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Lepidium Didymium Plant Extract as Eco-Friendly Corrosion Inhibitor for Steel in Acidic Medium

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The efficiency of *Lepidium didymiu*m's aerial part for corrosion inhibition, has been explored by applying weight loss analysis, SEM, and spectroscopy technique on mild steel corrosion in 1 M H_2SO_4. These techniques have been used to investigate the corrosion features of the steel in the absence and existence of various quantities of *Lepidium didymiu*m's extract. With the aid of weight-loss statistics, the ability of the plant extract to construct a defensive film on steel surfaces is investigated. The formation of a protective membrane on the steel surface by the extract has also been supported by a surface morphology analysis (SEM). At 2500 mg/L, *plant* extract has the best inhibitory efficiency for steel in 1 M H_2SO_4 is 91.16%. *Lepidium didymiu*m extract is thought to be an effective inhibitor because of the presence of heteroatoms and multiple bonds.

Keywords: Green corrosion inhibitor, Lepidium didymium, Mild Steel, SEM, Weight loss method

1 Introduction

Low carbon steel has become a preferred choice in a variety of industries due to its great qualities. It offers weldability and machinability, resulting in a massive increase in its usage. Corrosion of mild steel occurs as a result of interaction with corrosive media like sulfuric acid and hydrochloric acid, which are employed in industrial procedures such as cleaning and descaling. Among the different approaches for avoiding the corrosion of metal surfaces, corrosion inhibitors are one of the most well-known. The employment of inhibitors is one of the most efficient strategies to prevent the unexpected metal dissolution, particularly in acid solutions. From the literature, it is found that more acidic conditions, the more corrosion suffered by the metal, and corrosion affect the metal's tensile strength and fracture toughness[1]. Inhibitors have traditionally been used in industry due to their outstanding anti-corrosive properties. Many chemicals are being used as corrosion inhibitors to control corrosion, like synthetic compounds. However, many side effects appeared which causing environmental destruction and be harmful to human health[2]. As a result, scientists began looking for environmentally safe inhibitors, such as organic inhibitors. To overcome this problem, natural and biodegradable corrosion inhibitor must be employed due to their non toxicity and affordability. The common constitution of such plant extracts includes phenols, amino acids, proteins, etc, which contain heteroatom like nitrogen, oxygen, sulphur, that play an efficient role in defensive layer formation on the top of low carbon steel, when employed with aggressive medium contributes to their strong inhibition efficiency[3-5]. Potentiodynamic polarization and EIS were used by Lebrini et al.[6] to explain the efficient corrosion inhibition action of *Oxandraasbeckii's* extract on steel. The alkaloids plant extracts contained liriodenine, azafluorenone, and other terpenoids that are crucial for appearance of protective coating upon the metal surface. Similarly, J.Bhawsar et al.[7] demonstrated the efficiency of extract of *Nicotiana tabacum* which contains nicotine as a phytochemical and steel inhibitory molecule in 2 M H_2SO_4 with a 94% effectiveness at 1 g/L. *Such as Rauvolfia macrophylla* act as corrosion inhibitor in HCl as well as in H_2SO_4 because of presence of alkaloids perakine and tetrahydroalastonine[8].

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The current study's objective is

a) To find naturally occurring, cheap and environmentally safe natural products as corrosion inhibitors of steel.
b) To study the corrosion inhibition efficiency of natural products using weight loss method.

In this research, here will be the determination of inhibitory effect aerial part of *Lepidium didymium*. *Lepidium didymium* also known as lesser swine cress is a species of brassicaceae and grows between July and September. Medicinal properties of the *Lepidium didymium* have been studied earlier but corrosion properties are not investigated yet. From literature, it has been found out that *Lepidium didymium* extract contain Kaempferol, Quercetin and Chrysoeriolas.

2 Materials and Methods

Various methods were used to find out the corrosion inhibition properties plant extract of *Lepidium didymium* that is also pictorially represented via Fig. 1.

2.1 Specimen preparation

The constitution of low carbon steel employed in this project is as depicted in Table 1. Dimensions of steel sample are 2 cm^2. Before the corrosion inspection, with a variety of sandpapers, every low carbon steel coupons were washed.

2.2 Plant collection and inhibitor preparation

Plant of *Lepidium didymium* was collected from Fatehgarh sahib, Punjab. After drying, with the aid of a grinder, the aerial part was then ground. After that, using a soxhlet device and with around 250 ml of water, 200 g of dried sample then refluxed at 80°C for about 24 hours. Then solution was filtered and a hot water bath is used to dry that, yielding 5 gm. of extract.

2.3 Corrosive media preparation

In this current investigation, 1 M H_2SO_4 is used which is prepared in double-distilled water with LobaChemie's AR grade sulphuric acid. To obtain solutions with varying concentration levels, *Lepidium didymium* extract powder was dissolved in the electrolyte of 1 M H_2SO_4.

2.4 Measurements of weight loss

We kept the corrosive solution volume at 100 ml for weight loss studies. Low-carbon steels were dipped with 2 cm^2 measurements for 24 hours in corrosive medium in this weight-loss analysis. Emery sheets of various grades were used to polish the mild steel coupons after that, it was wash out with acetone and afterwards with double distilled water. The mils steel coupons were pre-weighed and then submerged in 1 M H_2SO_4 with several inhibitor strengths. The mild steel coupons were removed after being exposed to aggressive media; the mass loss was evaluated after being rinsed with acetone and dried. The weight loss data was estimated using the equations below[9-10]:

$$CR = \frac{\Delta W}{D \times A \times T} \times 87600 \quad \text{... (1)}$$

$$\eta\% = \frac{W_{\circ} - Wi}{W_{\circ}} \times 100 \quad \text{... (2)}$$

Table 1 — Constituents of low carbon steel coupons

S. No.	Metals present	% composition
1	Iron	99.2
2	Silicon	0.120
3	Carbon	0.105
4	Manganese	0.378
5	Phosphorus	0.079
6	Sulphur	0.0798

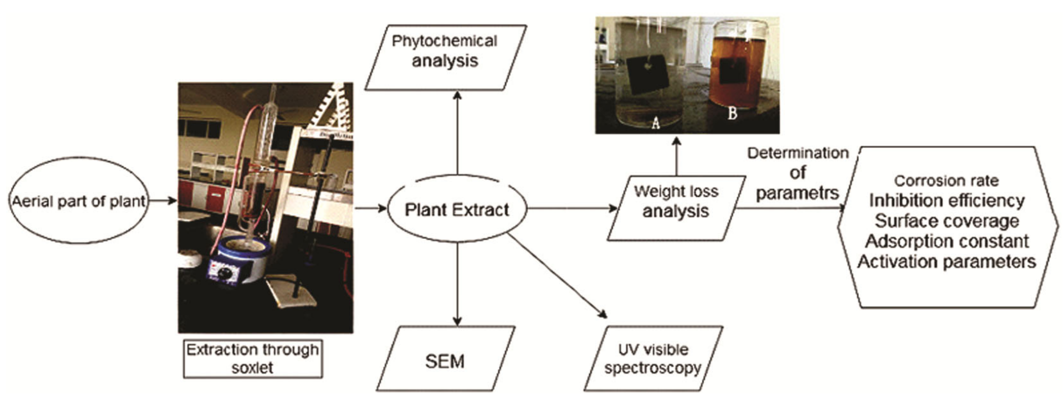

Fig. 1 — Pictorial representation of methodology.

$$\theta = \frac{W_{\circ} - Wi}{W_{\circ}} \quad \text{... (3)}$$

CR= Corrosion rate, Ƞ%= Inhibition efficiency, ΔW → weight loss (mg), A → area of mild steel coupon (cm^2), T → time of immersion, D → denotes density of mild steel strip (gcm^{-3}), (w_0 and w_i) → L Weight loss in the absence and involvement of inhibitors respectively, θ =surface coverage

2.5 Adsorption isotherm

To study the adsorption behavior, Langmuir isotherm can be applied with respect to data obtained in weight loss analysis. This isotherm is a plot of C/θ vs. logC. Adsorption is calculated by the equation 4:

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad \text{... (4)}$$

Where C is concentration of inhibitor, θ is surface coverage and K_{ads} is the adsorption equilibrium constant.

2.6 Phytochemicals analysis

The aqueous extract of *Lepidium didymiu*m was tested for phytochemicals investigation of alkaloids, Flavonoids, saponins, quinones, Coumarin, and sugar. Phytochemicals investigation of a crude concentrate of *Lepidium didymiu*m was carried out. The stock solution was prepared by dissolving 0.25 g of concentrate in 100 mL water and afterward used for phytochemicals investigated. Various tests were performed for the conformation of phytochemicals.

2.7 UV-visible spectroscopic analysis

The UV spectrum of *Lepidium didymiu*m extract in 1M H_2SO_4 was acquired using a UV-Visible absorption spectrophotometer.Shimadzu's UV-1900 as shown in Fig. 2 was utilised to characterise the extracts. The spectrum was obtained under two conditions: a solution comprising inhibitors into which the mild steel specimens were not submerged, and a solution where the steel specimens were soaked for 24 hrs. To understand out how inhibition works, each of the spectra was analyzed[11].

2.8 Surface inspection by Scanning Electron Microscope (SEM)

The surface morphological changes were monitored using a scanning electron microscope (SEM) as in Fig. 3 with a 20 keV accelerating voltage. SEM pictures of mild steel are used to assess the sample's surface morphology with pretreated mild steel specimens were soaked in a 1 M H_2SO_4 solution lacking extract and with 2500 mg/L extract for 24 hours before being washed with ultrapure water and acetone and then dried. The surface morphologies of these pieces were then investigated using a scanning electron microscope.

3 Result and Discussion

3.1 Weight loss measurements

Weight loss values illustrates in Table 2 at varying concentration of *Lepidium didymiu*m extract, corrosion rate (CR), inhibitory efficiency (%) and surface coverage (θ) for mild steel were estimated from weight loss by employing the expressions.

As clearly visible from the values of Table 3, there is a rise in inhibition efficiency with rise in concentration as outline in Fig. 4 and simultaneously surface coverage

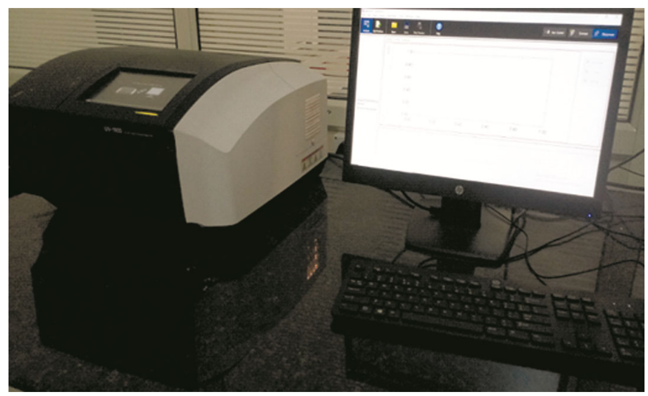

Fig. 2 — UV-Visible spectrophotometer.

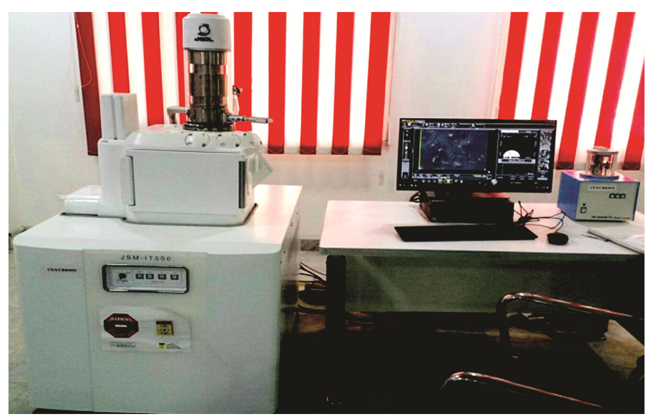

Fig. 3 — Scanning Electron Microscope.

Table 2 — Data of inhibition efficiency and surface coverage of *Lepidium didymiu*m in1 M H_2SO_4.

Corrosive medium	Inhibition conc. (mg/L)	Efficiency (%)	Surface coverage (θ)	Corrosion rate ($cm^{-1}h^{-1}$)
	200	42	.42	230.92
1M H_2SO_4	600	78.97	.7897	91.72
	1000	79.46	.7946	81.50
	1500	81.31	.8131	74.19
	2500	91.16	.9116	35.06

increases gradually as the concentration of inhibitors increases also outline in Fig. 5. This rise is possible only if heteroatoms and multiple bonds adsorbed on the exterior part of mild steel and decrease the pace of the corrosion of metal with the aggressive medium. As with increase in concentration of inhibitor corrosion rate also decreases shown in Fig. 6.

Table 3 — for mild steel in 1 M H_2SO_4 *in absence and presence of Lepidium didymiu*m plant extract.

S. No.	Inhibitor	Inhibitor conc. (mh/L)	ΔH_a (kJ/mol)	ΔS_a (J/mol/ K)
1	*Lepidium didymiu*m	0	8.59	20.12
		2500	62.99	220.21

3.2 Adsorption isotherm

Figure 7 shows the association between the surface coverage (C/θ) and the concentration of *Lepidium didymium* extract. The plot obtained is linear, and the plot's intercept allows for the determination of the

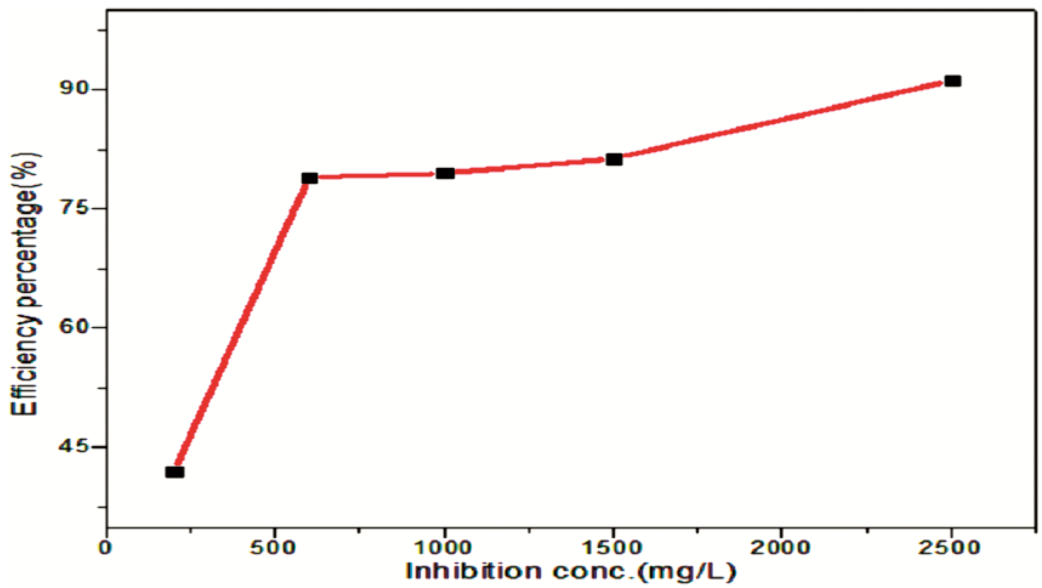

Fig. 4 — Corrosion inhibition efficiency of *Lepidium didymiu*m extract at different concentrations.

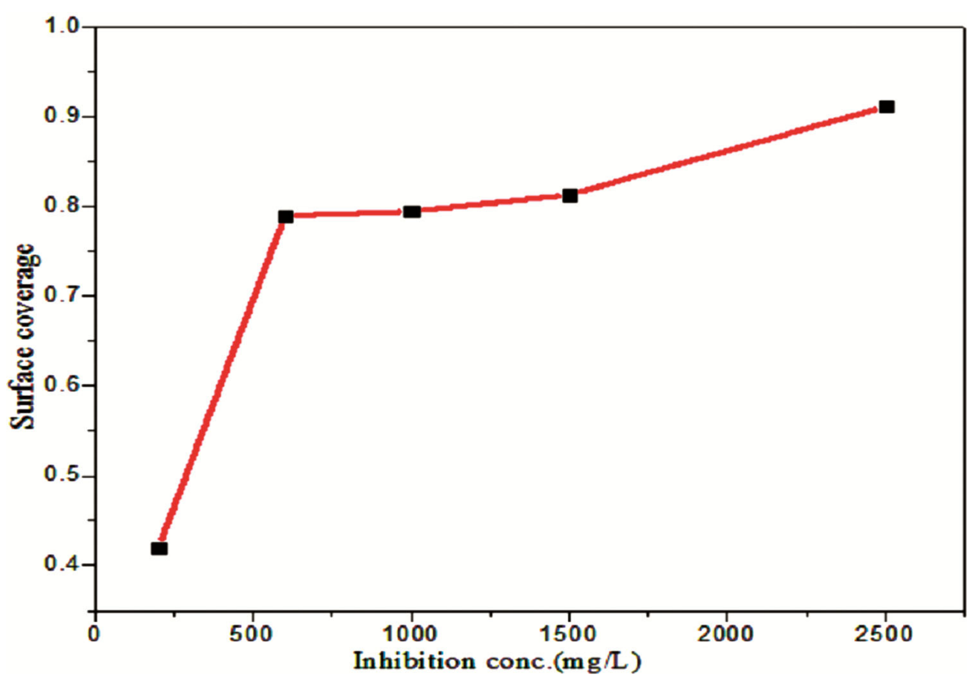

Fig. 5 — Surface coverage with extract of *Lepidium didymiu*m at different concentrations.

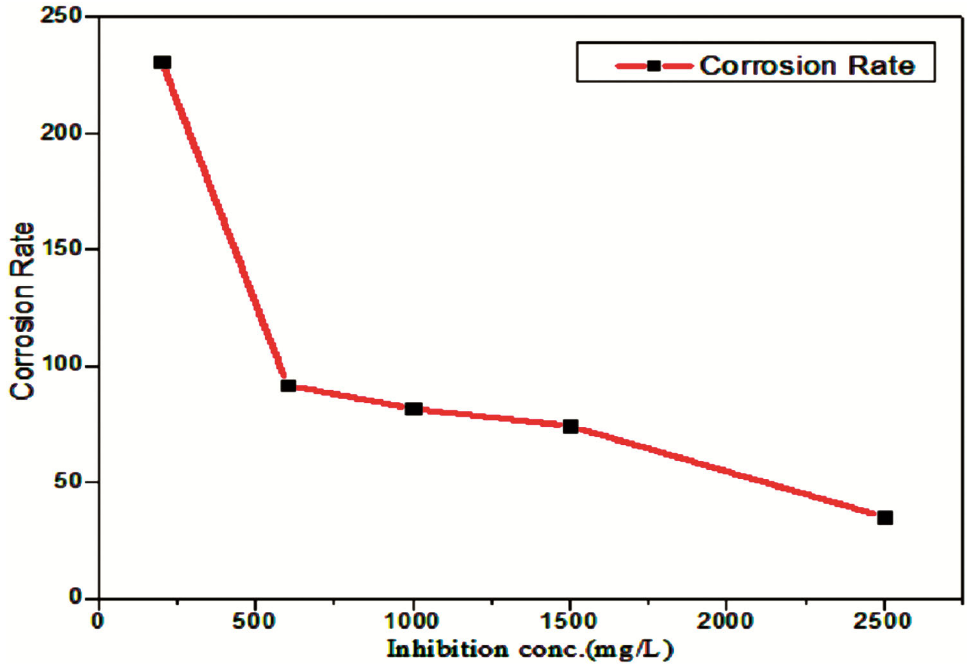

Fig. 6 — Corrosion rate with extract of *Lepidium didymiu*m at different concentrations.

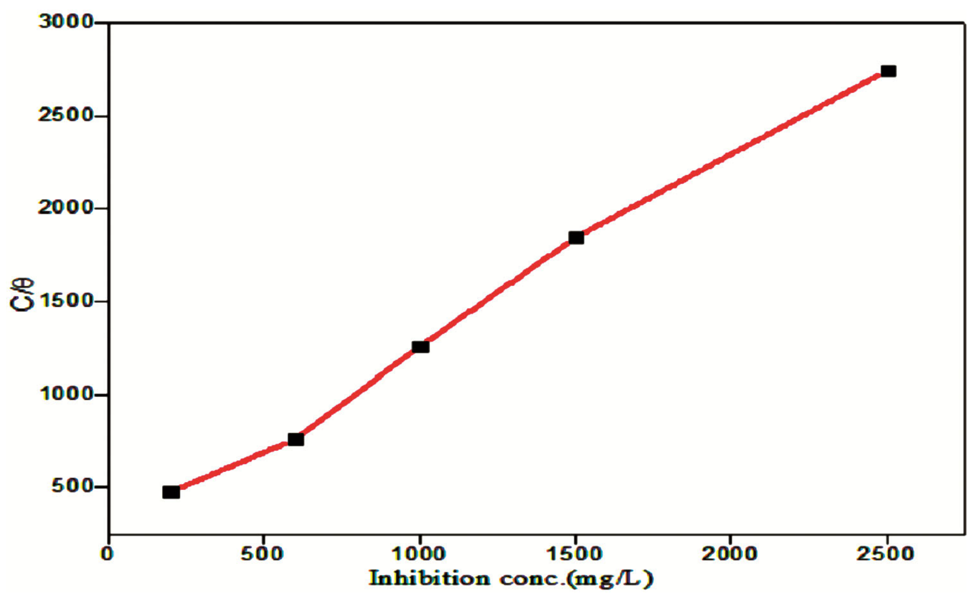

Fig. 7 — Adsorption isotherm for *Lepidium didymiu*m on low-carbon steel in 1 MH_2SO_4.

adsorption equilibrium constant (K_{ads}). At 298 K, 308 K, and 318 K, K_{ads} values are 4.12, 1.789, and 0.59 L/mg, respectively. The fact that K_{ads} decreases as temperature rises suggests that adsorption becomes reversed (desorption).

The K_{ads} values are used to calculate the ΔG_{ads} using the following formula:

$$\Delta G^{\circ}_{ads} = -RTln(55.5 \times K_{ads}) \quad \ldots (5)$$

Where R is the gas constant, T is absolute temperature; $\Delta G°ads$ is standard free energy of adsorption. The determined values of ΔG°_{ads} can be calculated using this equation is -13.45, -11.75, -9.2 KJ Mol^{-1} at 298 K, 308K and 318K respectively. It can be estimated that whether adsorption of inhibitor molecules on the surface occurs through the establishment of a chemical bond or through physical adsorption based on the ΔG°_{ads} values. The measurements of ΔG°_{ads} in this case show that the *Lepidium didymium* extract physically adsorbs on the steel surface.

3.3 Activation parameter

Figure 8 displays plot of log (corrosion rate) vs. 1000/T for metal dissolutions without and with varying inhibitor concentrations, respectively from which E^a is computed. The Arrhenius law predicts that the rate of metal corrosion increases as the temperature rises, with E^a fluctuating according to equation:

$$E^a = -Slope \times 2.303 \times 8.314 \quad \ldots (6)$$

E^a was determined to be 8.99 kJ/mol without such inhibitor, but when the inhibitor was added, it raised to 60.23 kJ/mol, showing physical adsorption of the inhibitor on the steel surface.

3.4 Entropy and enthalpy adsorption parameter

Entropy and enthalpy of adsorption can be calculated with the help of following equation:

$$log\left\{\frac{CR}{T}\right\} = log\left\{\frac{R}{N_a H}\right\} + \frac{\Delta S_a}{2.303R} - \frac{\Delta H_a}{2.303RT} \quad \ldots (7)$$

CR stands for corrosion rate, T stands for absolute temperature, h stands for Planck constant, and Na stands for Avogadro's number, ΔS_a is standard entropy of activation, ΔH_a is enthalpy of activation, R gas constant. By graphing log(CR/T) against 1000/T as shown in Fig. 9 the parameters of standard enthalpy of activation and standard entropy of activation have been computed and the results are presented in Table 3. The higher ΔH_a in the presence of an inhibitor (62.99 kJ/mol) than in the absence of an inhibitor (8.59 kJ/mol) suggests that the metal is better protected, presumably attributed to the presence of an energy barrier to the corrosion reaction. As a result of the inhibitor adsorption, the increase in enthalpy of the corrosion process. In comparison to the case of the blank solution entropy value (20.12 J/mol/ K), the value of ΔS_a (220.21 J/mol/ K) increased with the addition of the inhibitor. As move from reactant to activated compound, the entropy value increases, indicating that disorder is increasing.

3.5 Phytochemical analysis

Table 4 elaborates the presence of phytochemicals in the extract *Lepidium didymium*.

3.5.1 For alkaloids

Wagner's test: Wagner's reagent was used to treat the extract. The existence of alkaloids is suggested by the formation of brown/reddish ppt[12].

Mayer's Test: For this test of alkaloids filtrates were subjected to Mayer's reagent. Yellow colour is turn out[12].

3.5.2 Test for Flavonoids

A small quantity of the plant material extracts was mixed with several drops of concentrated HCl. The existence of flavonoids was determined by the rapid production of a red color[13].

3.5.3 Test for Saponins

With vigorous shaking, five ml of water is combined with five ml of filtrate (aqueous extract). The presence of the saponins suggested by the formation of stable froth[14].

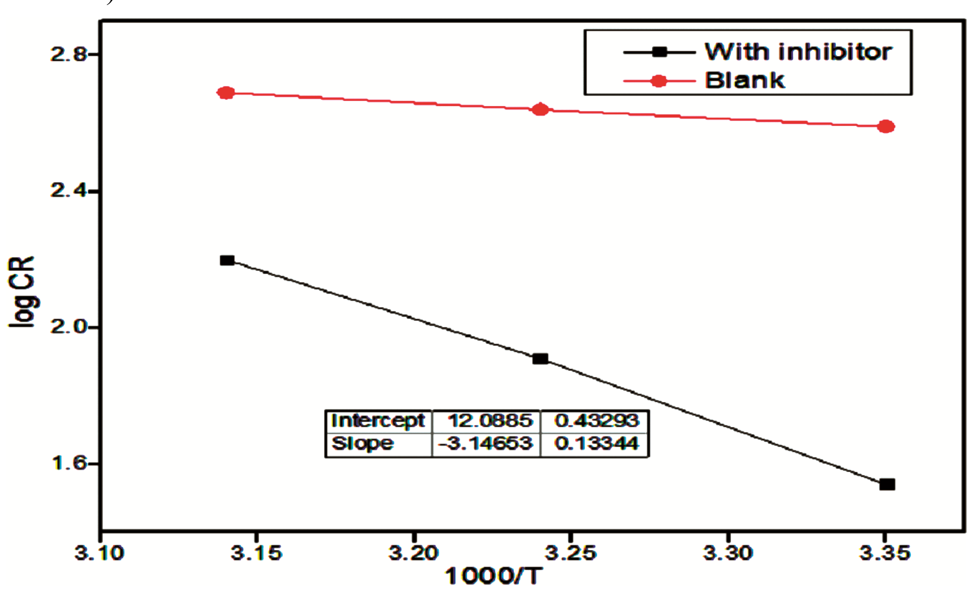

Fig. 8 — Plot of log (Corrosion rate) vs. 1000/T for metal dissolutions without and with extract.

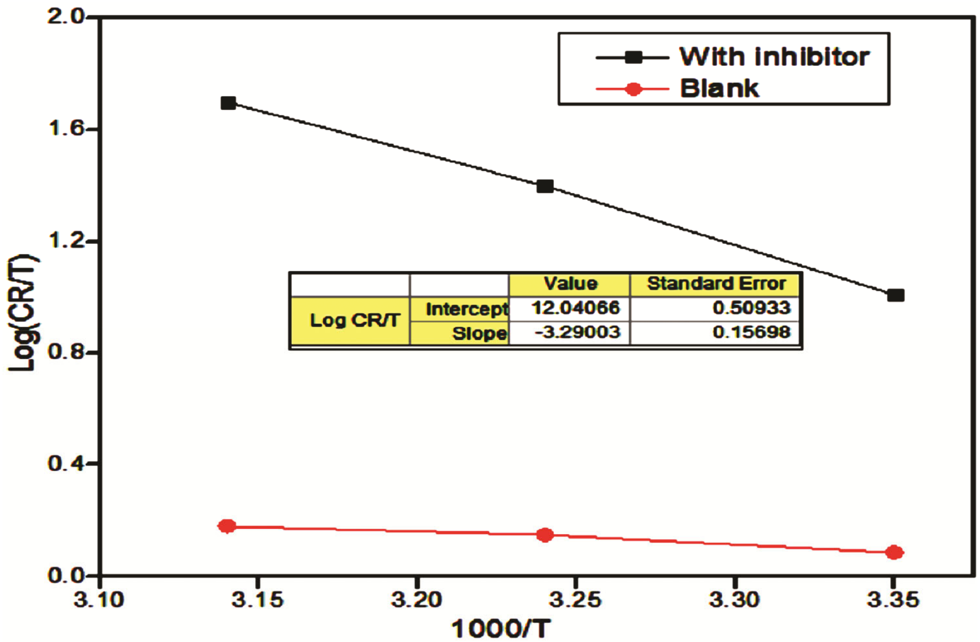

Fig. 9 — Plot of log corrosion rate/T vs. 1000/T for metal dissolutions without and with *Lepidium didymiu*m extract.

Table 4 — Result of phytochemicals of *Lepidium didymiu*m.

S. No.	Phytochemicals	Test	Result
1	For alkaloids	Wagner's test	- -
		Mayer's Test	- -
2	For flavonoids	Conc. hydrochloric acid test	+ +
3	For saponons	Froth test	- -
4	For quinones	Concentrated sulphuric acid test	+ +
5	For coumarins	Alcoholic NaOH test	- -
6	For sugar	Fehling solution test	+ +

3.5.4 Test for quinones

The presence of quinones was demonstrated by mixing 1 mL of the extract with concentrated H_2SO_4; the color change proves the existence of quinones[15].

3.5.5 Test for Coumarin

A few drops of alcoholic NaOH solution were added to 2 ml of the extract. The occurrence of Coumarin is confirmed by the formation of the yellow color[15].

3.5.6 Test for sugar

Fehling solution test: Fehling's solution A and B were put together in one Ml proportion and boiled for one minute. To that mixture, 1 ml of an extract was added up and heated on the water bath for about 5- 10 minutes. The presence of carbohydrates was indicated by the appearance of yellow and then brick red precipitates.

3.5 UV Visible spectroscopy

*Lepidium didymiu*m's UV spectrum is investigated before and after the corrosion process and from the spectra, it is clear that the absorbance of the aggressive medium containing inhibitor in which mild steel is not yet dipped is higher than the medium in which steel specimens dipped for 24 hours as shown in Fig. 10. So it shows that when steel specimen is immersed in a corrosive medium that contains inhibitor, phytochemicals of that inhibitor adsorbed on the top of the metal to construct a defensive coat for acidic medium and there is the formation of complex in between exterior part of steel and inhibitor[32].

3.6 Scanning Electron Microscope (SEM)

Figure 11 elaborated the changes in the morphology of cleaned mild steel surfaces after 24 hours of immersion in 1 M H_2SO_4 with and without extract. Figure 11(a) depicted that the refined mild steel surface was not homogeneous all over and had little imperfections that could operate as a focus point for corrosion reactions to begin. The quick and strong corrosion reactions during the immersion of mild steel in blank H_2SO_4 badly damaged the metal in several areas, as evidenced by the highly rough surface as depicted in Fig 11(b). The presence of an inhibitor, on the other hand, slowed the corrosion of mild steel in 1 M H_2SO_4 and less surface damage as shown in Fig. 11(c) explained the establishment of a defensive inhibitor coating on the surface of mild steel.

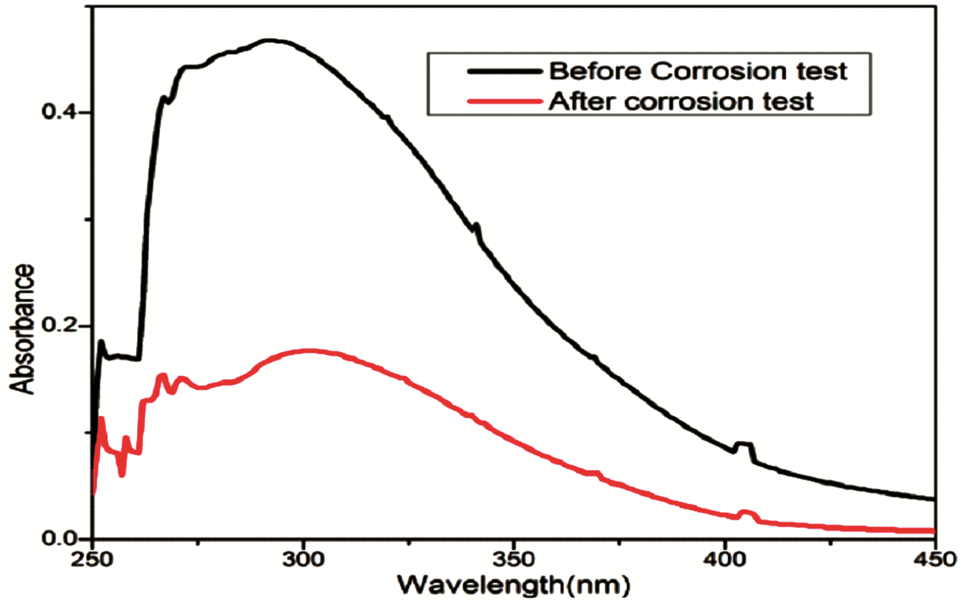

Fig. 10 — UV Spectrum of *Lepidium didymiu*m's extract before and after the corrosion inhibition performance.

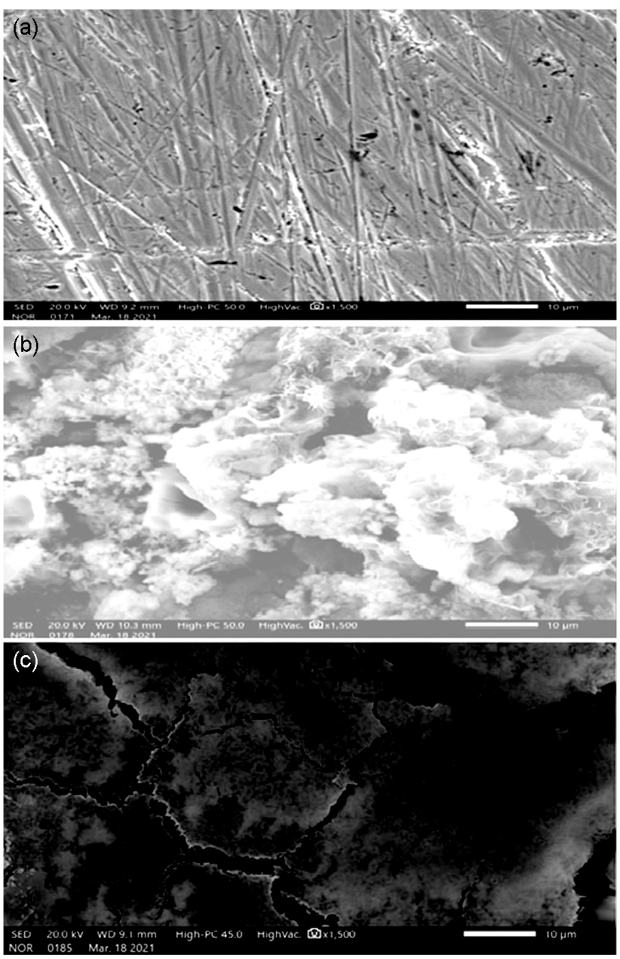

Fig. 11 — Surface pictures of (a) cleaned mild steel (b) corroded in 1 M H_2SO_4, and (c) inhibited by 2500 mg L-1 *Lepidium didymiu*m's extract.

3.7 Corrosion inhibition mechanism by Lepidium didymiumon mild steel

Corrosion is caused by two types of electrochemical reactions: oxidation on the anode side and reduction on the cathodic side, which is illustrated by the reaction below:

$$Fe \rightarrow Fe^{2+} + 2e^{-} \quad \text{... (1)}$$

$$2H^{+} + 2e^{-} \rightarrow H_2 \quad \text{... (2)}$$

The heteroatom's lone pair transfers electrons from the inhibitor towards the metal, producing co-ordinate bond[16-17]. *Lepidium didymium* extract contain phytochemicals having different functional groups like carbonyl group, hydroxyl group as well as carboxylic acid and also double bonds that can efficiently reduce mild steel corrosion in 1M sulfuric acid[18]. The heteroatom's lone pair and double bonds present in phytochemicals form co-ordinate bond with metal as shown in Fig 12. The inhibitory action of plant extract is caused to natural compound adsorption on the metal surface, resulting in a blockage of the active site. Because a protective film has formed on the surface, mild steel is no longer available for corrosion[19].

3.8 Comparison study with other natural inhibitors

Inhibition efficiency of *Lepidium didymiu*m extract compared with other natural corrosion inhibitors from literature, like K. Hassan[20] demonstrated the efficiency of extract of leaves of *Citrus aurantium* showing 89% at 10000 ppm in 1M H_2SO_4 due to presence phenolic compounds. Similarly, M. Prabakaran[21] worked upon

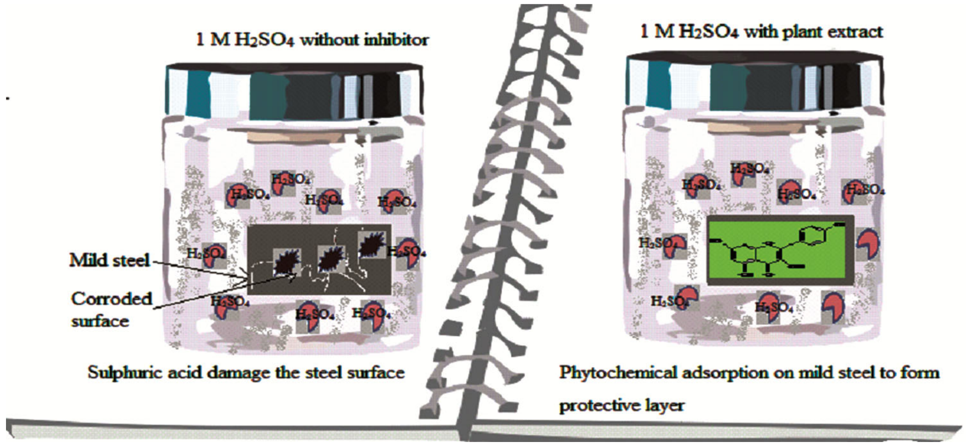

Fig. 12 — *Lepidium didymium*'s phytochemical adsorption on mild steel to form protective coating.

Cryptostegia grand flora's leaves with inhibition efficiency 87% at 500 ppm due to Hydroxyl cinnamic acid as active phytochemical.

4 Conclusion

Plant extract acts as an excellent steel corrosion inhibitor in a 1 M H_2SO_4 solution at low concentration. The weight loss method was used to assess the corrosion resistance of steel dipped in sulfuric acid prepared in distilled water, both in the absence and presence of an aqueous extract.

- Corrosion inhibition efficiency appears to improve in the presence of a *Lepidium didymium* extract.
- The inhibitor's inhibition efficiency results show 91.16 % inhibition efficiency at a concentration of 2500 ppm.
- The effectiveness of inhibition is influenced by the different phytochemicals present in the extract.
- As the inhibitor concentration is increased, the inhibition efficiency improves.
- The results of the SEM analysis suggested that the mechanism of corrosion inhibition is purely based on adsorption.

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