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EVALUATION OF THE CORROSION INHIBITION PROPERTIES OF ETHANOLIC EXTRACT FROM ACACIA NILOTICA POD ON MILD STEEL IN 0.1 M SULFURIC ACID: AN EXPERIMENTAL STUDY UTILIZING FTIR AND SEM TECHNIQUES

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Abstract: The ethanol extract of Acacia nilotica pod (ANP) was investigated for its corrosion inhibition potential on mild steel immersed in 0.1M H_2SO_4 solutions containing ANP concentrations ranging from 0.1 to 0.5 g/L. The study employed weight loss measurements, Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) to evaluate the extract's performance. Weight loss experiments demonstrated that the inhibition efficiency was influenced by both the concentration of ANP and the exposure duration of the mild steel in the acidic medium. The highest inhibition efficiency recorded was 87.57%. Surface morphology analysis using SEM revealed significant differences between the steel samples exposed to H_2SO_4 with and without the extract, indicating the protective role of ANP. FTIR spectroscopy further confirmed the adsorption of the extract onto the mild steel surface, with functional groups such as C-O and N=O playing a critical role in the adsorption process. These findings highlight the potential of ANP as an effective corrosion inhibitor for mild steel in acidic environments.
Keywords: Acacia nilotica pod; Corrosion inhibition; Mild steel; Sulfuric acid; Adsorption

1 INTRODUCTION

Mild steel is extensively utilized in various industries, including food processing, petroleum, power generation, chemical, and electrochemical sectors, due to its excellent mechanical properties and affordability [1]. However, exposure to aggressive environments often leads to the corrosion of metals such as iron and steel, causing mechanical failures and significant damage to infrastructure. These include oil, gas, and water pipelines, bridges, public buildings, vehicles, and domestic appliances, resulting in high repair and replacement costs as well as potential safety hazards [2]. Consequently, developing effective corrosion protection strategies for mild steel in harsh acidic and alkaline conditions has become a critical focus [3].

Numerous studies have been conducted to mitigate the corrosion of mild steel, given its importance in various industrial applications. Typically, corrosion inhibitors are compounds containing heteroatoms like nitrogen, oxygen, phosphorus, and sulfur, often with long carbon chains, triple bonds, or aromatic rings in their structures [4]. However, these inhibitors are often costly, toxic, and harmful to the environment [5].

In contrast, plant-derived materials have emerged as promising alternatives due to their low cost, non-toxicity, environmental friendliness, and abundant availability. They are rich in heterocyclic compounds and functional groups such as -C=C-, -OR, -OH, -COOH, $-NR_2$, $-NH_2$, and -SR, which donate electrons that facilitate adsorption onto metal surfaces, thus inhibiting corrosion. Recent advancements in corrosion research have focused on exploring various plant extracts as green inhibitors [6]. Studies reveal that the inhibitory effects of plant extracts are attributed to the presence of organic compounds like tannins, saponins, alkaloids, steroids, glycosides, and amino acids [7].

In line with these developments, the present study investigates the corrosion inhibition properties of ethanol extract from Acacia nilotica pod (ANP) on mild steel in an acidic environment. The evaluation was carried out using weight loss measurements, Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM) to assess the efficacy of the extract as a green corrosion inhibitor.

2 MATERIALS AND METHODS

2.1 Material Preparation

The mild steel used in this investigation was acquired from Bayero University Kano's Department of Mechanical Engineering (New Campus). Its chemical composition was assessed via Energy Dispersive X-ray Fluorescence (ED-XRF) and contained Mn (0.27%), Al (0.04%), C (0.066%), among other elements, with Fe making up the remainder. Coupons of 5 × 4 × 0.11 cm were prepared, degreased with ethanol, rinsed in acetone, and air-dried before

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being stored in a desiccator. Reagents were of analytical grade, with solutions prepared using double-distilled water, ensuring experimental reliability following Ibrahim Jimoh and Bishir Usman [8].

2.1.1 Coupon preparation

For the corrosion tests, 100 mild steel coupons were fabricated, each measuring 5 × 4 × 0.11 cm. The surfaces of the coupons were polished sequentially using emery papers of varying grit sizes (240, 400, 800, and 1000) to achieve a smooth finish. Following polishing, the samples were cleaned with ethanol, degreased using acetone, and allowed to air-dry. Each coupon was weighed to obtain its initial mass, and the values were recorded. The prepared samples were then stored in desiccators to prevent moisture absorption prior to corrosion analysis following Ibrahim Jimoh and Bishir Usman [8].

2.1.2 Preparation of plant extract

The ANP samples were initially air-dried to remove moisture, then finely ground to increase surface area for extraction. These ground samples were submerged in ethanol and left to soak for 48 hours, facilitating the extraction of bioactive compounds. After soaking, the mixture was cooled to room temperature and filtered to separate the liquid extract from the solid residue. The resulting filtrate was subjected to evaporation at a temperature of 352 K to completely remove ethanol, leaving behind a concentrated extract. This extract was then used to prepare stock solutions. Various concentrations of the extract were subsequently prepared by dissolving 0.1 g, 0.2 g, 0.3 g, 0.4 g, or 0.5 g of the extract in 1 liter of 0.1 M sulfuric acid (H_2SO_4) solution following Ibrahim et al., [9].

2.1.3 Preparation of acid solution

For this study, a 0.1 M sulfuric acid (H_2SO_4) stock solution was prepared to serve as the corrodent. This was achieved by carefully diluting 5.40 mL of concentrated 98% H_2SO_4 (Analar grade, Merck) in a 1000 mL volumetric flask. Initially, 150 mL of distilled water was added to the flask to dilute the acid, and the solution was then topped up to the 1000 mL mark with additional distilled water. This stock solution was used consistently throughout the experiments for preparing various solutions. The concentration of the ANP ethanol extract used in the study ranged from 0.1 g/L to 0.5 g/L following Ayuba, et al., [10].

2.1.4 Weight loss measurement procedure

Weight loss experiments were conducted in a 250-mL beaker at a constant temperature of 303 K. For each test, 250 mL of the prepared solution was used. Mild steel specimens were initially weighed and then immersed in the solution for a duration of 1 hour. After the exposure period, the specimens were removed, thoroughly washed with distilled water to eliminate any loose corrosion products, and subsequently rinsed with ethanol and acetone. They were then dried and reweighed.

The weight loss of the mild steel (in grams) was determined by calculating the difference between the initial and final weights. From these values, the corrosion rate (expressed in g/h/cm²), inhibition efficiency (%I), and surface coverage (θ) were evaluated using specific mathematical formulas (Equations 1–3) [11].

$$\text{CR (g/h/cm}^2) = \frac{\Delta W}{At} \quad (1)$$

$$\theta = 1 - \frac{W_1}{W_2} \quad (2)$$

$$\%\text{I} = \left(1 - \frac{W_1}{W_2}\right) \times 100 \quad (3)$$

Here, W1 and W2 represent the weight losses (in g/dm³) of the mild steel coupons in the sulfuric acid solution with and without the presence of the inhibitor, respectively. The degree of surface coverage () quantifies the extent to which the inhibitor protects the metal surface. A denotes the surface area of the metal coupon (in cm²), t is the immersion time (in hours), and W is the measured weight loss (in grams) of the mild steel coupon after the specified immersion period (t).

2.1.5 Fourier transform infrared spectrophotometry (FTIR)

FTIR analyses of the inhibitor and that of the corrosion products (in the presence and absence of the respective inhibitors) were carried out using an FTIR instrument, the 630 Cary series Agilent Technologies. Two coupons were separately dipped in 250 mL of 0.5 g/L inhibitor concentration for 2 days to form an adsorbed layer, after which they were retrieved, dried, and scraped with a sharp blade. The scraps were collected for analysis. The samples were prepared using KBr, and the analysis was done by scanning the sample through a wave number range of 400–4000 - 4000 cm-1 following Elabbasy et al.[12].

2 SURFACE ANALYSIS OF MILD STEEL COUPONS

The surface morphology of mild steel coupons was analyzed using an Inspect S50 scanning electron microscope to evaluate corrosion inhibition effectiveness. Coupons measuring 5 cm × 4 cm × 0.11 cm were immersed in both a blank and a 0.5 g/L inhibitor solution for 48 hours. Post-immersion, the samples were rinsed with distilled water, air-dried, mounted on metal stubs, and gold-coated for conductivity. SEM images were captured at accelerating voltages of 2.00 and 12.50 kV, highlighting morphological differences between inhibited and uninhibited surfaces following Okore et al.[13].

3 RESULTS AND DISCUSSION

3.1 Weight Loss Measurement

3.1.1 Effect of inhibitor concentration

The influence of varying concentrations of ANP extract on corrosion rate and inhibition efficiency for mild steel in 0.1 M H_2SO_4 at 303 K was studied. Figures 1 and 2 illustrate the relationship between inhibitor concentration and the corrosion protection performance. As depicted in Figure 1, increasing the concentration of the plant extract significantly reduces the corrosion rate of mild steel. This trend indicates the efficiency of ANP extract in mitigating corrosion, likely due to its ability to form a protective film on the metal surface, impeding further interaction between the metal and the corrosive environment [14].

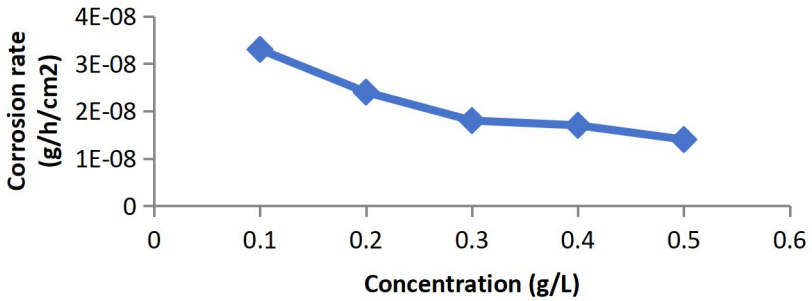

Figure 1 Variation of Corrosion Rate (g/h/cm2) of Mild Steel as a Function of Various Concentration of ANP Extract in 0.1M H2SO4 at 303K

The enhanced corrosion inhibition observed with increased concentrations of ANP extract can be attributed to greater surface coverage on the mild steel, forming a protective barrier that inhibits metal dissolution. This aligns with the findings of Niamien et al.,[15] and Olasehinde et al.,[16], where increased inhibitor concentration correlated with improved inhibition efficiency. The mechanism suggests that higher surface adsorption reduces the active sites for corrosive agents, effectively mitigating corrosion and enhancing the inhibitor's performance [17].

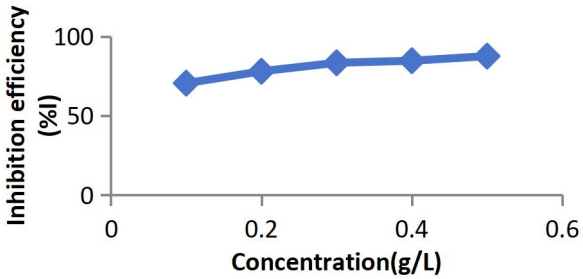

Figure 2 Variation of Inhibition Efficiency (%IE) against Various Concentrations of ANP Extract for Mild Steel Corrosion in 0.1 M H2SO4 at 303 K

Table 1 Inhibition Efficiencies (%IE) and Corrosion Rates for Corrosion of Mild Steel in the Absence and Presence of Various Concentrations of the Extract in 0.1M H2SO4 at 303–333 K

Conc.(g/L)	Corrosion Rate x10^{-4}($gh^{-1}cm^{-2}$)				Inhibition efficiency (%)			
	303K	313K	323K	333K	303K	313K	323K	333K
Blank	1.11	3.45	5.79	7.93	-	-	-	-
0.1	0.33	1.19	2.30	3.86	70.54	65.45	60.23	51.34
0.2	0.24	0.93	2.05	3.42	78.04	72.95	64.61	56.90
0.3	0.18	0.75	1.53	2.88	83.40	78.31	73.59	63.73
0.4	0.17	0.58	1.39	2.54	84.68	83.27	76.07	67.99
0.5	0.14	0.52	1.10	2.32	87.57	84.92	80.98	70.69

3.1.2 Effect of immersion time

The weight loss-time curve (Fig.3) for mild steel in 0.1 M H_2SO_4 with and without ANP extract indicates a clear trend. As exposure time increases, the weight loss of mild steel in the uninhibited solution rises due to ongoing corrosion.

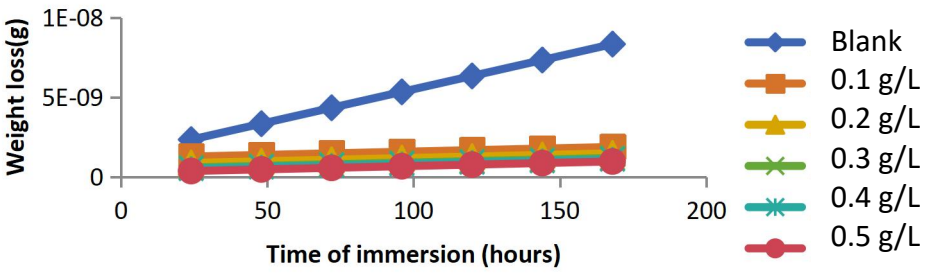

Figure 3 Effect of Immersion Time (hours) on Corrosion Rate of Mild Steel in 0.1 M H2SO4 in the Absence and Presence of ANP Extract at 303 K

However, the presence of ANP extract reduces weight loss significantly, which becomes more pronounced at higher inhibitor concentrations. This reduction is attributed to the formation of a protective layer on the mild steel surface, impeding the corrosive attack of sulfuric acid [18]. Such observations highlight the effectiveness of plant extracts as green corrosion inhibitors, consistent with findings from recent studies exploring plant-derived materials as eco-friendly solutions for metal protection in acidic environments[19].

3.1.3 Effect of temperature

The variation in corrosion rate of mild steel in 0.1M H2SO4 in the presence and absence of ANP extract inhibitor at different temperatures has been studied, and it is evident from Table 1 and the plot in Fig. 4 that the corrosion rate of mild steel with or without extract increased with an increase in temperature [20].

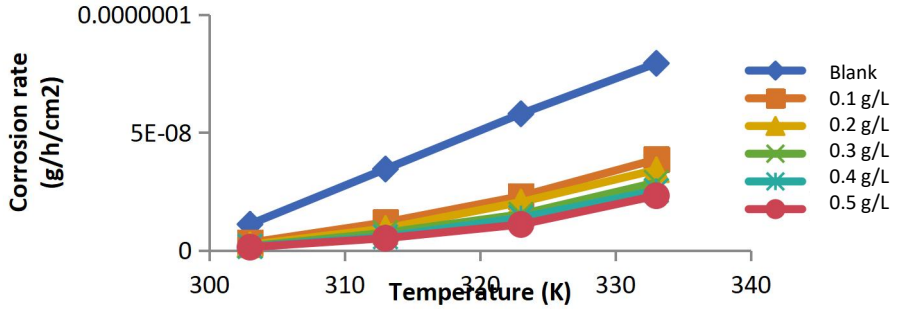

Figure 4 Variation of the Corrosion rate of Mild Steel Against Temperature (303–333 K) in the Absence and Presence of Different Concentrations of ANP Extract in 0.1 M H2SO4

This is due to the fact that as the temperature increased from 303 to 333 K, the rate of corrosion of the mild steel coupons also increased as a result of the increasing average kinetic energy of the reacting molecules [21]. However, the corrosion rate is retarded in the presence of the plant extract. The corrosion rate increases more rapidly with temperature in the absence of the extract. Two observations could be drawn from the result: (i) The mild steel surface is effectively damaged in the acidic medium [22], and (ii) ANP extract is a strong inhibitor for mild steel corrosion in 0.1M H2SO4 at a lower temperature. Furthermore, it is observed in Fig. 5 and Table 1 that the inhibition efficiency of plant extract decreased with an increase in temperature for all concentrations of the inhibitor. This may be as a result of the increasing solubility of the adsorbed protective inhibitor barrier on the mild steel surface, thereby increasing the susceptibility of these coupons to dissolution in the acid media [23].

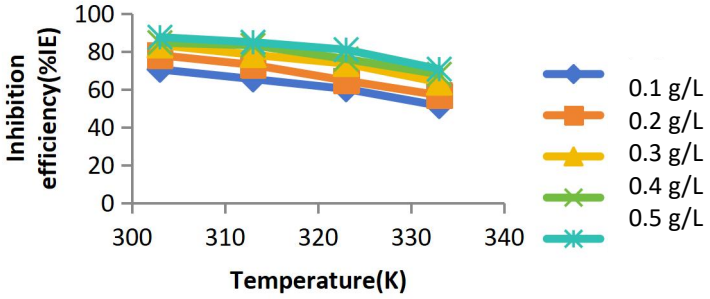

Figure 5 Variation of Inhibition Efficiency (%IE) Against Temperature for the Corrosion of Mild Steel in 0.1 M H2SO4 in the Presence and Absence of ANP Extract

3.1.4 Stability of the inhibitor

The stability of the ethanol extract of ANP for the corrosion of mild steel in H2SO4 (over a time range) was also studied by plotting values of inhibition efficiency versus the period of contact, as shown in Figure 6. The plots indicate that at 303 K, the ethanol extract of ANP retained more than 87% of its inhibition efficiency even after 168 hours of immersion. This agrees with the finding of Obot et al., [24].

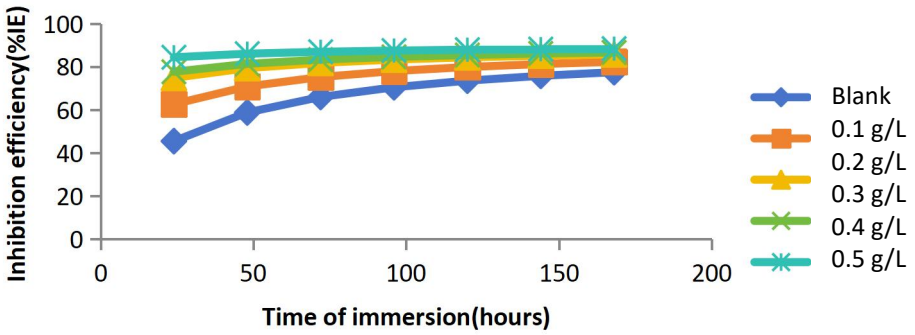

Figure 6 Effect of Immersion Time (hours) on Inhibition Efficiency (%IE) of ANP Extract on the Dissolution of Mild Steel in 0.1 M H2SO4 at 303 K

3.2 Surface Analysis

In Fig. 7b, the mild steel surface is highly damaged due to the effect of the acid on the surface, and in Fig. 7c, there is an improvement in the surface morphology, which shows a smooth surface when compared to the uninhibited surface.

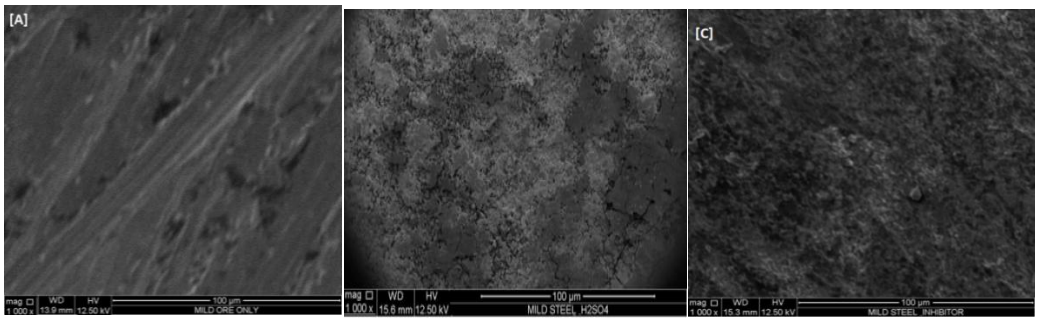

Figure 7 SEM Micrographs of Mild Steel: (a) Fresh Mild Steel; (b) Without Inhibitor; (c) With Inhibitor

It is evidence that the damaging effect of the acid on the mild steel is greatly reduced due to the protective layer of the adsorbed inhibitor that prevents corrosion caused by the acid attack on the mild steel surface. The smoothness of the mild steel surface in the presence of an inhibitor is due to the barrier of the protective film over the metal surface, which gives rise to more ordered corrosion products [25].

3.3 FTIR Study

In order to further support the adsorption behavior of the inhibitor on the surface of mild steel, FTIR spectroscopy was employed. Fig. 8a shows the FTIR spectrum of the ethanol extract of ANP alone. Fig. 8b shows the FTIR spectrum of the corrosion product when an ethanol extract of ANP was used as an inhibitor.

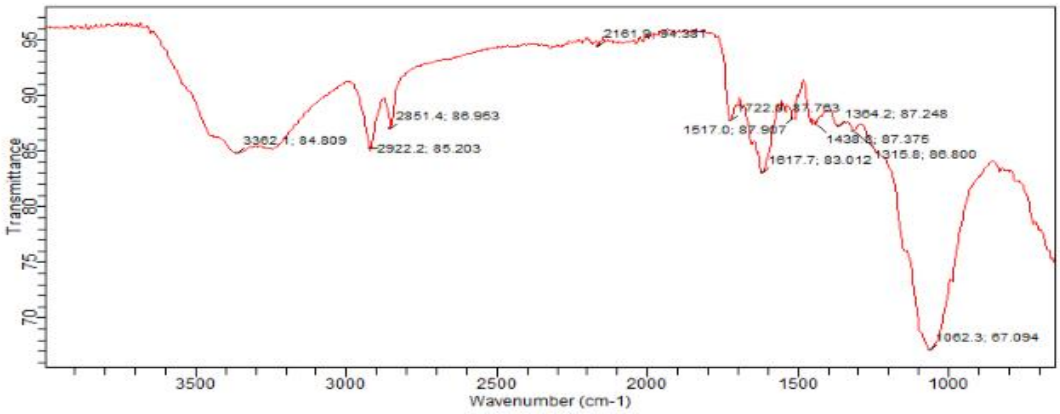

Figure 8 FTIR Spectral of (a) Ethanol Extract of ANP and (b) Corrosion Product of Mild Steel in the Presence of Inhibitor

Peaks and frequencies of FTIR adsorption for both spectra are presented in Table 2. From the results obtained, it is also evident that the C-O stretch at 1233.7 cm -1 was shifted to 1062.3 cm -1, the –C=C- stretch at 1617.7 cm -1 was shifted to 1517.7 cm -1, the C-H (Alkene stretch) was shifted from 2918.5 cm-1 to 2851.4 cm-1, the aromatic C=O was shifted from 1722.0 cm-1 to 1722.8 cm-1, the N=O (R-NO2) was shifted from 1518.3 cm-1 to 1384.2 cm-1 and the phenolic-OH stretch was shifted from 3283.8 cm-1 to 3362.1 cm -1. These shifts in frequencies also indicate that there is an interaction between the inhibitor and the metal surface [17, 18, 19]. It is also evident from the data obtained that the C-O stretch at 1159.2 cm-1 as well as N=O (R-NO2) at 1364.2 cm -1 were missing, suggesting that these bond frequencies might have been used for bonding between the vacant d-orbital of Fe and the inhibitor [26]. Therefore, ANLE was adsorbed onto the mild steel surface through these functional groups [27].

Table 2 Functional Groups Assigned to the Adsorption of the Ethanol Extract of the Pod and the Corrosion Product When the Extract is used as an Inhibitor

Ethanol extract			**Corrosion product**		
Wave No (cm^{-1})	Height	Assigned functional group	Wave No (cm^{-1})	Height	Assigned functional group
3283.8	87.207	O-H H-bonded	3362.1	84.809	O-H H-bonded
2918.5	77.986	C-H Alkene stretch	2851.4	86.953	C-H Alkane stretch
2851.4	81.634	C-H Alkene stretch	2922.2	85.203	C=O Aldehyde
1722.0	81.218	C=O Aldehyde	1722.8	87.783	C=O Aldehyde
1617.7	81.253	C=C Alkene	1517.7	83.012	C=C Alkene
1518.3	84.083	N=O Nitro (R-NO_2)	1384.2	87.248	N=O Nitro (R-NO_2)
1438.8	83.051	C-H -CH_3(bend)	1438.8	87.375	C-H -CH_3 (bend)
1364.2	82.596	N=O Nitro (R-NO2)	-	-	-
1159.2	75.688	C-O stretch	-	-	-
1233.7	78.520	C-O stretch	1062.3	67.094	C-O stretch

4 CONCLUSION AND RECOMMENDATION

The results obtained from the study indicate that ANP extract effectively inhibited the corrosion of mild steel in 0.1 M H2SO4. Inhibition efficiency increases with increasing extract concentration and immersion time and decreases with rising temperature. The inhibition potential of this inhibitor is attributed to the presence of phenol, tannin, alkaloids, and flavonoids in the extract. Hence, an increase in the reaction temperature of the medium will decrease the inhibition efficiency. In view of the above conclusion, the use of ethanol extracts of ANP as green inhibitors is recommended.

COMPETING INTERESTS

The authors have no relevant financial or non-financial interests to disclose.

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