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Surface Protection of Steel in Acid Medium by *Flacourtia jangomas* extract : Phytochemicals and Surface Study Evidence for Adsorption of Inhibitor

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ABSTRACT: We present the evaluation of eco-friendly corrosion inhibitor for the corrosion mitigation of mild steel using extract of leaves, roots and stem bark of coffee plum *(Flacourtia jangomas)* in 1M HCl solution was investigated by weight loss method at 30°C. The corrosion inhibition of mild steel by ethanol extracts coffee plum *(Flacourtia jangomas)* and some of its isolated phytochemical components; saponins extract, and flavonoids extracts have been studied using gravi-metric techniques. The surface of mild steel was examined by scanning electron microscopy (SEM) and Atomic force microscopy (AFM) techniques. The results of the study reveal that these ecofriendly extracts function as good inhibitors for mild steel corrosion in hydrochloric acid. Inhibition efficiency of the extracts increases with inhibitor concentration and temperature rise. Corrosion rate decreased significantly in presence of extract and inhibition efficiency increased with increasing concentration of extract. Inhibition efficiency performance followed the trend Leaf extract > Root extract > Stem Bark extract at 5% inhibitor concentration. No considerable changes in inhibition efficiency were observed above 5% in all the cases. The decreased corrosion rate and adsorption behavior have been explained by Langmuir, Temkin and Freundlich adsorption isotherm. The constituents responsible for inhibition were identified by Phytochemical analysis and Fourier Transform Infrared Spectroscopy FTIR spectroscopy.

Keywords: Coffee plum *(Flacourti jangomas)*, corrosion inhibitor, mild steel, AFM, SEM, weight loss, Langmuir, Temkin isotherm, FTIR.

INTRODUCTION

Steel is widely used iron-carbon alloy for construction of articles like pipes etc. structures and utensils of everyday use in our daily life. We prefer steel because of its strength and cheap rate. Metallic corrosion is a natural phenomenon which possess very serious problem in industries (Da Rocha *et al*., 2012). Corrosion of steel especially in acid medium is the most common form corrosion and directly impacts its cost and safety. From the previous data it was found that metallic loss is more in comparison of its production. Many inhibitors were reported by researchers but causes health issues and are not ecofriendly (Alrefaee *et al.*, 2021). Organic inhibitors have been used for corrosion inhibition of mild steel. For this reason, inhibitors extracted from natural sources, such as plant extracts were very helpful for this purpose. The advantages for using plant-based inhibitors include eco-friendliness and cheap. Organic compounds containing Nitrogen, Sulfur or Oxygen atoms act as corrosion inhibitor by adsorption on metal surface though co-ordinate bonding. Such inhibitors are generally toxic to the environment and human being as well. Plant extracts have been found more useful, naturally occurring low cost and environment friendly corrosion inhibitors instead of using organic compound. A large number of works are reported on biodegradable natural products as corrosion inhibitors (Vorobyova *et al*., 2022).

The present work deals with the corrosion inhibition and adsorption behavior of constituents of coffee plum extract as an environmentally begin inhibitor on mild steel corrosion in dilute hydrochloric acid solution. The inhibition efficiency of FJ extract was studied by means of weight loss measurements in 1 M HCl using weight loss method, phytochemical studies FTIR spectroscopy,

SEM and AFM techniques. Finding of the experiments show great potential of *FJ* extract as corrosion inhibitor for mild steel in HCl environment (Ahmed and Zhang 2021). The results are discussed in detail in the following sections.

Generally called as Paniala in Gorakhpur city (U.P). The plant is well known for its good medicinal values. It is useful in toothache, bleeding gums etc.

MATERIALS AND METHODS

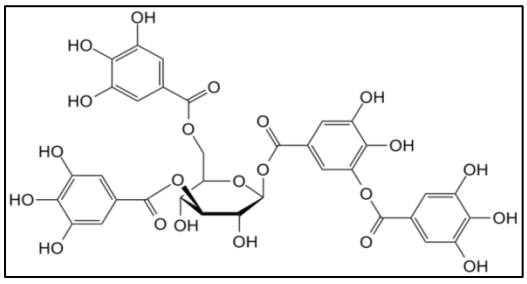
Alloy Used: Commercially available mild steel (C 0.15% by weight) was used for all experiments. The mild steel sheet was mechanically press-cut into 2.5×2.5×0.1 cm coupons. The steel coupons were immersed in 5% HCl to remove rust and sequentially polished using Sic emery papers of grade 220, 400, 600 and 1000, washed thoroughly with distilled water and degreased with acetone.

Chemicals Used: 1M HCl was prepared using analytical grade concentrated 37% HCl (Merck products) respectively and double distilled water.

Preparation of Inhibitor: *Flacourtia jangomas* (FJ) extraction was performed by means of reflux method as done in the previous studies. Fresh leaves stem bark and roots of Coffee plum (Paniala) were collected from Gorakhpur city of (U.P.), India. The parts of the plant were left for dying in shade in air atmosphere for about 6 days. Dried leaves, stem bark and roots were crushed and ground to make powder. Extract was prepared separately in 1M HCl for investigation in 1M HCl medium.10 g of dried powder of leaves were digested in 200 mL 1M HCl and kept overnight. Next day it was filtered and the filtrate volume was made up to 200 mL using 1M HCl. The extracts so far prepared were taken as stock solutions from which 0.1, 0.5, 1 and 5 % test solutions were prepared. Similarly extracts of stem bark and root were also prepared.

Phytochemical Screening

Test for Tannin. 1 g of powdered sample was boiled with 20 ml distilled water for five minutes in a water bath and is filtered while hot. 1 ml of cool filtrate is diluted to 5 ml with distilled water and a few drops of 10 % ferric chloride are added and observed, A bluish-black or brownish-green precipitate indicated the presence of tannins (Umoren *et al.*, 2008).



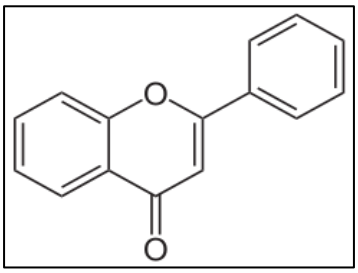
Tannin

Test for Saponins. 1 g of powdered dried sample is boiled with 10ml of distilled water for 10 minutes. The mixture was filtered while hot and allowed to cool. The following tests are then carried out.

Frothing: 2.5 ml of filtrate is diluted to 10ml with distilled water and shaken vigorously for 2minutes

Emulsifying properties: 2 drops of olive oil was added to the solution obtained from diluting 2.5 ml filtrate to 10 ml with distilled water (above), shaken vigorously for a few minutes (formation of a fairly stable emulsion indicates the presence of saponins) (Singh *et al.*, 2019).

Test for flavonoids. 1 g of the powdered dried leaves of sample is boiled with 10 ml of distilled water for 5 minutes and filtered while hot. Few drops of 20 % sodium hydroxide solution is added to 1 ml of the cooled filtrate. A change to yellow color which on addition of acid changed to colorless solution indicates the presence of flavonoids (Ong *et al.*, 2019).



Structure of Flavone (2-Phenyl-1-benzopyran-4-one)

Mass Loss Measurement. The weight loss studies were carried out at 30ºC by immersing steel coupons of known weight and surface area in 100 ml each of blank 1M HCl and test solutions containing various concentrations of extracts for 48 hours.

The dissolution of iron takes place and reaction can written as (Aslam *et al*., 2022)

$Fe + 2HCl \rightarrow FeCl_2 + H_2\uparrow$

$Fe + H_2SO_4 \rightarrow FeSO_4 + H_2$

After 48 hours of reaction, the specimens were taken out, washed with water, dried and weighed. Corrosion rates (in terms of $g.cm^{-2}h^{-1}$) were calculated using following expression.

$$\text{Corrosionrate (CR)} = \frac{Wi - Wf}{\text{Surface area (cm}^2) \times \text{Time (h)}}$$

Where, W_i = initial weight of coupon,

W_f = weight of coupons after treatment

$W_i - W_f$ = weight loss (g)

The surface coverage (θ) as a result of adsorption of inhibitor and inhibition efficiency (η%) were calculated from corrosion rates using the following expression:

$$\theta = \frac{CR_{blank} - CR_{inhibitor}}{CR_{blan}}$$

$$\eta = \frac{CR_{blank} - CR_{inhibitor}}{CR_{blan}} \times 100$$

where,

CR_{blank} = corrosion rate in absence of inhibitor

$CR_{inhibitor}$ = corrosion rate in presence of the inhibitor

Characterization

Fourier Transform Infrared Spectroscopy (FTIR) Analysis: The identification of the corrosion inhibitor components in the extract was carried out by FTIR spectroscopy. FTIR spectra were recorded for aqueous extract of the leaves of coffee plum (Paniala). This technique relies on the fact that infrared light absorbed by a sample will eventually be converted into heat (Rawat *et al*., 2021). It is applicable, substantial and relatively simple method for studying the plant extract on inhibitory characteristics (Tan *et al.,* 2021).

Surface analysis: The scanning electron microscope (SEM) used to gather information on the nonscale surface morphology of particles. The surface morphology of the samples was recorded in the presence and absence of inhibitor (Fossati *et al*., 2006). The study was conducted by CIF Manipal (Noor, 2008).

Adsorption Studies: The adsorption of inhibitor molecule on the surface of metal surface plays a major role on its corrosion inhibition mechanism. Various adsorption isotherms like Langmuir, Temkin were tested and, among them Langmuir was found to be the best, giving a straight line (Hsissou, 2019).

RESULTS AND DISCUSSION

Weight loss measurements. Table1 shows the variation of corrosion rate, surface coverage (θ) and percent inhibition efficiency (η%) in 1M HCl. The data revealed that corrosion rates were significantly lowered down in presence of inhibitor. The corrosion rate decreased with increasing extract concentration (Hegazy and Atlam 2016). The maximum lowering in corrosion rate was calculated in presence of 5% leaf extract of Paniala. 5% of extract concentration was also evaluated as the optimum concentration as on increasing the extract concentration above 5% no significant change in corrosion rate lowering was observed. In case of leaf extract maximum inhibition efficiency (98.1%) was noticed and no considerable change in inhibition efficiency was observed after this. With roots it was found 97.3 % and with stem bark 96.4% as maximum inhibition efficiency at the same concentration of inhibitor (Mahima, 2020).

The decreasing corrosion rate and increasing inhibition efficiency was attributed to the fact that the adsorption of inhibitor on the metal surface.

Table 1: Variation of corrosion rate, surface coverage and inhibition efficiency in 1M HCL.

Corrosion Rate (g $cm^{-2}h^{-1}$)				Surface Coverage (θ)			% Inhibition efficiency (η)		
Extract conc.%	leaves	Roots	Stem bark	Leaves	Roots	Stem bark	Leaves	roots	Stem bark
Blank	1.16×10^{-3}	—	—	—	—	—	—	—	—
0.1	8.85×10^{-5}	1.02×10^{-4}	1.95×10^{-4}	0.924	0.912	0.833	92.4	91.2	83.3
0.5	3.09×10^{-5}	8.82×10^{-5}	9.95×10^{-4}	0.973	0.924	0.913	97.3	92.4	91.3
1.0	2.95×10^{-5}	6.84×10^{-5}	5.94×10^{-5}	0.975	0.941	0.948	97.5	94.1	94.8
5.0	2.24×10^{-5}	3.13×10^{-5}	4.12×10^{-5}	0.981	0.973	0.964	98.1	97.3	96.4

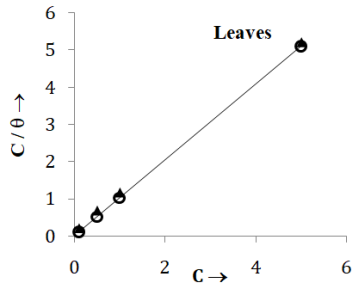


Fig. 1. Langmuir Adsorption isotherm for FJ leaves.

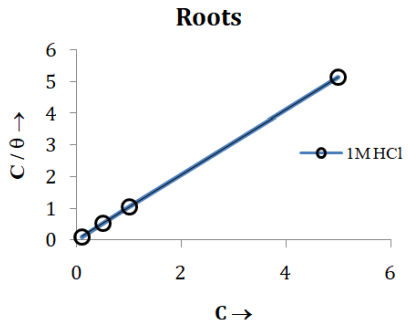


Fig. 2. Langmuir Adsorption isotherm for FJ roots.

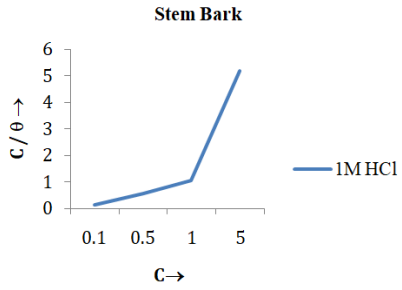


Fig. 3. Langmuir Adsorption isotherm for FJ stem bark.

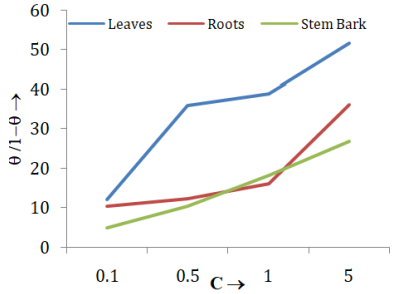


Fig. 4. Freundlich Adsorption isotherm in 1M HCl for FJ leaves, roots and stem bark.

Adsorption Behavior: The plant extracts are rich source of naturally synthesized chemical compounds which can form organo-metallic intermediates (Khadom *et al*., 2018). The formation of insoluble intermediates on the surface of metal substrate, are responsible for inhibitive action (De Souza and Spinelli 2009). Secondary metabolites present in extract have complicated molecular structure and number of O, S and N atoms. The adsorption of such compounds creates a barrier for metal dissolution. The experimental findings were applied to various adsorption isotherms and suitable isotherms were found (Salhi *et al*., 2017). Adsorption of Paniala extract depends on its chemical composition which showed the presence of various secondary metabolites such as flavonoids, steroids, tannins and phenolic compounds etc. which have oxygen atoms with lone pair of electrons for coordinate bonding with metal substrate (Noor, 2009). As a general consideration of FTIR spectra, the groups such as hydroxyl −OH, phenolic, ketone and amine are responsible for inhibition by adsorption of organic Phytochemicals through O and N atoms (Zhang *et al*., 2015). The adsorption of Phytochemicals is also attributed to the presence of pi - electrons and aromatic rings (Xiang and He 2021).

FTIR Analysis of Leaf Extract: FTIR data for aqueous extract of leaves and prominent peaks are given in Table 2. The data furnished relevant information that reflects the corrosion inhibition was due to adsorption of such groups present in the extract. FTIR confirms the adsorption of inhibitor on mild steel. FTIR spectra of FJ extract of pure sample (Qiang *et al*., 2017). The characteristics peak at 3421 cm^{-1}, 2923cm^{-1}, 2854cm^{-1} attributed to stretching vibration of -O-H bond of the hydroxyl group. These peaks do not appear in the spectrum in the IR of the compound. The modification of these bands indicates that the formation of chemical bonds between aromatic compounds and the metal surface. The available data indicates a wide range of values concerning peaks at different domain in 3421cm^{-1}, 2923cm^{-1}, 2854cm^{-1}, 1621cm^{-1}, 1519cm^{-1}, 1256cm^{-1} and 1075cm^{-1}.

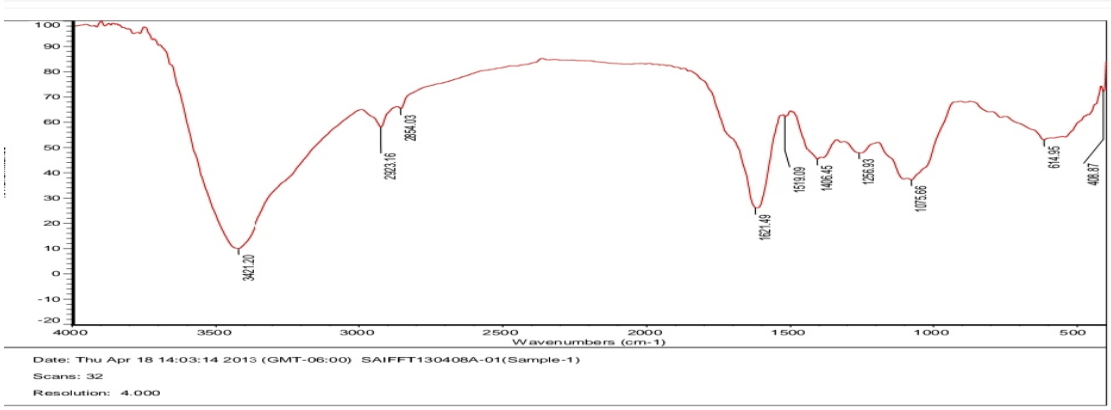
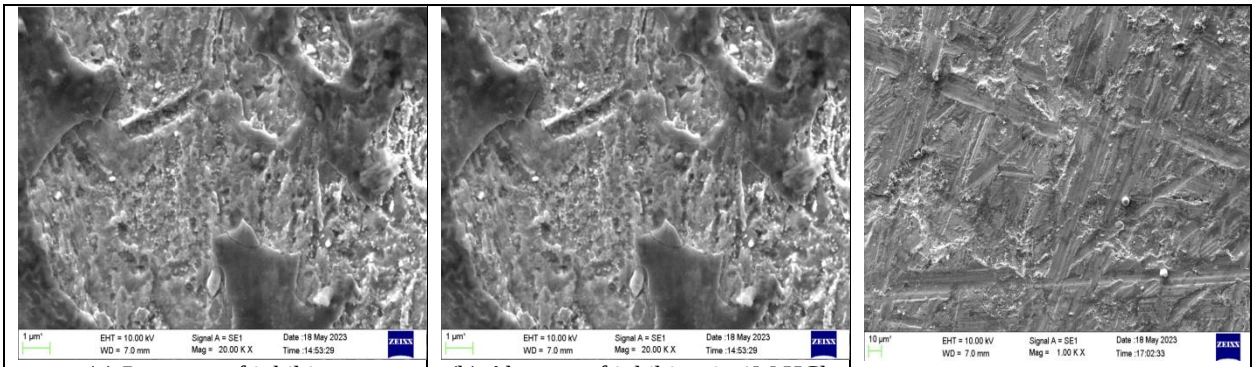


Fig. 5. FTIR Analysis.

Table 2: Prominent FTIR peaks for FJ extract.

Frequency (cm^{-1})	Band Assignment
3421	−OH (Hydroxyl group)
2923	Aromatic (Ar−H) stretching
2854	Alkane C−H stretching
1621	Alkene C=C and C=O stretching
1519	Aromatic rings
1256	Amines C−N stretching
1075	Oligosaccharide linkage O−C−O

SEM analysis: Surface morphology FJ was investigated by after submerging an metal steel coupon in the acidic solution in the presence, absence in 1M HCl and polished sheet (Sanaei *et al*., 2019). As shown in SEM image the metal surface which was exposed to corrosive solution without inhibitor is severely damaged in Fig. 6b. However adding the FJ extract to the acidic solution prevented the formation of corrosion products in Fig. 6a.(Kannon *et al*., 2018) Lower damage to the metal surface in the presence of FJ extract could be due to the efficient adsorption of corrosion inhibitors which diminish the contact between the corrosive ions and the metal surface and lessens the dissolution (Muralidharan and Iyer 1997). The mild samples, highly damaged due to direct attack of 1M HCl. It is clear that the irregularities on the metal surface is exposed in acid solution containing the inhibitor. The results reveal that the protective layer formed on the surface by adsorption does offer excellent corrosion protection properties (Prabakaran *et al*., 2016).



(a) Presence of inhibitor (b) Absence of inhibitor in 1M HCl (c) Polished sheet

Fig. 6.

Fig. 6. SEM micrographs of the mild steel in a) Presence of inhibitor b) Absence of inhibitor c) Polished sheet.

AFM micrograph

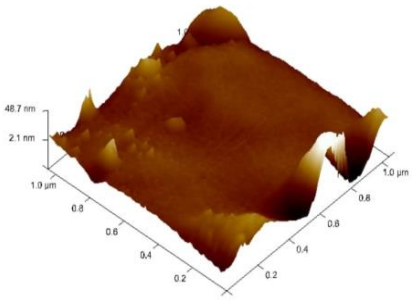


Fig. 7a. Presence of inhibitor.

Image Raw Mean	-0.000010 nm
Image Mean	-0.000010 nm
Image Z Range	1207 nm
Image Projected Surface Area	100
Image Surface Area Difference	28.4 %
Image Rq	102 nm
Image Ra	69.3 nm
Image Rmax	1207 nm
Raw Mean	0.00 nm
Mean	0.00 nm
Z Range	0.00 nm
Surface Area	0.00 l'm¸
Projected Surface Area	0.00
Surface Area Difference	0.00 %
Rq	0.00 nm
Ra	0.00 nm
Roughness Rmax	0.00 nm
Skewness	0.00
Kurtosis	0.00
Rz	0.00 nm
Rz Count	0.00
Peak Count	0.00
Valley Count	0.00
Max Peak ht (Rp)	0.00 nm
Average Max Height (Rpm)	0.00 nm
Maximum Depth (Rv)	0.00 nm
Average Max Depth (Rvm)	0.00 nm
Line Density	0.00 /l'm
Box X Dimension	0.00 l'm
Box Y Dimension	0.00 l'm

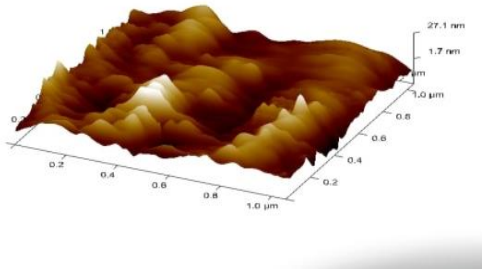


Fig. 7b. Absence of inhibitor.

Image Raw Mean	0.000025 nm
Image Mean	0.000025 nm
Image Z Range	1576 nm
Image Surface Area	140 l'm¸
Image Projected Surface Area	100 l'm¸
Image Surface Area Difference	40.4 %
Image Rq	123 nm
Image Ra	95.7 nm
Image Rmax	1576 nm
Raw Mean	0.00 nm
Mean	0.00 nm
Z Range	0.00 nm
Surface Area	0.00 l'm¸
Projected Surface Area	0.00 l'm¸
Surface Area Difference	0.00 %
Rq	0.00 nm
Ra	0.00 nm
Roughness Rmax	0.00 nm
Skewness	0.00
Kurtosis	0.00
Rz	0.00 nm
Rz Count	0.00
Peak Count	0.00
Valley Count	0.00
Max Peak ht (Rp)	0.00 nm
Average Max Height (Rpm)	0.00 nm
Maximum Depth (Rv)	0.00 nm
Average Max Depth (Rvm)	0.00 nm
Line Density	0.00 /l'm
Box X Dimension	0.00
Box Y Dimension	0.00

Fig. 7. AFM micrograph of surface dipped in 1M HCl in a) presence b) absence of inhibitor.

CONCLUSIONS

1) The FT-IR results demonstrate that the target compounds had been synthesized successfully.
2) The inhibition efficiency of FJ extract in a 1 M HCl solution at room temperature was evaluated by weight loss method. The data of the weight loss experiments indicate that the inhibition efficiency increases with increasing inhibitor concentration.
3) Surface analyses including FT-IR, AFM and SEM confirmed the protective effect of these inhibitors.
4) The result showed that corrosion rate was significantly decreased in presence of the extract and percent inhibition efficiency increased with increasing the concentration of extract. 98.1% inhibition efficiency was found in 1M HCl.
5)The decreased corrosion rate was due to adsorption of plant extract which was discussed on the basis of Langmuir, Freundlich and Temkin adsorption isotherm.
Leaf extract > Root extract > Stem Bark extract

FUTURE SCOPE

The present work deals with the corrosion inhibition and adsorption behavior of constituents of Paniala Extract of different parts in acidic medium as an eco-friendly inhibitor as they offer a sustainable, nontoxic, high – quality ,and aesthetically pleasing option for painting homes and other spaces.

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Conflict of Interest. None.

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