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Original Research Article

Corrosion Behaviour of Mild Steel in the Presence of *Ceiba pentandra* Extract in Acidic Solution: Adsorption and Mechanism

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ABSTRACT

Despite the sustained interest in applying plant extracts as inhibitors of metal corrosion, deducing the detailed mechanisms of the inhibition process continues to pose some experimental challenges. To assign the inhibitive effect to the adsorption of any particular constituent is difficult due to the complex chemical compositions of the extracts. *Ceiba Pentandra* extract (*CP*) was evaluated as a green and renewable corrosion inhibitor for mild steel corrosion in a 1.0 mol/L HCl solution. The inhibition behavior was studied using weight loss, potentiodynamic polarization (PDP), and electrochemical impedance spectroscopy (EIS) measurements. In this study, the plant material was separated into various fractions and named the acidic fraction (AF), the basic fraction (BF), the neutral fraction (NF), and the crude fraction (CF). The basic fraction (BF), which yielded over 90% inhibition efficiency at the 500 mg/L dosage level, is considered the most promising fraction. PDP results suggest the BF was more a cathodic inhibitor while the rest were mixed-type inhibitors. Adsorption of the plant material on the mild steel surface obeyed the Langmuir adsorption isotherm. The basic fraction was further analyzed using Gas chromatography-mass spectrometry (GC-MS). The GC-MS result shows that CP extract possesses polar functional groups such as hydroxyl, carbonyl, and hetero-aromatic rings. The inhibition mechanism is closely related to these polar groups.

Keywords: *Ceiba Pentandra, Corrosion inhibitor,* Adsorption behavior, *Mild steel,* Potentiodynamic polarization, Electrochemical impedance.

Introduction

Metal corrosion is an aggressive and detrimental electrochemical process. Corrosion inhibitors are substances that can be added in small amounts to an environment to prevent the material from further dissolution.[1-3] Corrosion inhibitors have been extensively used in pickling, extraction, and refining operations in the oil and gas, chemical industry, energy, public infrastructure, construction, and associated industries.[3,4] The protection provided by corrosion inhibitors of organic origin is due to the adsorption of the inhibitor molecules on the metal surface that results in its surface modification and the consequent formation of a protective film.[5-7]

When choosing a corrosion inhibitor, factors such as efficiency, cost, availability, toxicity, and environmental safety need to be considered. Many of the inhibitors in current use, though efficient, are often toxic or even allergic.[7-9] Therefore, the need to search for more environmentally friendly and biodegradable inhibitors obtained from renewable resources with minimal health and safety concerns.[10-12] In the last two decades, the majority of the investigations on the development of new corrosion inhibitors are focused on cheap, efficient, and eco-friendly inhibitors of plant origin.[12-14] Plant extracts contain alkaloids, saponins, tannic acids, sugars, amino acids, and aromatics. Their electronic structures invariably possess π-electrons, electrons associated with O, N, and other hetero-atoms on non-ligands, with a proven corrosion inhibition effect. Thus, they may behave similarly to conventional organic compound-based corrosion inhibitors.[15-18]

Despite the sustained interest in plant extracts as inhibitors for metal corrosion control, deducing the detailed mechanisms of the process continues to pose a considerable experimental challenge. To assign the inhibitive effect to the adsorption of any particular constituent is difficult due to the complex chemical compositions of the extracts. Knowledge of the adsorption behavior of the inhibitor molecules on the substrate surfaces enables an understanding of the corrosion inhibition mechanism. A fundamental insight into the interaction between the metal surface and the inhibitor molecules can be provided by adsorption isotherms.[6, 11, 18-24]

The plant *Ceiba pentandra* is a member of the sub-family of *Malvaceae* (kapok, cotton family). In the past, this plant was classified under the *Bombacaceae* group. However, modern molecular and phytochemical findings supported the group under a subfamily of *Malvaceae*. The plants are tall perennial trees with swollen trunks, and brightly colored flowers, with large branches utilized as ornamentals or purely economic trees.[27] Some of the phytoconstituents identified from the CPE include alkaloids, anthocyanins, coumarins, flavonoids, lignans, neolignans, sesquiterpenes, lactones, sterols, tannins, and triterpenes, supporting evidence of its medicinal and nutritional uses.[27-30] Thus far, there is no mention of this plant as a source of corrosion inhibition.

The present study used Ceiba pentandra extract (CP) as a green, non-toxic, and renewable corrosion inhibitor for mild steel in a 1.0 mol/L HCl solution. The inhibition performance was studied based on weight loss and electrochemical measurements. Various fractions of the plant extract were obtained, named the acidic fraction (AF), the basic fraction (BF), the neutral fraction (NF), and a crude fraction (CF) prepared as described by Ejele *et al.*[25,26] The chemical structures of the extract were studied using gas chromatography-mass spectrometry

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(GC-MS) and in-depth absorption studies on mild steel by the active constituents in the extract were undertaken.

Materials and Methods

Collection and authentication of plant material
Fresh, matured, and disease-free leaves of *Ceiba pentandra* were collected from a garden at the Federal University of Technology Owerri in December 2020. A taxonomist authenticated the leaves at the Department of Crop Science Federal University of Technology Owerri, Imo State. The voucher number of the specimen deposited is CST/BC-010104. The leaves were taken to our laboratory, where they were, air-dried, ground, and kept in desiccators until needed.

Preparation of Inhibitor
The extraction of the plant active constituents was achieved in a Soxhlet extractor using 80 g of dried and ground material with 250 mL of ethanol and kept under reflux for up to 6 hours to complete extraction. The liquid extract materials were filtered using a Whatman No.1 filter paper to remove any solid plant material and subsequently concentrated using a rotary evaporator under vacuum at approximately 40^{o}C to obtain a gel. The gel was then reconstituted to desired concentration using the extracting solvent depending on the analysis.
The Phyto-screening of the extract showed the presence of alkaloids, amino acids, flavonoids, fatty acids, glycosides, saponins, and tannins. Also, from the crude extract, various inhibitor concentrations were prepared to evaluate the corrosion inhibition efficiency.

Preparation of the basic fraction
The preparation of basic fractions followed the method described by Ejele et al.[25,26] Some 100 mL of the extract was treated with dilute HCl and extracted with chloroform in a separating funnel. The lower chloroform layer was removed and reserved for preparing other fractions. The upper (HCl) layer was treated with dilute NaOH solution until the solution became basic. This solution was allowed to evaporate completely to form a gel, which was re-dissolved in ethanol and filtered.

Preparation of the neutral fraction
The chloroform layer obtained above was placed in a separating funnel and treated with dilute NaOH solution. After equilibration, the lower (chloroform) layer was removed and allowed to evaporate to dryness at room temperature to form a gel. This gel was re-dissolved in ethanol and filtered, and the solution obtained was used for preparing the acidic fraction.[25, 26]

Preparation of the acidic fraction
The aqueous alkaline layer obtained above was placed in a separating funnel and treated with 30 mL of dilute NaOH solution. After equilibration and removing the chloroform layer, the aqueous alkaline layer was treated with dilute HCl until the solution became acidic and a reddish-brown precipitate formed. This precipitate was filtered, washed with distilled water, and allowed to dry in air at room temperature, after which it was re-dissolved in ethanol and filtered. [25, 26]

Preparation of solutions
All chemicals were analytical-grade reagents. The electrolyte was a 1 mol L^{-1}HCl solution prepared using double-distilled water. Various concentrations of the additives were prepared from 50 to 1000 mg L^{-1}, with 200 mL of electrolyte for the weight loss studies.

Weight loss measurement
Working electrodes prepared from steel specimens with a composition (in wt%) of C: 0.17–0.24, P: 0.04, Mn: 0.30–0.60, S:0.05, and Fe: balance and cut to 4.0 x 2.0 x 0.15 cm dimensions were used for weight loss measurements. The duplicate specimens were immersed in the acid test solutions in the absence and presence of various concentrations of the crude CPE for 24 h at room temperature (30^{o}C). The time duration during the determination of the effect of temperature on the inhibition efficiencies was 3 hours in the temperature range of 40–60^{o}C. The specimens were then removed, rinsed in water and acetone, and finally dried and stored in a desiccator. The weights of the coupons before and after immersion were determined using an analytical balance with a precision of 0.1 mg.

Electrochemical measurements
Electrochemical techniques used in this study are limited to potentiodynamic polarization (PDP) and electrochemical impedance spectroscopy (EIS). The corrosion cell was a Potentiostat (VERSASTAT400) system with a V3 studio software containing a three-electrode configuration; a graphite rod was the counter electrode, and a saturated calomel electrode (Ag/AgCl) in a Lugging capillary bridge was the reference electrode. The working electrode was a mild steel sample (1.5 x 1.5 x 1.5 cm2, dimension). The mild-steel samples were embedded in epoxy resin so that only a 1.0 cm^2 area was available to the electrolyte. The exposed metal surfaces were abraded with 400, 500, and 600-grade emery paper, washed with double-distilled water, degreased with acetone, and dried before each experiment. The PDP curves were recorded in the potential range ±250mV and a scan rate of 0.33mVs-1 at 303K. Similarly, the EIS data obtained, using a voltage of 10mV and frequency from 100 kHz to 10 MHz was analyzed with the ZSimpwin program. We took triplicate measurements to ensure the data obtained was reproducible.

Results and Discussion

Analysis of results of weight loss method
The corrosion process of mild steel in 1M HCl in the absence and presence of various concentrations of CPE (crude extract) was studied by the weight loss method at 303K. Table 1 shows the variation of corrosion rate (mg/cm^2-h) and inhibition efficiency (IE %) with varying extract concentrations.
Equations 1 and 2 represent the formula for calculating the inhibition efficiency of the additive and the resulting decrease in the corrosion rate (C_R) of the mild steel.[20]

$$\text{IE\%} = \frac{w_o - w_1}{w_o} \text{x100} \qquad (1)$$

$$C_R = \frac{\Delta w}{st} \qquad (2)$$

Where w_o and w_i are the weight losses in the absence and presence of the extract, respectively, Δw is the mean weight loss (mg), s is the area of the specimen (cm^2), and t is the immersion time (h).
The extract showed a maximum efficiency of 86% at an optimum concentration of 500 mg/L. Further increase in extract concentration did not cause any significant change in the extract's performance. The decreasing corrosion rate and increasing inhibition efficiency can be attributed to the phytoconstituents of the extract.

Table 1: Weight loss data for mild steel corrosion in 1 M HCl solution at 303 K without and with several concentrations of CP extract

System Mg/L	**Weight loss (mg)**	**C_R (mg/cm^2-h)**	**(IE%)**	**Surface Coverage (θ)**
1 M HCl	159.7	0.216	-	-
50	35.9	0.078	68.51	0.69
100	26.8	0.058	76.16	0.76
300	20.7	0.042	81.58	0.82
500	15.8	0.034	85.95	0.86
700	16.3	0.036	85.49	0.86
1000	19.1	0.041	83.03	0.83

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Effect of acid concentration and time on the inhibition efficiency of CP

To assess the stability of inhibitive behavior of the extract on a time scale, weight loss measurements were performed in 1 M HCl in the absence, and the presence of 500 mg/L CP extract for 168 h immersion period at 303 K. The obtained results are presented in Figure 1(a) which shows that the IE% of the extract decreased with increasing immersion time.

The variation of inhibition efficiency with an increase in acid concentration is shown in Figure 1(b). From this plot, it can be seen that the inhibition efficiency decreased from 86% to 70%. This result shows that this inhibitor will still be effective beyond 5 M acid concentration.

Effect of temperature

The effect of temperature on the inhibitory action of CPE was determined by the weight loss method for a fixed immersion time of 3 h at different temperatures (Figure 2). The results in Figure 2 clearly show that the inhibition efficiency decreased with increasing temperature at all concentrations.

The increasing temperature introduced some instability in the system, leading to desorption of the already adsorbed extract constituent, suggesting that the extract constituents were only physically adsorbed on the surface of the metal.[27-29]

The dependence of corrosion rate on temperature can be expressed by the Arrhenius equation: [31-34]

$$C_R(T) = Ae^{-E_a/RT} \text{ or } \ln C_R(T) = lnA - E_a/RT \quad (3)$$

Where E_a is the apparent activation energy, R is the universal gas constant, T is the temperature (K), and A is the Arrhenius pre-exponential factor.

Figure 3 shows the plot of $\ln C_R(T)$ vs. 1/T. The apparent activation energy (E_a) was obtained from the slopes of the graphs. The value of the activation energies (E_a) of the corrosion of mild steel in 1 M HCl solution in the presence of extract is higher than that in the free acid solution, 116.32 and 120.30 kJ/mol-K, respectively.

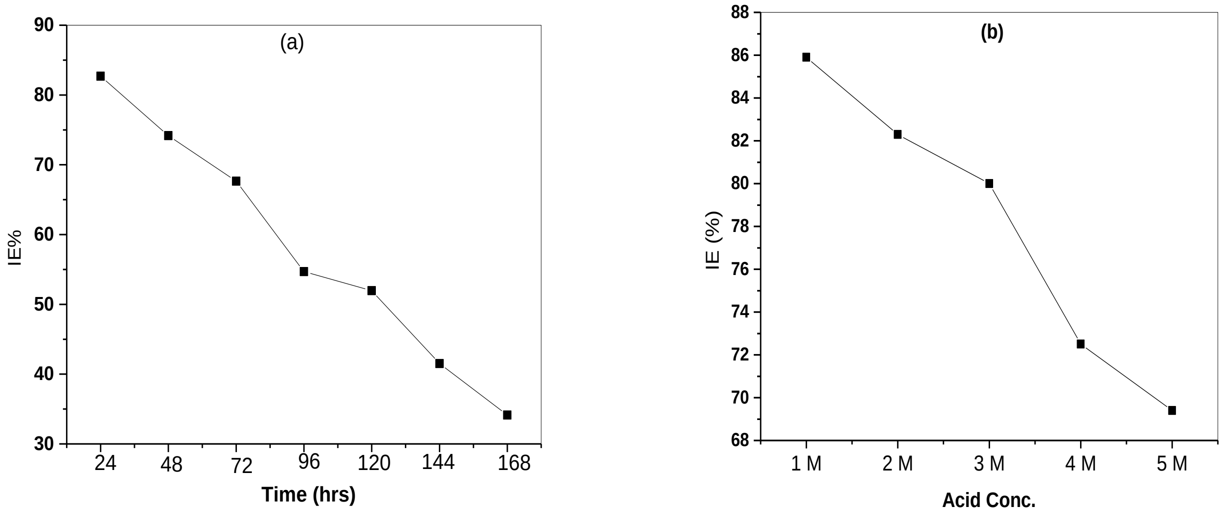

Figure 1: Variation of inhibition efficiency with (a) immersion period and (b) acid concentration at 303K

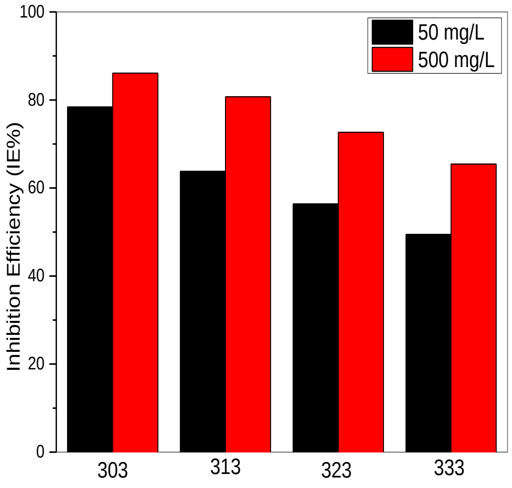

Figure 2: Variation of inhibition efficiency of CP at different temperatures during corrosion of mild steel in acidic medium

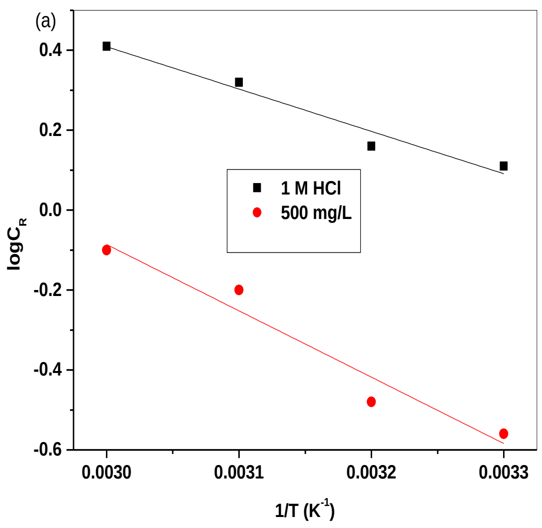

Figure 3: Arrhenius plot for mild steel corrosion in 1 M HCl containing 500 mg/L CP extract

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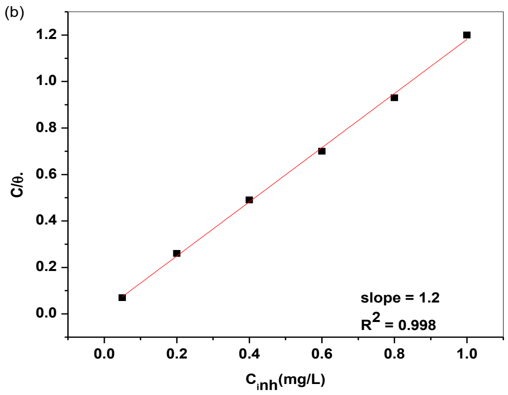

Figure 4: Langmuir adsorption isotherm for adsorption of CP extract on mild steel surface at 303 K

Szauer and Brand,[35] Popova *et al.*[36] attributed the higher activation energy of the inhibited solution than that in the free acid to physical adsorption arising from the decrease in the adsorption of the inhibitor on the mild steel surface with rising temperature.

Adsorption behavior

In view observations above, the inhibitive action of CP extracts toward the acid corrosion of steel can be attributed to the adsorption of its phytochemical components onto the steel surface. The adsorbed layer acts as a barrier between the steel surface and the aggressive solution, leading to a decrease. To obtain the isotherm, the surface coverage values (θ) as a function of inhibitor concentration were obtained from the weight-loss data as follows: [37-39]

$$\theta = \frac{w_o - w_i}{w_o} \quad (4)$$

w_o, nd w_i were defined in equation 1.

When the extract concentration (C)) is plotted against (C/θ), a straight line with almost a unit slope (1.2) is obtained, as shown in Figure 4. This behavior suggests that the adsorption of CP extract phytoconstituents onto the steel surface obeys the Langmuir adsorption isotherm expressed according to Eq. (5):[38, 39]

$$\frac{C}{\theta} = 1/K + C \quad (5)$$

where C is the inhibitor concentration, and K is the equilibrium constant for the adsorption/desorption process. The strong correlation coefficient (R^2 = 0.998) for the Langmuir adsorption isotherm plot confirms the validity of this approach. It is essential to note that discussion of the adsorption isotherm behaviour of natural product extracts as inhibitors in terms of the standard free energy of adsorption value is not possible because the extract components' molecular mass is unknown.

The inhibitive action of the extract fractions

Polarization measurement

Figure 5 shows mild steel's cathodic and anodic polarization curves in uninhibited and inhibited acidic solutions containing 50 and 250 mg/L of the different CP extract fractions. As can be seen from the polarization results after adding the extract fractions, a decrease in bathodic and anodic currents was observed.

The reduction in the corrosion current in the presence of the additives suggests that the corrosion of mild steel in the acidic solution was hindered. As shown in Figure 5c, there was a significant shift in the E_{corr} potential to less negative values. The addition of the BF indicates that this fraction represents the predominant cathodic reaction and behaves as a cathodic inhibitor.[40] The extrapolation of the Tafel straight line allowed the calculation of the corrosion current density (I_{corr}), the corrosion potential ($E_{corand\ r}$), cathodic and anodic Tafel slopes (β_c, β_a) as given in Table 2. The inhibition efficiency η% values were obtained from the measured I_{corr} using eq. 6:[40]

$$\eta\% = 1 - \frac{I_{corr}(inh)}{i_{corr}} \quad (6)$$

Where (I_{corr}) and ($I_{corr(inh)}$) are thisorrosion current densities, measured in solution without and with inhibitor.

The variations in the β_c values in the absence and presence of CP extract fractions, especially for NF, represent a change in the mechanism of inhibition. From the inhibition efficiencies obtained by this method, it is evident that the basic fraction represents the most effective fraction in 1 M HCl. Figure 6 shows the impedance spectra in Nyquist format obtained in the absence and the presence of 50, and 50 mg/L of the various fractions of CPE fractions (NF, BF, AF, and CF) in the acidic solutions, respectively. It was observed that the higher concentrations of the investigated inhibitor, the more elevated the impedance values. In addition, the presented impedance spectra take the form of characteristic capacitive semi-circles that correspond to one time constant. To gain more detailed information on the adsorption mechanism of the CP extract, impedance data were analyzed using an appropriate equivalent electrical circuit, as shown in Figure 7. This circuit considers the inhomogeneity at the solid/liquid interface, where R_s is the solution resistance, R_{ct} represents the charge transfer resistance, and CPE represents constant phase elements to replace the double-layer capacitance (C_{dl}).[41,42] Double-layer capacitance (C_{dl}) is defined as:[42]

$$C_{dl} = Y_o(\omega_{max})^{n-1}$$

whereY_o is the magnitude of the CPE, ω_{max} is the frequency at which the imaginary part of impedance has a maximum (rad/s), and n is the C_{dl} exponent. The charge transfer resistance (R_{ct}) obtained from the analysis of appropriate electrical allowed calculating the inhibition efficiency (η%) in the absence and presence of different inhibitor concentrations according to the equation below:

$$\eta\% = \frac{R_{ct(inh)} - R_{ct(bl)}}{R_{ct(inh)}} \quad (7)$$

Where$R_{ct(inh)}$ and $R_{ct(bl)}$ are charge transfer resistance values and without inhibitors, respectively.

It can be seen from Table 3 that an increase follows an increase in the Rct value e in the CP extract inhibition efficiency in all the fractions.

The change in C_{di} values might be explained in one of tw either by decreasing the local dielectric constant or increasing the double layer thickness. The former occurs when water molecules adsorbed on the mild steel surface are substituted by CPE constituents. The dielectric constant for water equals 80. Once the greater area of the metal surface is covered with CP extract the $C,_{di}$values decrease. Larger CP extract constituents' coverage causes an increase in the double layer thickness. However, the most significant result was obtained with the basic fraction, with a value equal to 92.7% at 250 mg/L. This is evident from the size of the diameter of the plot containing BF and points to the fact that the BF could be the most effective fraction in an acidic medium. The potential dynamic polarization results corroborate this observation.

Inhibition mechanism

GC-MS chromatogram of the ethanol extract of CP showed seven prominent peaks in Figure 8. The seven active constituents with their retention time (RT), molecular formula, molecular weight (MW), and peak area (%) are presented in Table 4. Inspection of the structures reveals that these compounds contain O in functional. In an acidic solution, the oxygen atom of these chemical compounds can be protonated easily, leading to positively charged inhibitor species. In acid solution, the metal surface is negatively charged with the presence of the specifically adsorbed chloride anions on the metal surface.

The anodic dissolution of iron occurs according to the following steps: [41, 43]

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$Fe + Cl^{-} \leftrightarrows (FeCl^{-})_{ads}$

$(FeCl^{-})_{ads} \leftrightarrows (FeCl)_{ads} + e^{-}$

$Fe + Cl^{-} \leftrightarrow (FeCl)_{ads} + e^{-}$

$(FeCl)^{+} \leftrightarrow Fe^{2+} + Cl^{-}$

The cathodic hydrogen evolution follows the steps:[41, 43]

$Fe + H^{+} \rightleftarrows (FeH^{+})_{ads}$

$(FeH^{+})_{ads} \mapsto (FeH)_{ads}$

$(FeH)_{ads} + H + e^{-} \mapsto Fe + H_2$

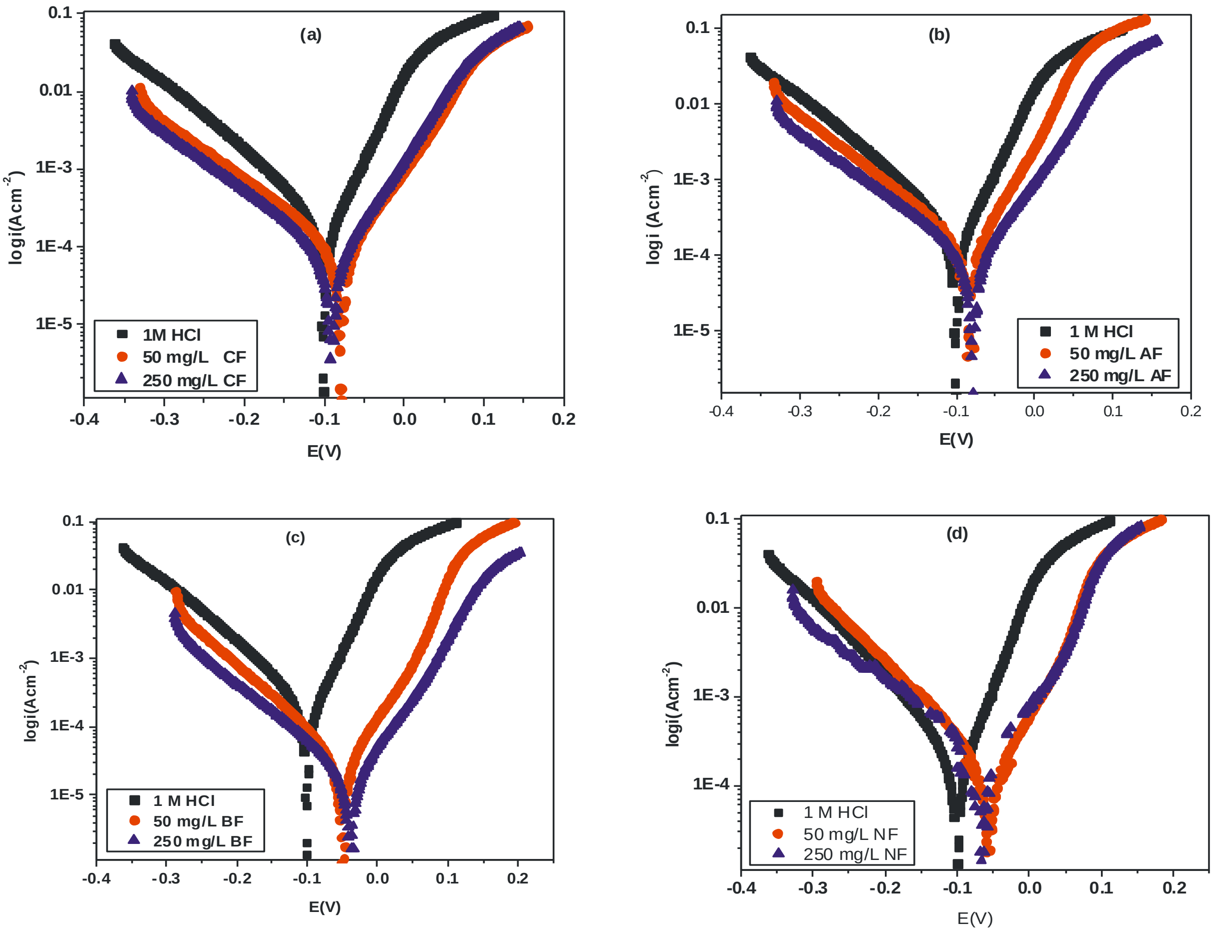

Figure 5: Polarization curves in the absence and presence of the various fractions of CP extract (a) crude fraction (CF); (b) acidic fraction (AF); (c) basic fraction (AF); and (d) neutral fraction (NF

Table 2: Potentiodynamic polarization parameters for the corrosion of mild steel in 1 M HCl without and with different CP extract fractions

System	**conc. mg/L**	**-E$_{corr}$ (μAcm-^{2})**	**I$_{corr}$ (mVde**	**ß$_{c}$ (mVdec^{-1})**	**ß$_{a}$ (μAcm-^{2})**	**IE%**
Blank	0.00	-100.60	176.51	108.16	55.80	0.00
AF	50	-78.78	82.37	109.90	53.80	52.35
AF	250	-82.25	84.10	107.30	72.90	53.31
BF	50	-49.24	22.06	117.42	61.65	87.48
BF	250	-38.93	16.48	112.91	70.71	90.67
CF	50	63.55	76.17	129.40	76.10	56.83
CF	250	-90.36	68.46	126.60	70.11	61.20
NF	50	-57.37	90.08	155.84	66.81	49.00
NF	250	-62.09	62.00	151.21	94.00	64.87

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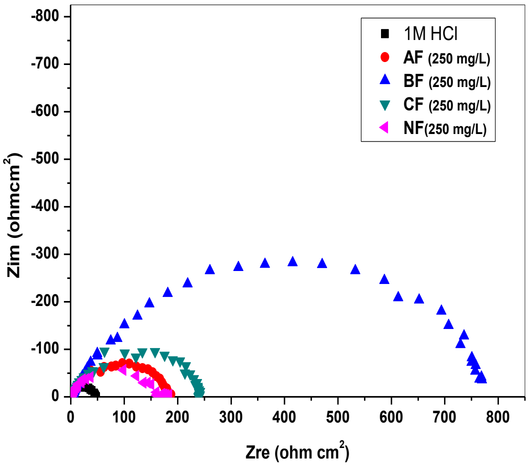

Figure 6: Nyquist plots in the absence and presence of various CP additives in 1 M HCl (a) the acidic fraction (AF), the basic fraction (BF), the crude fraction (CF), and the neutral fraction (NF).

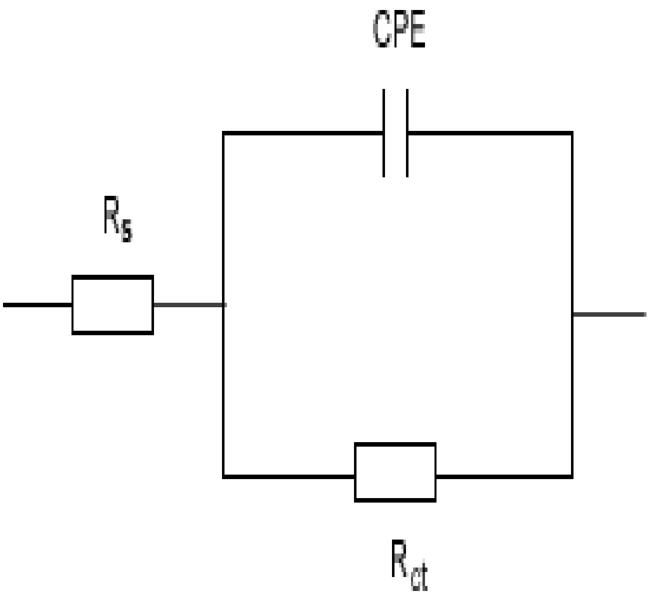

Figure 7: Equivalent circuit model represents the metal/solution interface, CPE the constant phase element,R_{ct}, Charge transfer resistance, and RS the solution resistance

Table 3: Electrochemical impedance parameters for mild steel in 1 M HCl in the absence and presence of various concentrations of CPE fractions

System	Conc (mg/L).	R_s (Ω cm^2)	R_{ct} (Ω cm^2)	C_{dl} (μFcm^{-2})	I.E. (%)
Blank	0.00	1.22	57.44	456	0.00
AF	50	1.48	131.2	222	56.2
AF	250	3.62	178	127	67.7
BF	50	4.96	478	17.6	88.0
BF	250	2.64	789	7.1	92.7
CF	50	2.17	95.5	455	40.0
CF	250	3.12	240.2	70.1	76.0
NF	50	1.76	112.5	315	48.9
NF	250	1.96	164.3	172	65.0

Table 4: Phytocomponents identified in the ethanol extract of the leaves of CP extract (Basic Fraction) by GC-MS

Peak Number	Peak area	Retention time	Name of Compound	Molecular Formula	Molecular Weight
1	2.74	5.211	Glyceric aldehyde	$C_3H_6O_8$	90
2	13.07	14.561	1,6-Anhydro-beta-D-glucopyranose	$C_6H_{10}O_5$	162
3	25.95	18.408	Hexadecanoic acid	$C_{16}H_{32}O_2$	256
4	2.64	20.142	11-Octadecanoic acid methyl ester	$C_{19}H_{36}O_2$	296
5	2.30	20.375	3,7,11,15-Tetramethyl-2-hexadecen-1-ol	$C_{20}H_{40}O$	296
6	40.42	21.092	Oleic acid	$C_{18}H_{34}O_2$	282
7	12.87	21.400	Octadecanoic acid, 2-(2 hydroxyethyl) ester	$C_{22}H_{44}O_4$	372

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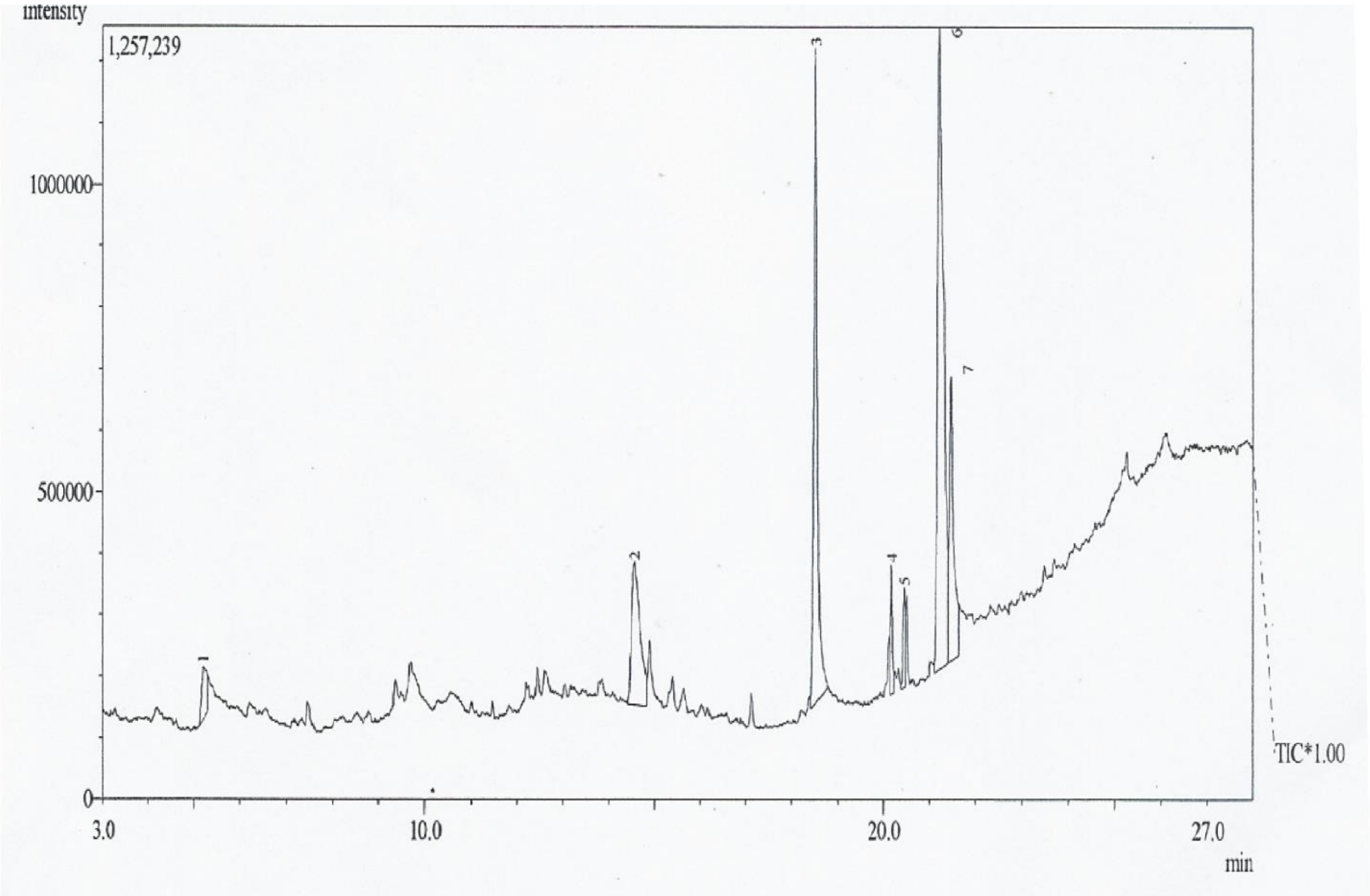

Figure 8: GC-MS for CP extract from ethanol solution

The $FeCl^-$adsorbed competed with chloride anion in the anodic branch, and then the species $(FeCl^-)_{ads}$charged inhibitor cations to form monomolecular layers as a complex on the mild steel surface. These layers preserve the metal surface from attack by chloride ions. Also, the protonated extract molecules are adsorbed at cathodic sites in competition with hydrogen ions, reducing hydreducedevolution.[15] Polarization curves indicate that all used extract fractions have an inhibition effect on both cathodic and anodic reactions of the corrosion process. Therefore, these compounds can be classified as mixed-type inhibitors with a chiefly cathodic action.[44-46]According to this mechanism, the adsorption between the positively charged inhibitor molecules and the negatively charged metal surface can only lead to the physisorption of the inhibitor molecules on the metal surface.

Conclusion

Corrosion behavior of mild steel in 1 M HCl solution in the absence and presence of *Ceiba pentandra* extract was examined by gravimetric and electrochemical measurements after separation into various fractions, named the acidic, the basic, and neutral fraction.s

Both Tafel polarization and electrochemical impedance techniques revealed that the higher the concentration of the inhibitor, the greater the inhibition efficiency achieved. In addition, the highest inhibition efficiency of around 90% was obtained with the basic fraction at 500 mg/L concentration for both methods.

The observed inhibition efficiency is a synergic contribution of the various component that make up the extract. Some components can perform even better if liberated from the extracted matr,ix as confirmed by the higher inhibition efficiency of the basic fraction.

The corrosion process was inhibited by adsorption of the organic matter on the mild steel surface and approximated by the Langmuir isotherm.

Conflict of Interest

The authors declare no conflict of interest.

Authors' Declaration

The authors now declare that the work presented in this article is original and that they will bear any liability for claims relating to the content of this article.

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