

Eleusine Coracana as an Eco - Friendly Inhibitor Against Mild Steel Corrosion in Acid Environment

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ABSTRACT

Corrosion is the major problem that needs to be addressed for environmental and financial reasons. Several efforts have been made using corrosion preventive practices and use of green corrosion inhibitors is one of them. This research aims to study the corrosion inhibition of finger millet (*Eleusine coracana*) seed extract (FIE) on mild steel corrosion in 1N HCl was investigated by phytochemical screening, weight loss, potentiodynamic polarization and electrochemical impedance spectroscopy. Phytochemical profiling of FIE reveals the presence of phytochemicals which prevents mild steel corrosion. The weight loss study was carried for concentrations range from 0.05% - 3% v/v in different temperatures range from 303 K to 343 K. FIE exhibited excellent inhibition efficiency, reaching 98.3 % for 3 % v/v FIE at 303 K after 1 hour immersion. The adsorption process was found to best fit with Langmuir adsorption isotherm compared to other models. Activation parameters, namely activation energy (E_a), activation enthalpy (ΔH^*), activation entropy (ΔS^*) and free energy (ΔG_{ads}) were determined, elucidating the energetics of the corrosion and inhibition processes. Potentiodynamic polarization and electrochemical impedance spectroscopy studies revealed maximum inhibition efficiencies of 94% and 97% respectively, demonstrating the excellent corrosion protection offered by FIE in 1N HCl medium.

Keywords: Green inhibitor, mild steel, corrosion inhibition, acid medium, adsorption

INTRODUCTION

The mineral acid solutions are mainly used in industries such as oil and gas wells, petrochemical and power plants to remove scales, rust or mineral deposits. Majority of the acid cleaning solutions are aggressive resulting in dissolution of metals [1]. Therefore, effective conventional inhibitors such as phosphates and chromates are employed but they are harmful to the environment. Also, they cause serious environmental, economic and safety damage with yearly losses surpassing \$2.5 trillion.

Hence, there is an urgent demand for environmentally friendly substitutes made from natural sources. Consequently, the use of green or eco-friendly inhibitors is becoming increasingly popular due to their effectiveness, affordability and lack of toxicity. Numerous plant extracts have been shown to be highly effective corrosion inhibitors in both acidic and alkaline conditions. The heteroatoms and double bonds found in the secondary metabolites from plant extract aid in the molecule's adsorption on metal surfaces and shield them from corrosion [2 – 5]. Several plant extracts such as *Musa paradisica* [6], *Saraca ashoka* [7], fenugreek and gooseberry leaves [8], *Mimosa elangi* [9], eucalyptus [10], *Malus domestica* [11], guava leaves [12], *Terminalia arjuna* [13], *Adhatoda vasica* [14], rubber leaves [15], *Parthenium hysterophorus* [16], pineapple leaves [17], henna [18] and natural gums [19] have been reported as effective green corrosion inhibitors for mild steel in acidic environments.

Experimental

Materials

Preparation of specimens

Mild steel specimens were polished using 150, 200, 400, 600 and 800 grades of emery sheets. The specimens were well cleaned using distilled deionized water and then again by acetone and stored in dessicator.

Preparation of FIE Extract

The Eleusine coracana seeds were collected and ground well into powder. 25 g of it was refluxed with 500 ml of 1N HCl for 3 hours. It was kept overnight and filtered. From the filtrate, the inhibitor test solutions of 0.05%, 0.1%, 0.5 %, 1% 1.5%, 2%, 2.5%, 3% v/v concentrations were prepared. 1N HCl was used as blank corrodent.

Method

Phytochemical screening

The filtered FIE extract was qualitatively analysed for the presence of phytochemicals which prevents corrosion by using standard procedure. The preliminary screening was done at Avinashilingam University, Coimbatore.

Weight Loss method

The polished mild steel (MS) samples were weighed and immersed in blank and FIE inhibitor test solutions of 0.05%, 0.1%, 0.5 %, 1% 1.5%, 2%, 2.5%, 3% v/v concentration by using glass hooks in 100 ml beaker. The MS samples in beakers were maintained in temperature ranges of 303K – 343K at 1 hour of immersion. After 1 hour, the MS samples were removed, washed, dried and weighed. The inhibition efficiency (IE) and corrosion rate (CR) were evaluated from the following formulas [20].

Corrosion rate (CR)

The rate of corrosion is expressed in Mils per year (mpy)

$$\text{Corrosion Rate CR (mpy)} = \frac{534 W}{D A t} \quad (1)$$

W is the weight loss in mg,

D is the density of mild steel ($7.8/\text{cm}^3$),

A is the area of specimen (4.7244 in^2)

t is the time of immersion.

Inhibition Efficiency (IE%)

The percentage inhibition efficiency (IE %)

$$\text{IE \%} = \left[\frac{W_o - W_i}{W_o} \right] \times 100 \quad (2)$$

W_o and W_i are the weight loss value in absence and in presence of inhibitor.

Kinetic study

The kinetic study provides information regarding the nature of the interaction between the inhibitor molecules

and the mild steel surface as well as how the corrosion process varies with temperature. Weight loss measurements at various temperatures (e.g., 303 K, 313 K, 323 K, and 333 K) with and without different concentration of FIE extract are used to assess the kinetics of corrosion inhibition. The impact of temperature on the corrosion process was evaluated, and the corrosion rate (CR) was computed using the equation (1) based on mass loss.

To determine the activation energy (E_a), the Arrhenius equation was employed [21].

$$\ln CR = \ln A - \frac{E_a}{RT} \quad (3)$$

Where CR is the corrosion rate in mpy, A is the Arrhenius pre exponential factor, E_a is the activation energy in KJ mol^{-1} , R is the universal gas constant and T is the absolute temperature in kelvin

Thermodynamic parameters

Thermodynamic parameters including free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) were examined in order to understand the interaction between the plant extract and the metal surface. These values aid in determining the kind of interaction occurring and whether or not the inhibitor's adsorption on mild steel is spontaneous.

Adsorption isotherm

Adsorption isotherm models were utilized to further investigate the FIE extract's surface coverage and inhibitory mechanism on mild steel. The following formula was used to determine surface coverage (θ) using the weight loss data:

$$\theta = \frac{\text{IE \%}}{100} \quad (4)$$

To identify the best-fitting model to explain the nature of the interaction between the inhibitor molecules and the metal surface, the experimental data were fitted to a variety of adsorption isotherm models, including Langmuir, Temkin, Freundlich and El Awady [22, 23].

$$\text{Langmuir isotherm: } \frac{C}{\theta} = \frac{1}{K} + C \quad (5)$$

$$\text{Temkin isotherm: } \theta = \frac{1}{f} \ln K_{\text{ads}} + \frac{1}{f} \ln C \quad (6)$$

$$\text{Freundlich isotherm: } \log \theta = \log K_{\text{ads}} + n \log C \quad (7)$$

$$\text{El Awady isotherm: } \log \frac{\theta}{(1-\theta)} = \log K + y \log C \quad (8)$$

Electrochemical analysis

Electrochemical measurements were used to investigate the corrosion behavior of mild steel in 1N HCl and to assess the inhibition performance of FIE. Polarization studies provide information on corrosion potential (E_{corr}), corrosion current density (I_{corr}), polarization resistance (R_p), anodic Tafel constant (β_a) and cathodic Tafel constant (β_c) calculated from Tafel plot, enabling the determination of corrosion rates and the classification of inhibitors as anodic, cathodic or mixed-type. Electrochemical impedance parameters including charge transfer resistance (R_{ct}) and double layer capacitance (C_{dl}) were calculated from Nyquist plot. An increase in R_{ct} and decrease in C_{dl} in the presence of inhibitor reveals the formation of protective adsorbed layer on the mild steel surface.

RESULT AND DISCUSSIONS

Phytochemical screening

The preliminary phytochemical screening of FIE extract reveals the presence of flavonoids, sterols, anthraquinone, proteins, quinones, tannin, saponin and cardiac glycosides are listed in the Table 1. The adsorption of these phytochemicals on the mild steel are responsible for the corrosion inhibition process.

Table 1: Preliminary Phytochemical Screening of FIE Extract

Phytochemicals	Observation
Alkaloids	-
Flavonoids	+
Sterols	+
Terpenoids	-
Anthraquinone	+
Proteins	+
Phenolic compounds	-
Quinones	+
Carbohydrates	-
Tannin	+
Saponins	+
Phytates	-
Coumarins	-
Cardiac glycosides	+
Volatile oils	-

+ = Presence

- = Absence

Weight Loss method

The corrosion rate increases with increase in temperature because the hydrogen evolution over potential increases [24]. The influence of temperature on the corrosion of mild steel in 1N HCl without and with the presence of FIE extract was studied using weight loss method in the temperature range of 303K – 343K with 1h of immersion. The CR and IE values are listed in Table 2. The CR decreases and the % IE increases with an increase in concentration of FIE extract. The influence of temperature on corrosion rate of mild steel and the inhibition efficiency of FIE for various concentration is shown in Fig 1& 2. The IE and CR is plotted against temperature is shown in Fig 3 & 4 respectively. The CR decreases from 549 mpy in blank to 9 mpy in 3% v/v extract and the inhibition efficiency increased from 55% to 98% at 303 K. Maximum inhibition efficiency of 98% was obtained at 3% v/v FIE concentration at 303 K as well as in 343 K.

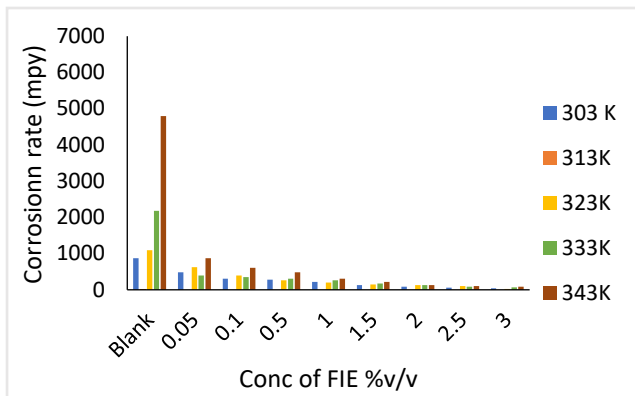


Fig. 1. Influence of temperature on corrosion rate of mild steel with different concentration

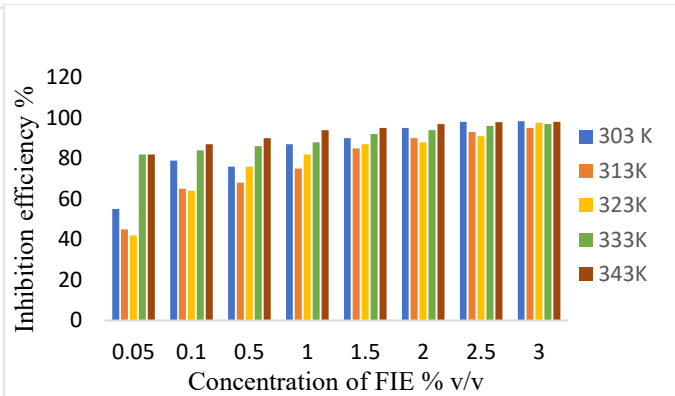


Fig. 2. Effect of temperature on inhibition efficiency of FIE with various concentrations

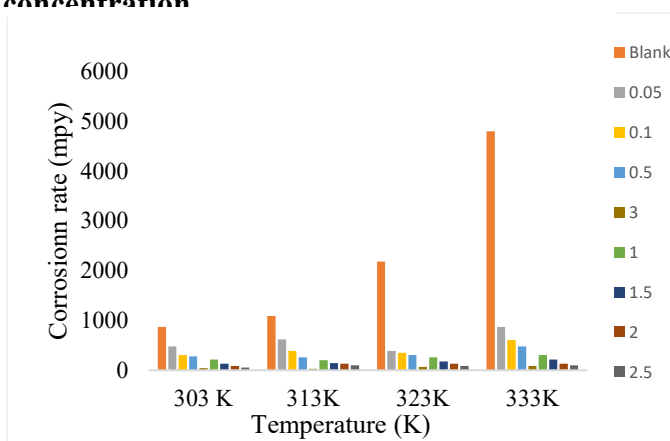


Fig. 3. Inhibition efficiency of FIE at different temperatures

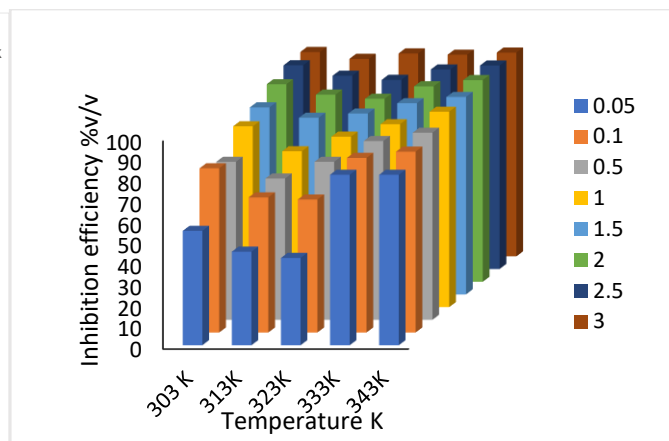


Fig. 4. Corrosion rate of mild steel with different temperature for various concentration

Table 2: Corrosion rate of mild steel and inhibition efficiency of FIE in 1N HCl at different temperatures

Conc. of FIE extract % v/v	303 K		313 K		323 K		333 K		343 K	
	CR	IE	CR	IE	CR	IE	CR	IE	CR	IE
	mpy	%	mpy	%	mpy	%	mpy	%	Mpy	%
Blank	549	-	872	-	1090	-	2180	-	4797	-
0.05	246	55	479	45	623	42	392	82	872	82
0.1	116	79	305	65	392	64	349	84	610	87
0.5	131	76	278	68	262	76	305	86	480	90
1	70	87	218	75	200	82	262	88	305	94

1.5	57	90	130	85	144	87	174	92	218	95
2	26	95	87	90	131	88	131	94	131	97
2.5	11	98	57	93	100	91	87	96	100	98
3	9	98	43	95	26	97.6	65	97	87	98

Kinetic study

The activation parameters for FIE on mild steel corrosion in 1N HCl are given in Table 3. Arrhenius plot and Eyring plot for FIE in 1N HCl on mild steel corrosion are shown in Fig 5 and 6. The result revealed that FIE in 1N HCl shows the deflation of E_a values from 45.05 kJ / mol to 43.07 kJ / mol with the increase in the concentration of inhibitor. It is seen that the adsorption phenomena can be classified into physisorption ($E_a < 40$ kJ / mol) and chemisorption ($E_a > 80$ kJ / mol) [25, 26]. When the inhibitor is present, the activation energy for FIE falls in comparison to the blank solution indicating the formation of a strong adsorbed film through chemisorption. However, the majority of E_a values are less than 40 kJ/mol, indicating that physisorption is predominantly responsible for the corrosion inhibition. The positive values for activation enthalpy ΔH^* of adsorption indicate that the adsorption process is endothermic. The decrease in ΔS^* reflects a decrease in randomness at the metal / solution interface due to the formation of an ordered inhibitor layer [27].

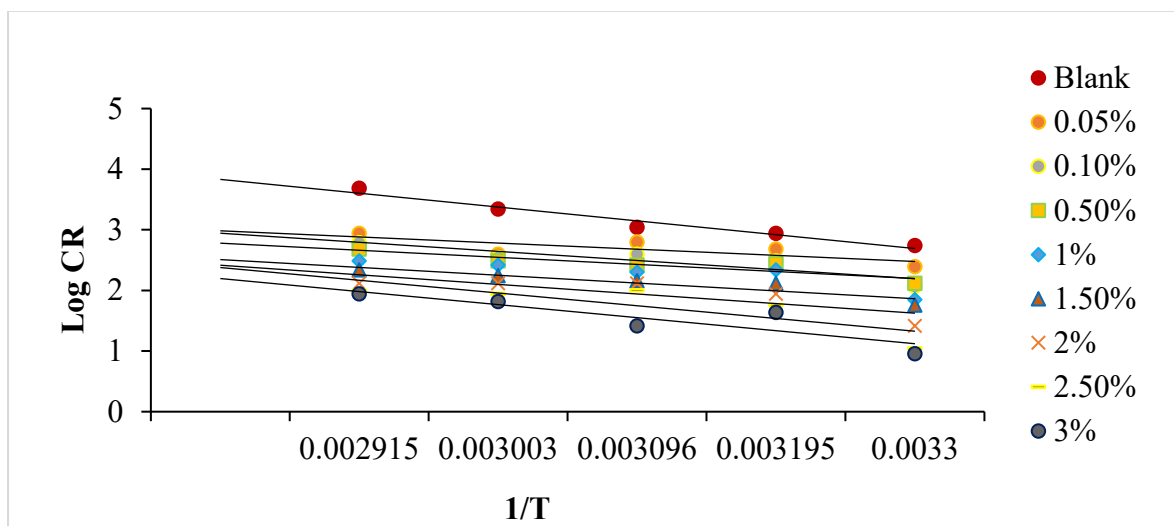


Fig 5: Arrhenius plot for FIE in 1N HCl on mild steel corrosion inhibition

Table 3: Activation parameters for FIE in 1N HCl

Concentration (% v/v)	E_a (kJ / mol)	ΔH^* (kJ / mol)	ΔS^* (J / mol)
Blank	45.05	18.4	259.9228
0.05	20.26	7.63	222.6384
0.1	30.16	11.92	234.4068
0.5	23.34	8.97	224.6939
1	27.45	10.74	228.9431

1.5	25.99	10.11	225.6912
2	32.23	12.81	232.567
2.5	42.78	17.39	245.152
3	43.07	17.52	243.9321

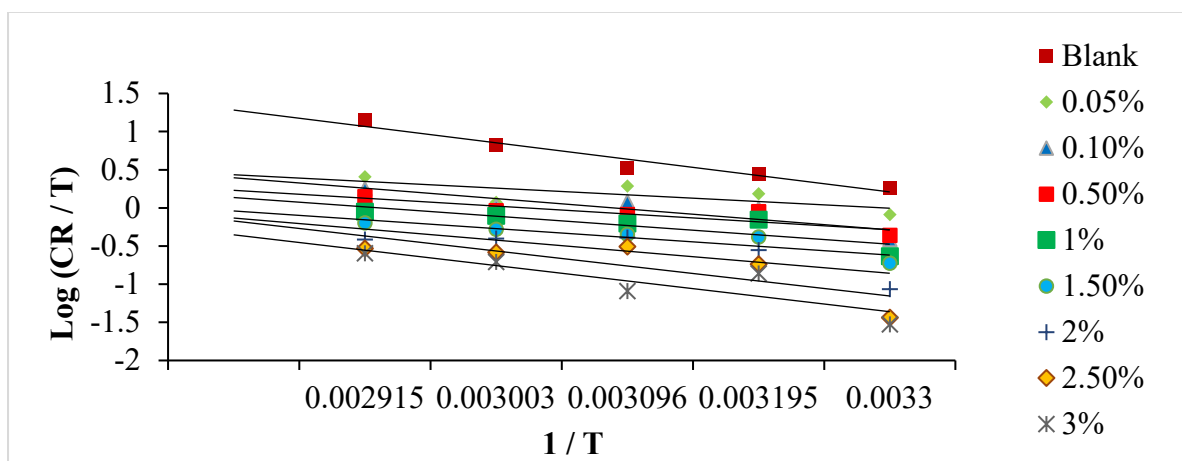


Fig 6: Eyring plot for FIE in 1N HCl on mild steel corrosion inhibition

Thermodynamic parameters

The thermodynamic parameters for FIE in 1N HCl on mild steel corrosion are shown in Table 4. The variation of ΔG_{ads} with increase in temperature is shown in Fig 7. The ΔG_{ads} values are negative ranging from -13.29 to -24.29 kJ / mol, confirming the spontaneous adsorption of the FIE on the mild steel surface and the adsorption process is mainly physisorption. The magnitude of ΔG_{ads} suggests that the adsorption is physisorption involving electrostatic interactions between the inhibitor molecules and the charged metal surface [28]. The positive ΔH_{ads} indicate that the adsorption process is endothermic. The ΔH_{ads} decrease from 36.79 kJ/mol at 0.05% to 1.16 kJ/mol at 3% shows the adsorption is favourable which is responsible for increased surface coverage and the formation of a more stable adsorbed layer on the metal surface. The development of a highly ordered and stable inhibitor layer on the mild steel surface results in a decrease in randomness, as indicated by the ΔS_{ads} values (143.54 to 24.54 J / mol. K).

Table 4: Thermodynamic parameters for FIE in 1N HCl

Concentration of FIE (% v/v)	ΔG_{ads} (kJ/mol)					ΔS_{ads} (J / mol. K)	ΔH_{ads} (kJ / mol)
	303 K	313 K	323 K	333 K	343 K		
0.05	-18.15	-17.70	-17.94	-23.59	-24.29	143.54	36.79
0.1	-19.23	-18.03	-18.49	-22.06	-23.42	85.39	18.09
0.5	-14.74	-14.19	-15.72	-18.04	-19.67	100.37	26.71
1	-14.88	-13.29	-14.83	-16.61	-19.27	82.24	21.55

1.5	-14.61	-13.89	-14.78	-16.73	-18.67	72.34	18.39
2	-15.77	-14.34	-14.25	-16.79	-19.36	56.53	12.92
2.5	-17.59	-14.77	-14.51	-17.36	-19.77	27.59	2.88
3	-17.55	-15.23	-17.76	-17.68	-19.39	24.54	1.16

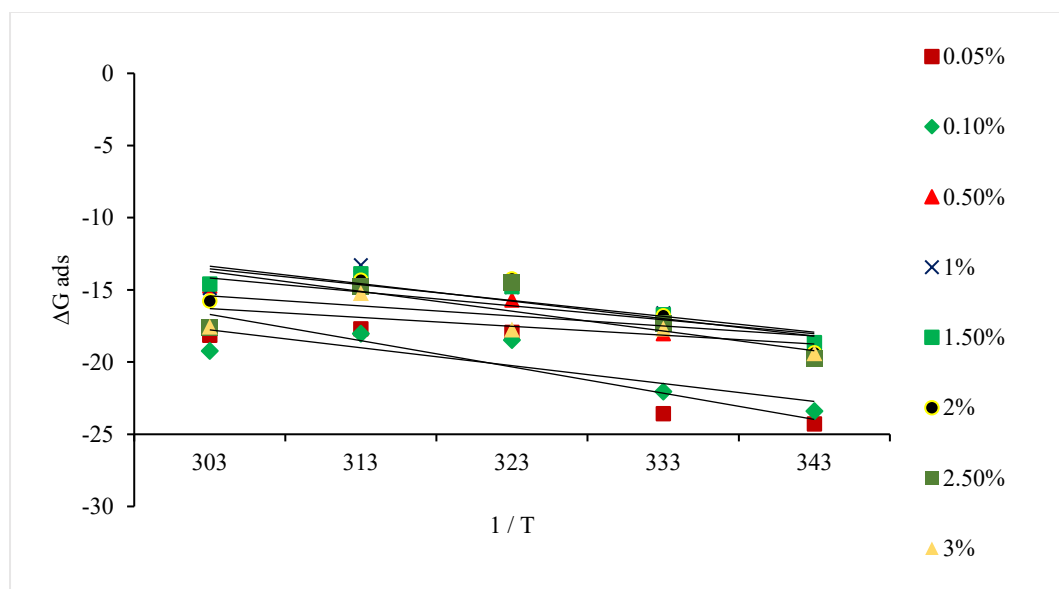


Fig 7: Effect of temperature on change in free energy of adsorption for FIE in 1N HCl

Adsorption isotherm

The Langmuir, Freundlich, Temkin and El-Awady isotherm models were used to assess the adsorption behavior of FIE inhibitor on mild steel in 1N HCl is shown in Fig 8 to 11. The formation of a monolayer adsorption on a comparatively homogenous metal surface was confirmed by the Langmuir isotherm (C/θ vs C), which demonstrated the best fit among these, with slope values approaching unity and good linearity. The correlation coefficients for the Freundlich adsorption plots ranged from 0.829 to 0.974, suggesting a moderate to strong fit. Temkin and El awady provides moderate fit.

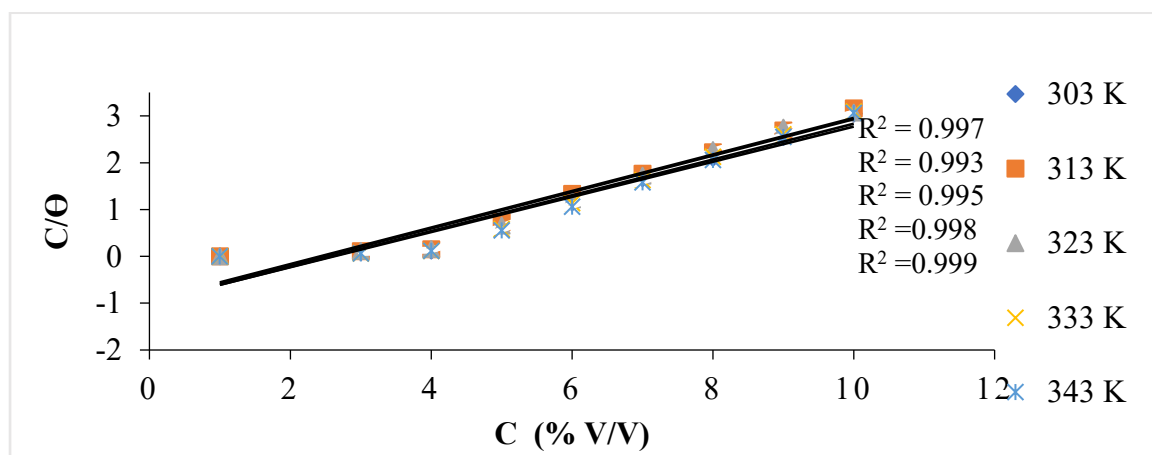


Fig 8: Langmuir plot for mild steel in 1N HCl containing different concentration of FIE extract

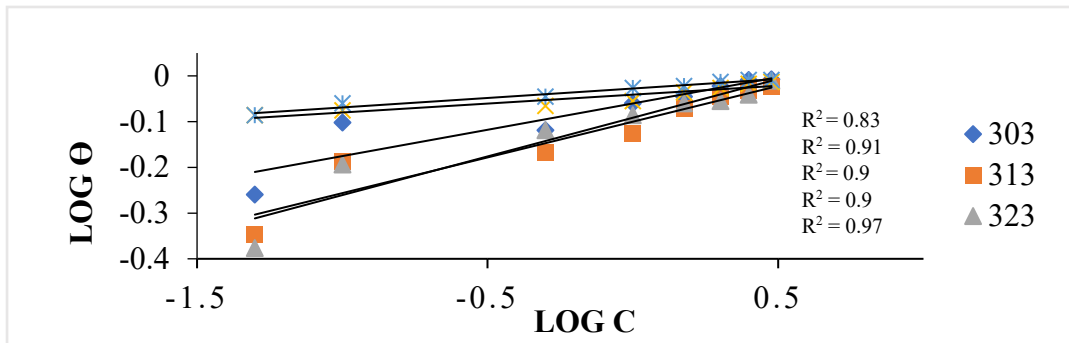


Fig 9: Freundlich plot for mild steel in 1N HCl containing different concentration of FIE extract

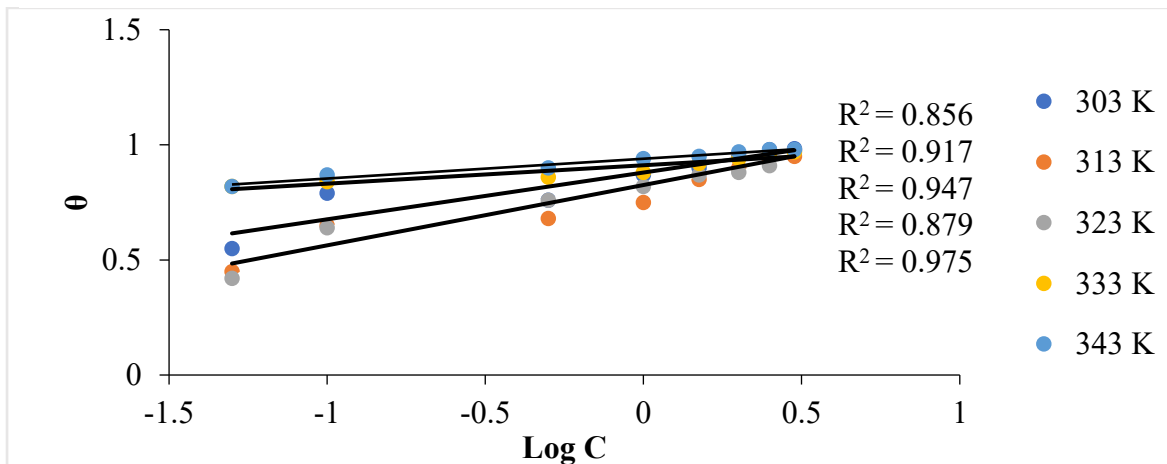


Fig 10: Temkin plot for mild steel in 1N HCl containing different concentration of FIE extract

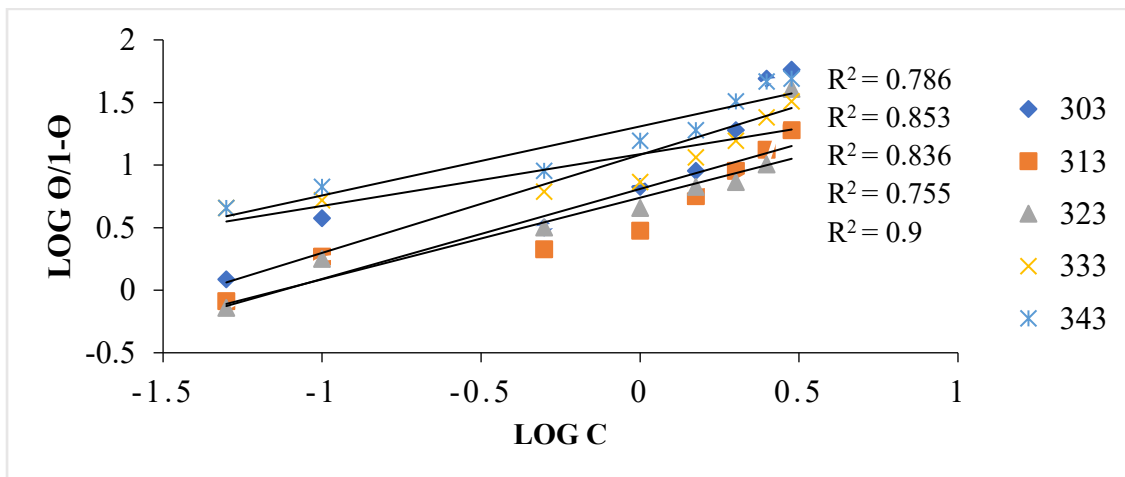


Fig 11: El – Awady plot for mild steel in 1N HCl containing different concentration of FIE extract

Electrochemical analysis

The electrochemical polarization and impedance characteristics for mild steel in 1N HCl, both in the absence and presence of FIE, are detailed in Table 5, while the related Tafel polarization is shown in Fig 12 and Nyquist plots are illustrated in Fig 13

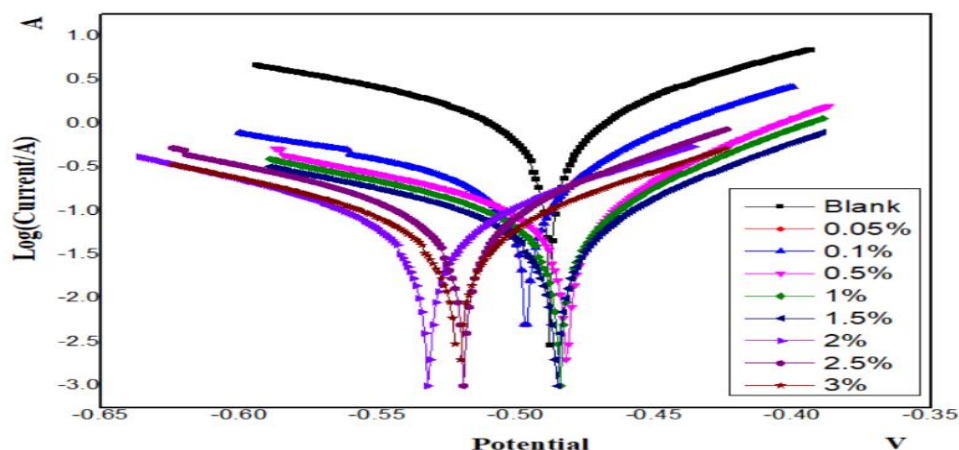


Figure 12: Tafel plot for mild steel in 1N HCl solution in absence and presence of FIE

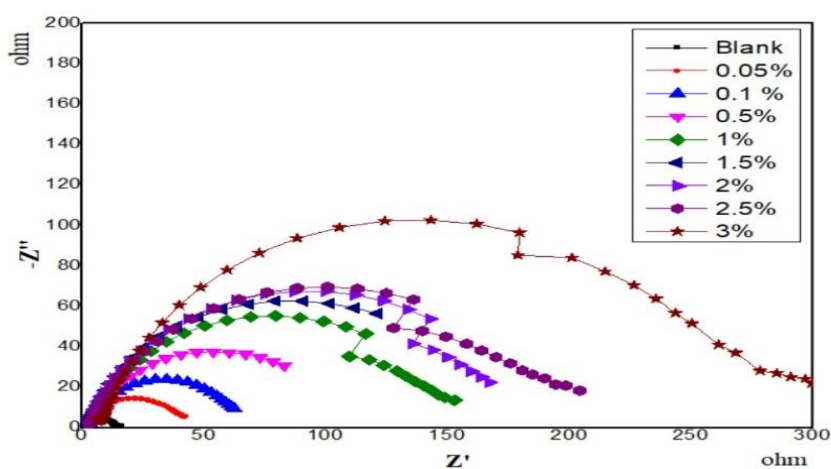


Fig 13: Nyquist plot for mild steel in 1N HCl solution in absence and presence of FIE

As the concentration of FIE increased, the corrosion current density (I_{corr}) decreased from 1.118 to 0.06325 mA cm^{-2} , suggesting a notable decrease in the rate of corrosion. As a result, the inhibition efficiency raised from 73% to 94%. The slight shift in corrosion potential (E_{corr}) from -0.4851 to -0.5173 V ($E_{corr} < 85 \text{ mV}$) reveals that FIE acts as mixed – type inhibitor [29 - 31]. According to Tafel and LPR methods, the highest inhibitory efficiency of 94% was attained at 3% FIE concentration. As the FIE concentration increased, the Nyquist plots showed an increase in semicircle diameter, which led to an increase in R_{ct} from 6.57 to 237.21 ohm and inhibitory efficiency from 78% to 97%. The observed variation in C_{dl} may due to partial desorption or rearrangement of adsorbed inhibitor molecules at metal - solution interface [32]. A charge-transfer mechanism is primarily responsible for controlling the corrosion process.

Table 5: Polarization and impedance parameters for FIE in 1N HCl on mild steel corrosion

Conc. of FIE (% v/v)	Polarization parameters							Impedance parameters			
	E_{corr}	I_{corr}	β_a	β_c	R_p	IE %		C_{dl}	R_{ct}	IE %	
	V	mA cm^{-2}	V / dec	V / dec	ohm	From Tafel	From LPR	(F) $\times 10^{-5}$	Ohm	C_{dl}	R_{ct}
Blank	-0.4851	1.118	0.092	0.136	27.3	-	-	2.5	6.57	-	-

0.05	-0.4928	0.3037	0.08	0.143	93.88	73	71	1.85	30.55	26	78
0.1	-0.4996	0.1646	0.075	0.135	127.3	85	78	2.01	49.37	20	87
0.5	-0.4798	0.1062	0.067	0.14	237.6	91	88	4.2	76.95	-68	91
1	-0.5149	0.09201	0.081	0.124	295.7	92	91	3.73	120.55	-49	94
1.5	-0.4812	0.08769	0.07	0.136	293.5	92	91	3.53	126.23	-41	95
2	-0.5276	0.07145	0.088	0.117	390.5	94	93	2.76	141.69	-10.4	95
2.5	-0.4824	0.06942	0.075	0.131	383.2	94	93	3.75	155.10	-50	96
3	-0.5173	0.06325	0.088	0.119	443.2	94	94	3.46	237.10	-38	97

CONCLUSION

Based on the experimental results, the following conclusions were drawn from the study

- The phytochemical screening of FIE in 1N HCl reveals the presence of flavonoids, sterols, anthraquinone, proteins, quinones, tannin, saponin and cardiac glycosides.
- The CR decreases and the % IE increases with an increase in concentration of FIE extract. Maximum inhibition efficiency of 98.3% and 98% was obtained at 3% v/v FIE concentration at 303 K and 343 K respectively.
- FIE adsorption on mild steel predominantly follows the Langmuir isotherm, indicating the formation of a homogeneous monolayer protective film on the metal surface.
- The kinetic and thermodynamic parameters suggest that FIE inhibits mild steel corrosion through a physisorption mechanism. The positive ΔH^* & ΔH_{ads} values shows the adsorption is endothermic, while the decrease in ΔS^* & ΔS_{ads} indicates the formation of a more ordered protective inhibitor film onto the metal surface.
- Electrochemical investigations demonstrated that FIE effectively suppresses mild steel corrosion in 1N HCl by forming a protective adsorbed layer on the metal surface. Maximum inhibition efficiencies of 94% and 97% were achieved from polarization and EIS studies, respectively by behaving as mixed type inhibitor.
- Thus, the acid extract of FIE is considered to be an affordable, eco-friendly and effective corrosion inhibitor for mild steel in acid media.

Ethical approval

Not required

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Credit authorship contribution statement

Mrs. S. Valli: Investigation, Methodology, Formal analysis, Writing original draft, **Dr. K. Anbarasi:** Supervision, Validation & Conceptualization.

Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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