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CORROSION INHIBITION OF *ELETTARAI CARDAMOM* BARK EXTRACT ON MILD STEEL IN 1M H_2SO_4

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ABSTRACT

Elettaria cardamom (Alaichi) is a spice available in Nepal, from the bark of which alkaloid can be extracted to use as a corrosion inhibitor. The corrosion inhibition of methanolic extract of Elettaria cardamom (EC) for mild steel (MS) in 1M H_2SO_4 was studied using the weight loss method. The effect of time of immersion and concentration of EC extract on corrosion inhibition was investigated. The result showed that the corrosion inhibition efficiency increased with an increase in the time of immersion and concentration of the EC extract. The maximum inhibition efficiency of 1000 ppm EC extract was 93.36% for 3 hours of immersion in 1M H_2SO_4 solution. FTIR spectroscopic analysis showed the presence of functional groups containing oxygen and nitrogen in the extract, which is responsible for the adsorption of the extract onto mild steel and reduces the corrosion rate.

Keywords: Corrosion inhibition, *Elettaria cardamom*, Mild steel, FTIR spectroscopic analysis, Corrosion inhibition efficiency

INTRODUCTION

Plant extracts are the potential candidate to substitute synthesized organic and inorganic inhibitors. Globally, a clean and safe environment is a significant concern. Currently, natural products extracts as green and eco-friendly inhibitors are significantly employed for the corrosion prevention of metals and alloys (Afia et al., 2014; Medupin et al., 2023; Okafor et al., 2012; Raja & Sethuraman, 2009; Stiadi et al., 2020). Green corrosion inhibitors are plant extracts containing alkaloids, polyphenolic compounds, flavonoids, etc., that, when introduced in small amounts to corrosive media, slows down the rate of corrosion of metals. Green inhibitors are renewable, inexpensive, effective at low concentrations, and safe with other medium components at working temperatures(Cáceres et al., 2021; Karki et al., 2020; Stiadi et al., 2020). Therefore, organic and inorganic inhibitors are being reduced and eventually abandoned. Photochemicals are found to have nitrogen, oxygen, sulfur, and phosphorous, resulting in a shielding layer by physical or chemical adsorption on the metal surface, which reduces the deterioration of metal (Abdallah et al., 2018; Arab & Noor, 1993; Behpour et al., 2012; Belafhaili et al., 2023; Karki et al., 2023; Medupin et al., 2023). The inhibitors obtained from natural products address the advantages of both organic and inorganic substances. The pertinent literature shows that natural products have extensively been applied in controlling corrosion.

The various parts of plant extract are used as green corrosion inhibitors to protect metals and alloys from their surroundings. Lantana camara (Shrestha et al., 2019), Euphorbia royleana (Thapa et al., 2019), Artemesia vulgaris (Karki et al., 2018), Jatropa curcas (Gupta, Kafle, et al., 2020) Eucalyptus globulus(Gupta, Awasthi, et al., 2020) Equistem helyme (Karki et al., 2021) Elettarai cardamomum(Tarfaoui et al., 2021) extracts have been investigated as a green corrosion inhibitor,

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which shows adequate corrosion protection with high inhibition efficiency. Thermodynamically they offer physisorption (Srivastava & Srivastava, 1981). Faisal et al. (2018) explored the tobacco, black pepper, caster seeds oil, acacia gum, and lignin are suitable green inhibitors in acidic media for steel. When Lavandula mairei Humbert extract was employed by Berrissoul et al. as a new eco-friendly corrosion preventative material for MS in HCl, 0.4 g/L of the extract concentration revealed the highest inhibition efficacy that was discovered 92% at 303 K. Thomas et al. investigated that Garcinia indica fruit peel aqueous extract acted as potential inhibitor for a mild steel (MS) in an acid. Plant extracts of cardamom (dried stems, leaves) and other high-altitude plants were used in H2SO4 pickling baths as early as 1930 (Gellings, 1985). The effectiveness of Elettaria cardamomum essential oil (EO) was studied by Tarfaoui et al. (2021) as an inhibitor for MS in 1.0 M HCl by potentiodynamic polarization, electrochemical impedance spectroscopy (EIS), and computational studies. The result showed that the essential oil prevented mild steel corrosion, and its prevention efficacy rose with concentration. At a 2 g/L of inhibitor concentration, Elettaria cardamomum demonstrated a remarkable 88% inhibitory efficiency.

In order to prevent copper alloy—zinc corrosion in the center (brass) in 1 M HCl solution, Nadeem et al. (2016) studied the essential oils derived from the *Nigella Sativa and Elettaria Cardamomum* seeds. The results illustrated that reduction in the corrosion current density from 0.204 mA/cm2 to 0.042 mA/cm2 and 0.056 mA/cm2 with the addition of Nigella sativa and Elettaria cardamomum oils, respectively. Electrochemical impedance spectroscopy (EIS) showed that the charge transfer resistance increased from 383.6 ohm cm2 to 1871 ohm cm2 and to 996 ohm cm2 with the addition of oil of Nigella Sativa and *Elettaria cardamomum*, respectively. These inhibitors were employed to demonstrate their significant efficacy in preventing corrosion of copper-zinc alloy in acid solution (HCl). They yielded results ranging from 72.5 to 80% for the oil of *Nigella sativa* and *Elettaria cardamom*.

In contrast, the growth of a film on the brass surface is due to the adsorption of these molecules on the alloy. The pharmacological effects of these plants essential oil reveal antioxidant, anti-diabetic, antibacterial, anti-cancer, gastro-protective, and insecticidal properties (Ahmed et al., 2019; Tarfaoui et al., 2021). Asthma and cardiovascular disease are treated using Elettaria cardamomum's fragrant seeds. 1,8-cineole, α-terpinyl acetate and linalool are the main compounds (Ashokkumar et al., 2020; Tarfaoui et al., 2021). Since the active ingredient's composition determines how green inhibitors work, numerous researchers have proposed various theories to account for this occurrence. One approach is that the active substances result in onium ions in acidic solutions getting adsorbed on the metal surface's cathodic sites, which obstruct the cathodic reaction.

Elettaria cardamom, known as alaichi in Nepal, is used as a spice. The plant is inhabitant to Nepal, India, and Srilanka and is widely planted around Taplejung, Panchthar, and Terhathum in Nepal. It is an aromatic, sweet-smelling, herbaceous, persistent plant, growing to about 2-4 m in height. The leaves have a long pointed tip and alternate in two ranks. Cardamom usually grows in the tropical region. The seeds and the oil are very useful to formulate medicine. It has been using for thousands of years to facilitate digestion. Cardamom is mixed with other medicine and used to relieve discomfort, vomiting, and nausea. Furthermore, it treats the common cold, cough, sore throat, mouth, and the tendency to get infected (Ahmed et al., 2019; Ashokkumar et al., 2020). Nepal is rich in high-altitude plants, and there is a high possibility that such plant extract can work as an efficient corrosion protection coating on active metal such as steel. Very few natural products of Nepali origin have been investigated as corrosion inhibitor. *Elettaria cardamomum*, Zingiberaceae family is known for containing oil in its seeds. Phytochemical screening of *Elettaria cardamom* has shown that the plant contains various chemical constituents like alkaloids, steroids, saponins, glycosides, reducing sugar, and amino acids (Ahmed et al., 2019). In this context, the primary goal of this research is to examine how effectively EC extract prevents mild steel from corroding in an acidic environment because it is renewal, natural and environmentally safer resource.

MATERIALS AND METHOD

Preparation of Extracts

Stems of Elettaria cardamom (EC) were collected from the Phulbari-07 area of Taplejung (Latitude: 27°21'0"N and Longitude 87°40'0"E). The barks of stems were then peeled off and dried in a shaded area for about a month. The dried bark was ground into fine powder by milling it in the grinding mill of Tribhuvan University.

The powdered sample was steeped in hexane for two days and then performed filtration. The residue was again washed with hexane and filtered. The residue was then mixed with methanol for seven days and filtered. The filtrate was made basic by adding NH4OH and later acidified by 3% tartaric acid to separate the alkaloids from non-alkaloid compounds. The methanol extract was then transferred into a separating funnel, and an appropriate amount of acetone was added. The solutions were gently mixed with an occasional release of pressure from the funnel. Two separate layers of liquid were observed. The top layer was aqueous, and the bottom layer was an organic layer. The organic layer was slowly removed from the funnel and put into the rotator evaporator. *Elettaria cardamom* (EC) extract was obtained as residue when concentrated at 65°C. The concentrated alkaloid extract was dried by evaporation in a water bath at 65°C.

Preparation of EC inhibitor solution

1L of 1M H_2SO_4 solution was prepared. 0.25g of EC extract was dissolved in 1M H_2SO_4 solution to prepare 1000 ppm of 250 mL EC extract solution. Likewise, 800 ppm, 600 ppm, 400 ppm, and 200 ppm of the EC inhibitor solution were prepared from 1000 ppm inhibitor solution by serial dilution in 1M H_2SO_4.

Preparation of Mild Steel Sample

A mild steel (MS) sheet was procured from a local market in Kathmandu and cut into small pieces of 3 cm x 3 cm. MS samples were then polished with SC paper of different grits (#100 to 1200). Each of the samples was cleaned with hexane separately, washed with ethanol, and dried with a blower.

Weight Loss Method

All the MS samples were weighed in a digital balance of 4 digits, and weights were noted. The samples were dipped separately in 1M H_2SO_4 solution and 1000 ppm of EC extract solution. The loss in weight of MS was estimated from the difference in weight before and after the immersion in acid and inhibitor solution. Likewise, the consequence of EC extract concentration was investigated by immersing MS in acid and different EC concentrations for 3 hours (200, 400, 600, 800, and 100 ppm). The corrosion rate (CR), surface coverage (θ), and inhibition efficiency (IE%) were estimated by the following relation.

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

Where,

W = weight loss of the mild steel after time, t (gram)

A = area of the mild steel sample (cm)

t = time of immersion (hour)

d = density of mild steel (g cm^{-3})

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

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

Where,

W_o = weight loss for the mild steel in the absence of an inhibitor

W_i = weight loss for the mild steel in the presence of an inhibitor.

a b

Figure 1: Mild steel sample in (a) inhibitor solution and (b) 1M H2SO4

Characterization by FTIR

Fourier transmission infrared (FTIR) spectra were studied using an IR prestige 21-FTIR instrument (by Shimadzu, Japan). EC extract was dissolved in H2SO4 solution, and IR spectra were recorded. FTIR characterization of the extract was performed to identify its functional groups.

RESULTS AND DISCUSSION

Phytochemical Screening Analysis

The presence of alkaloids was confirmed by the phytochemical screening of the *Elettaria cardamom (EC) extract*

Table 1: Results of phytochemical screening test for alkaloids.

Mayers Test	Wagners Test	Dragendroffs Test
+	+	+

Effect of Time of Immersion

The results of the weight loss experiment for mild steel (MS) samples immersed in 1M H_2SO_4 and EC extract solution were studied. Table 2 reveals the weight loss of MS, corrosion rate, surface coverage, and inhibition efficiency of 1000 ppm inhibitor solution for different times (1, 2, 3, 4, 5, and 6 hours). Figure 2a represents the variation of corrosion rate with immersion time with and without EC inhibitor solution. Inhibitor solution considerably reduced the corrosion of MS in 1M H_2SO_4. Figure 2b shows the variation of inhibition efficiency of EC with time. The inhibition efficiency increased with the time of immersion. The maximum inhibition efficiency of 90.98% was obtained in 6 hours of immersion. EC extract decreased the corrosion rate due to the adsorption of organic molecules present in the extract on the surface of MS forming a passive layer that resulted in a protective inhibitive effect (Tarfaoui et al., 2021).

Table 2: Corrosion rate of MS, surface coverage, and inhibition efficiency of *EC* at different times

Time (hours)	Corrosion Rate (mm/year)		Surface Coverage (θ)	Inhibition Efficiency%
	For acid	For inhibitor solution		
1	86.75	13.68	0.842	84.23
2	62.07	8.48	0.863	86.33
3	62.19	7.01	0.887	88.72
4	48.3	5.32	0.889	88.98
5	42.6	4.33	0.898	89.83
6	40.45	3.65	0.909	90.98

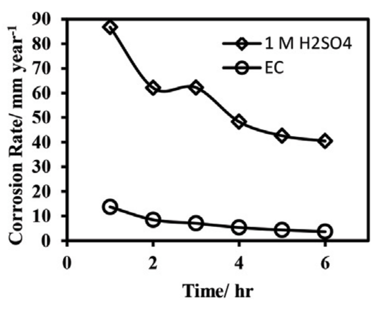

Figure 2a: Variation of corrosion rate with immersion time

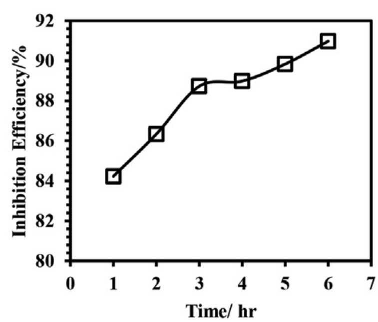

Figure 2b: Variation of inhibition efficiency of EC with time

Effect of EC Inhibitor Concentration

Table 3 shows the weight loss of MS, corrosion rate, surface coverage, and inhibition efficiency of various concentrations of EC extract for a 3-hour immersion. Table 3 demonstrates that the surface coverage of MS increased with an increase in concentration and consequently dropped the corrosion rate. Figure 3a depicts the variation in corrosion rate as a function of the EC extract solution. Due to the significant surface coverage of MS, the corrosion rate was greatly decreased by 1000 ppm EC extract solution, which also lessened the reactivity with acid. Figure 3b depicts the variation of inhibition efficiency with a concentration of EC solution. The effectiveness of the inhibition increased with the increase in the concentration of EC extracts (Nadeem et al., 2016; Tarfaoui et al., 2021) because the MS surface was almost entirely covered by 600 ppm inhibitor solution. The inhibitory action is enhanced gradually. The release of H2 gas and the dissolution of the metal typically accompany metal corrosion in acidic environments. The organic molecule adsorption on the metal surface causes the EC extracts corrosion inhibitory action against MS. The adsorption prevents the corrosion sites of the metal, which restricts the breakdown of the metal, hence reducing weight loss. After a 3-hour immersion, the inhibitory efficacy of 1000 ppm EC extract solution is about 94%.

Table 3: Corrosion rate of MS, surface coverage, and inhibition efficiency of *EC* at concentrations

Concentration (ppm)	Weight Loss(g)	Corrosion Rate (mm/year)	Surface Coverage(θ)	Inhibition Efficiency %
Acid	0.3013	62.18		
200	0.14	28.89	0.5353	53.53

400	0.1	20.63	0.6681	66.81
600	0.06	12.38	0.8008	80.08
800	0.04	8.25	0.8672	86.72
1000	0.02	4.12	0.9336	93.36

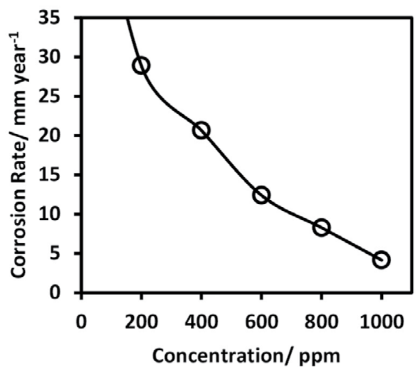

Figure 3a: Variation of corrosion rate with concentrations for 3 hours

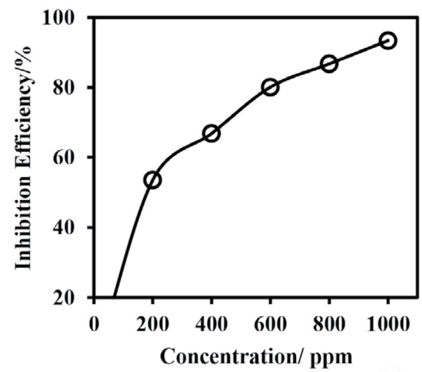

Figure 3b: Variation of inhibition efficiency of EC with concentrations

FTIR Spectroscopic Analysis

Figure 4 shows the FTIR spectra of *Elettaria cardamom* (EC). The absorption peaks reveal the presence of different functional groups in the EC responsible for the adsorption of the extract on the MS. The absorption band obtained from the FTIR spectrum showed broadband at 3207 cm-1, representing O-H's stretching mode. The peaks at 1583 cm-1 showed the stretching C- N, and the peak at 1418 cm-1 represented C-H bending. Similarly, at 1068 cm-1 sharp peak showed a C-O bond. The organic molecules in EC that are corrosion inhibitors can bond directly to create a stable complex via adsorption on the MS surface (Tarfaoui et al., 2021)

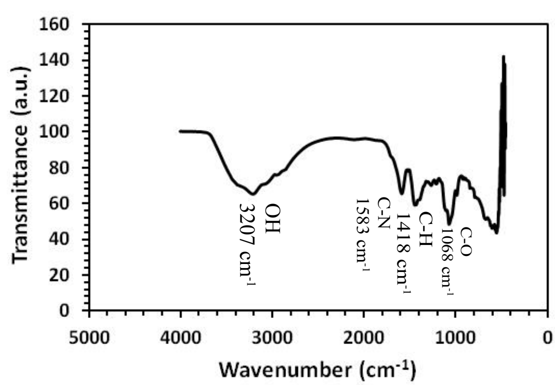

Figure 4: FTIR spectra of *Elettaria cardamom* (EC)

CONCLUSION

The study reveals that the bark extract of *Elettaria cardamom* (EC) is an efficient green corrosion inhibitor in 1 M H_2SO_4. The weight loss of MS is significantly reduced by EC bark extract due to the adsorption of organic molecules present in the EC. The corrosion rate of MS is reduced with the increase in the inhibitor concentration and time of immersion in 1M H_2SO_4. The utmost inhibition efficiency of 1000 ppm EC extract is 93.36% for 3 hours of immersion in an acidic solution. FTIR analysis confirms that the adsorption of EC extract on MS is due to the presence of functional group-containing oxygen and nitrogen, resulting in the protective barrier layer.

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