0.903	90.39

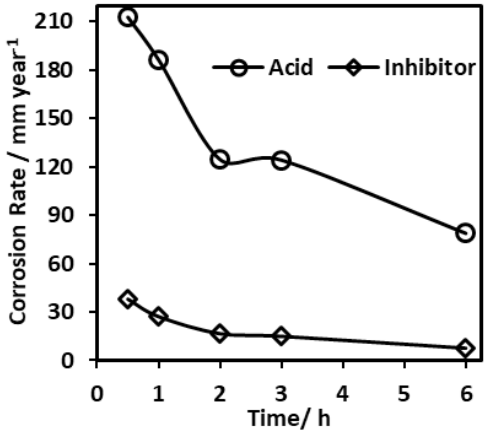


Figure 3: *Variation of rate of corrosion with time of immersion for 1000 ppm of EP inhibitor in 1M H_2SO_4 at room temperature*

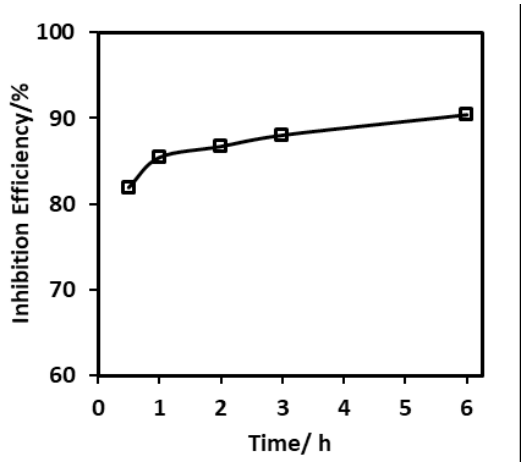


Figure 4: *Variation of IE with time of immersion for 1000 ppm of EP inhibitor in 1M H_2SO_4 at room temperature*

Effect of EP extract concentration and inhibition efficiency

The weight loss measurement against different concentrations of the EP inhibitor in 1M H_2SO_4 revealed that the weight loss of MS decreased with rise in concentration of EP as shown in Table 3.

Table 3

Weight loss, corrosion rate, surface coverage, and inhibition efficiency of EP extract at different concentrations for 3 hrs immersion

Concentration (ppm)	Weight Loss(g)	Corrosion Rate (mm/year)	Surface Coverage(θ)	Inhibition Efficiency %
Acid	0.3005			
200	0.1635	67.57	0.455	45.59
400	0.128	52.9	0.574	57.4
600	0.0949	39.22	0.684	68.41
800	0.0671	27.73	0.776	77.67
1000	0.041	16.94	0.88	88.01

Figure 5 depicts the corrosion rate variation with the EP concentration. The results reveal that the EP extract significantly affects the corrosion rate of MS in 1M H_2SO_4. Figure 6 depicts the inhibition efficiency of different concentrations of EP extract for 3 hours. The result exhibited that the rise in concentration of the EP extract reduced the corrosion of MS and consequently increased the inhibition efficiency. Owing to the adsorption of the inhibitor molecules on the MS leads to the corrosion inhibition. Similar findings were observed by the gravimetric method(Rukaiyat, et al. 2018). When extract concentration rises, more inhibitor molecules become available for adsorption on the MS. This reduces the amount of metal surface area that can be directly attacked by acid (Moussaoui, et al. 2016). At the concentration of 1000 ppm, the extract showed the highest inhibition efficiency of about 88.01% when the sample was immersed for 3 hours at room temperature.

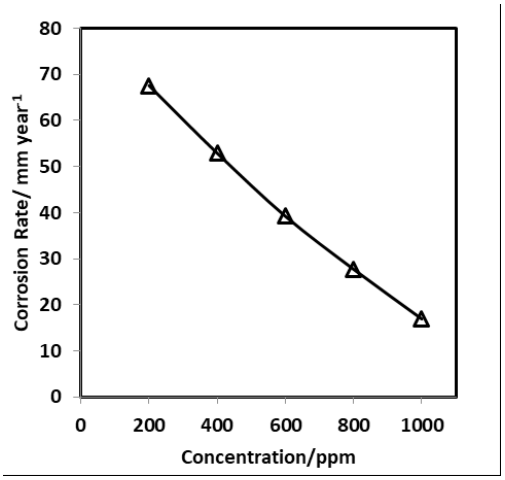


Figure 5: *Variation of corrosion rate with concentration of EP in 1M H_2SO_4 at room temperature*

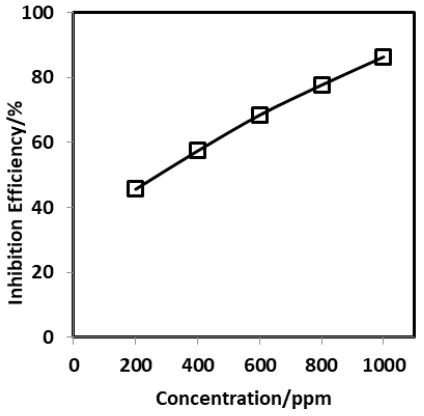


Figure 6: *Variation of inhibition efficiency with Concentration of EP in 1M H_2SO_4 at room temperature.*

Variation of open circuit potential with time

Figure 7 depicts the change in open circuit potential (OCP) with immersion time of MS in various concentrations of EP in acid solution. The result revealed that the OCP for the MS shifted to more positive direction as the concentration of the EP extract increased in the acidic solution, which shows slight anodic control. However, the shifts in OCP values are less than 85 mV, in both dipped and as dipped solution representing the EP extract as a mixed type of inhibitor.

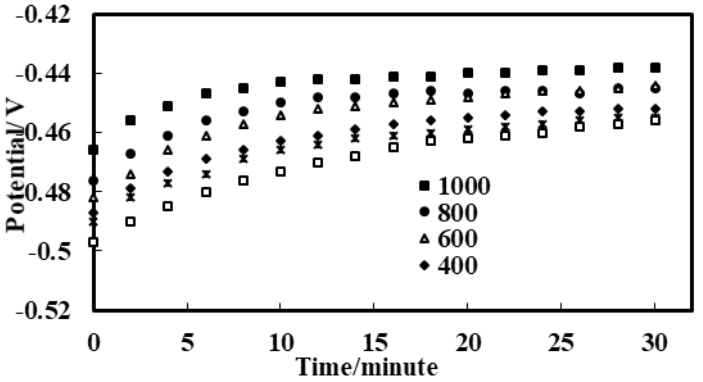


Figure 7: *Change in OCP with immersion time of MS in various concentrations of EP in 1M H_2SO_4*

Potentiodynamic polarization of MS 1M H_2SO_4 and EP extract

The polarization was executed in the MS samples immersed in 1M H_2SO_4 and EP inhibitor solution of various concentrations after 1-hour immersion (dipped). Polarization of mild steel was also carried out after 30 minutes of immersion in 1M H_2SO_4 and 1000 ppm of EP extract solution. Figure 7 shows

the comparative polarization behavior of the MS sample in 1M H_2SO_4 and 1000 ppm inhibitor solution for both as dipped (30 min immersion) and dipped (1 hour). The figure exhibits a considerable decrease in the current density of the MS sample in both cases as dipped and dipped for 1 hour. When the extract is added, the current of the cathodic curve significantly decreases, demonstrating the inhibitive activity of the EP extract. However, the cathodic curves continue to follow a parallel path, therefore the corrosion mechanism is unaffected. By adhering to the MS surface, the inhibitor molecules create a barrier layer and stop the hydrogen reduction reaction. As a result, the dissolution of iron slowed down(Gupta, et al. 2020; Karki et al. 2018; Raghavendra and Bhat 2017). It also reveals that OCP shifts marginally in a positive direction compared to the acid solution implying that the inhibitor molecule acting as a mixed type.

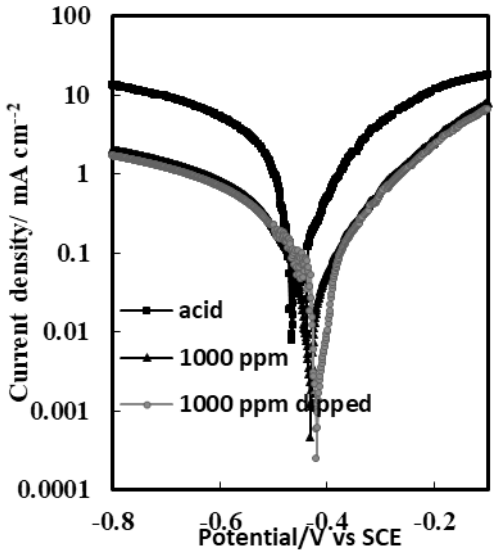


Figure 8: *Comparison of polarization behavior of the MS in 1 M H_2SO_4, 1000 ppm EP extracts dipped for 1 hour and after 30 min immersed (as immersed).*

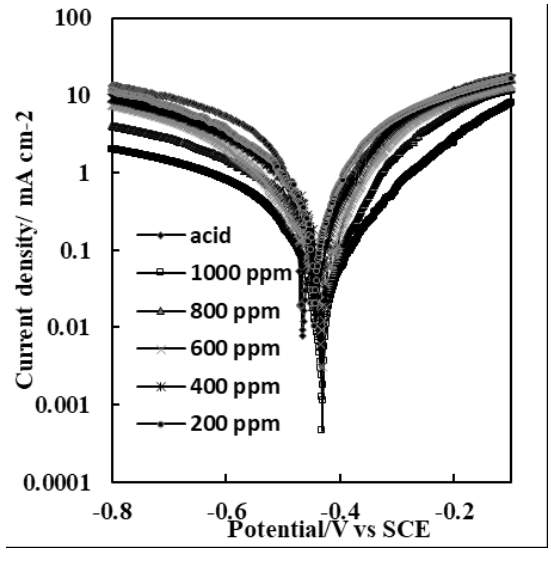


Figure 9: *Polarization behavior of the MS in different concentrations of EP extract in 1 M H_2SO_4 for 1 hour immersion.*

Figure 9 represents the potentiodynamic polarization behavior of MS with and without EP inhibitor at different concentrations ranging from 200, to 1000 ppm in 1M H_2SO_4 solution for 1 hour immersion. The result shows that the corrosion potential was shifted in a positive direction but less than 85mV which also again implies that inhibitory action is of mixed type. However, it can be noticed that lowering in current density upon the addition of the EP. This is the reflection of the inhibitive behavior of the inhibitor molecules. Likewise, the lowering in corrosion current density with the rise in the EP extract concentration. Thus, it suggested that the *Euphorbia pulcherrima* bark extract is an effective inhibitor for MS corrosion even for only 1 hour of immersion in the inhibitor. The effectiveness of EP extracts

in inhibiting corrosion current on the cathodic and anodic polarization curves due to the hydrogen ions reduction and the adsorption of inhibitor molecules on the MS surface respectively, forming a barrier that prevents the evolution of hydrogen gas and metal dissolution (Al-Dokheily, et al. 2014). The inhibitor's functional groups play a crucial role in the adsorption process. Water molecules are substituted for extract molecules during adsorption. The following describes a adsorption phenomenon on MS.

$Ex\ (sol) + XH_2O\ (ads) \rightarrow Ex\ (ads) + H_2O\ (sol)$ (5)

Ex(sol) = extract molecules in solution

Ex(ads)= extract molecules adsorbed on the MS

H_2O(ads)= water molecule adsorbs on the MS

X =the number of water molecules which is replaced by one molecule of the extract

Effect of concentration on inhibition efficiency

Figure 10 reveals the IE of the EP extracts on the MS increasing with the rise in the EP extract concentration. The fraction of the MS surface covered by the adsorbed molecule increases that boosts the effectiveness of the inhibition. The maximum inhibition efficiency of 86.36% was found at 1000 ppm EP extract after 1hour of immersion.

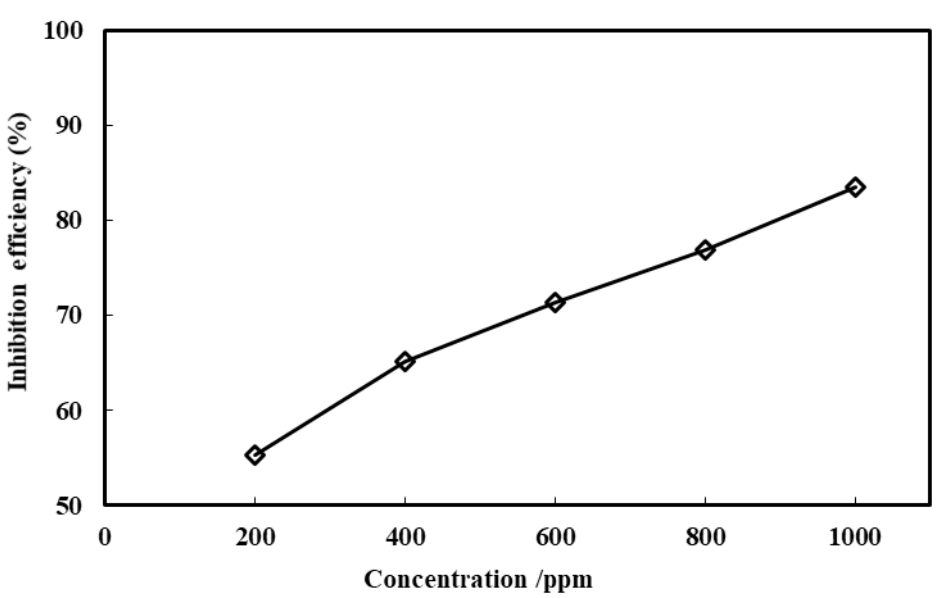


Figure 10: *Inhibition efficiency of different EP inhibitor concentration in 1M H_2SO_4 on MS after 1 hour of immersion*

Fourier Transform Infrared (FTIR) Spectroscopy Aanalysis

Figure 11 represents the FTIR spectra of EP extract. The functional groups present in the extract were identified which showed the band at frequency 3356-3188 cm^{-1} which are characteristic frequencies for stretching of functional groups O-H (3550-3200 cm^{-1}) and N-H (3500-3300 cm^{-1}). The

multiple peaks appearing in the range 1700-1500 cm^{-1} indicate the presence of C=O stretching, N-H bending, and α,β- unsaturated ketones (C=C) stretching. The peaks at frequencies 1465 cm^{-1} and 1450 cm^{-1} indicate aromatic C=C stretches. From this information, it can be inferred that the alkaloid extract contains electron-rich compounds that are responsible for adsorption on the MS which consequently formed protective film, resulting in suppression of corrosion.

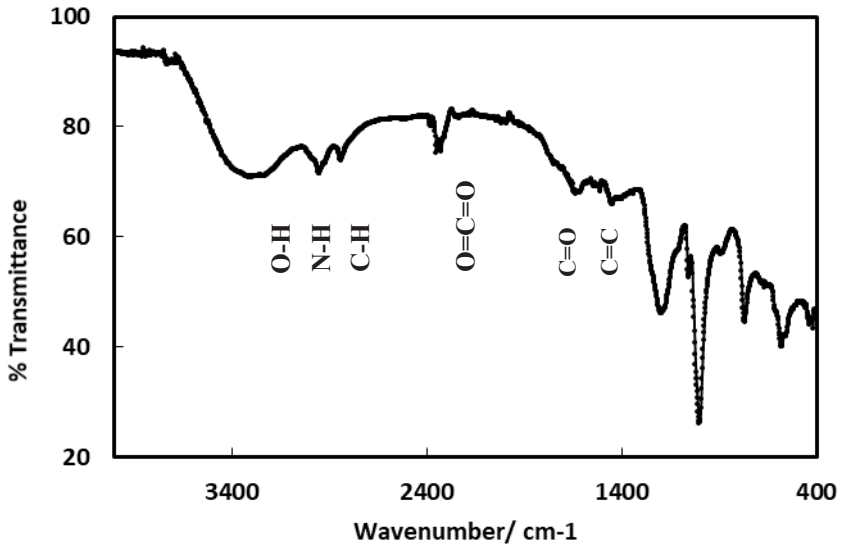


Figure 11: *FTIR spectra of alkaloid extract of Euphorbia pulcherrima labeled with functional group stretching at respective absorption frequency.*

Conclusion

In this study, *Euphorbia pulcherrima* (EP) exhibits efficient, environmentally benign corrosion inhibitors of MS in acid solution. The extent of weight loss of MS is greatly diminished in the presence of EP inhibitors caused by the adsorption of inhibitor molecules. The inhibition efficiency (IE) of EP is affected by the immersion time and inhibitor concentration in 1M H_2SO_4. The maximum IE from the weight loss method is found to be 90.38% for 1000 ppm extract immersed for 6 hours at room temperature and 86.36% IE is obtained for 1000 ppm concentration of the EP extract after 1hr of immersion by potentiodynamic polarization. The open circuit potential (OCP) of the MS in the inhibitor solution shows the extract behaving as a mixed inhibitor. FTIR spectrum confirms the presence of N-H, O-H, α, β- unsaturated ketones, C=C, and C=O stretching group liable for the adsorption of the extract on MS and thereby reducing the corrosion.

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