

Determination of the corrosion inhibiting properties of ethanol extract of *Pennisetum purpureum* in acidic solutions

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DOI: <https://doi.org/10.51244/IJRSI.2023.10730>

Received: 01 July 2023; Revised: 26 July 2023; Accepted: 29 July 2023; Published: 28 August 2023

Abstract: - The corrosion inhibition of mild steel by leave extract of *Pennisetum purpureum* (PP) in 1 M HCl and H₂SO₄ was studied using gravimetric, electrochemical and computer simulation methods of corrosion monitoring. The results obtained showed that the PP extract retarded the mild steel corrosion in both acidic solutions and that inhibition efficiency improved with concentration increase but decreased gradually with prolonged exposure time. Electrochemical results revealed that the extract reduced both the anodic and cathodic corrosion reactions by the adsorption of some of its chemical constituents on the metal surface. To further confirm the adsorption characteristics of the extract, some of its constituents were simulated using density functional theory simulation method.

Keywords: Corrosion, inhibitor, *Pennisetum purpureum*, polarization, simulation

I. Introduction

Corrosion is the gradual and continuous deterioration of metals exposed to the environment, corrosion is as old as age itself [1-3]. Scientists have employed many methods to control this environmental menace. One of the most effective ways of corrosion control is the use of corrosion inhibitors [4-5], these are compounds known to effectively retard the undesirable destructive effects of aggressive solutions and prevent the dissolution of metals and their alloys [6-7]. Acidic media which are always employed in industrial acid cleaning, acid pickling as well as acid descaling, need the use of corrosion inhibitors to reduce corrosion on metallic materials. Lately, many corrosion inhibitors used in corrosion control are sourced from either organic or synthetic background with little or no modification. Some of these inhibitors are imidazole based inhibitors and Chromates. The applications of these materials are currently restricted due to their toxicity and other environmental issues. Many plant materials have been in use as corrosion inhibitors for metals in acidic and alkaline environments, [8-12]. Their main advantages include low cost, environmentally friendly and readily available [13-15]. The inhibitive performance of plant materials are usually due to the presence in their composition of the species of complex organics which includes alkaloids, tannins and nitrogenous bases as well as proteins, carbohydrates and degradation products [16-17]. These inhibitors function in many ways to control corrosion: by adsorption as a thin film onto the metal surface. Inhibitors reduce corrosion reactions by reducing both the anodic or cathodic corrosion current density in an electrochemical environment, reducing the movement or diffusion of electrons to the surface of the metal and increasing the electrical resistances of the surface [18]. This work reports the corrosion inhibition behavior of *Pennisetum purpureum* in 1 M HCl and 0.5 M H₂SO₄ respectively.

II. Materials and method

2.1 Materials

The mild steel specimen used for the experiments has the composition (weight %) C -0.30, Si – 0.30, Mn – 0.30, P – 0.045, S – 0.050, Cr – 0.064, Cu – 0.040, Ti – 0.04 and the balance Fe [19]. Analytical grade reagents (distilled water, acetone, absolute ethanol, HCl and H₂SO₄) were used in the preparation of materials. The plant material used for the experiment was collected from the botanical garden of the Imo State University Owerri, dried at 25°C (room temperature) ground with a mechanical grinder and kept for further use. 25 g of the plant material was dipped 1000 ml of absolute ethanol and kept for 72-h, and then triple filtered. Quantification of the amount of the plant material that was extracted into the solution was done by subtracting the weight of the dried residue from the weight of the plant material before extraction. The test solutions were then prepared from the stock solution by diluting in the concentrations of 200, 400, 600, 800 and 1000 mg/L.

2.2 Gravimetric Analysis

The metal specimens used for the gravimetric experiments have the dimension: 3 cm x 3 cm x 0.14 cm [20], they were abraded under clean water with silicon carbide abrasive paper of range #150 – #1000, washed with clean water, dried using acetone and warm air, weighed and stored in a desiccator for further use. Gravimetric tests were performed under aerated conditions by fully immersing the coupons into 300 ml test solutions at 303 K, the Coupons were removed and washed at 24h intervals progressively

for 120-h, the cleaning process entails dipping the metal coupons into a solution containing 20% NaOH and zinc dust [21] to momentarily stop the corrosion process and scrubbing the metal under running water, re-weighed and re-immersed into the test solution. The weight loss was calculated by subtracting the initial weight of the metal coupons from the weight after a given time. Each test was done in triplicate to ensure good results.

2.3 Electrochemical experiments

All electrochemical experiments (electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (pdp)) were conducted in PAR-2273 Advanced Electrochemical System workstation [19] using a conventional three electrode corrosion system. The working electrode was a mild steel specimen encased in epoxy resin with an exposed surface of area 1 cm², saturated calomel and platinum sheet served as the reference and counter electrodes respectively. The temperature of reaction was fixed at 3 ± 1 °C, all the electrochemical measurements were done in an aerated and unstirred solutions at the end of 1800 s of immersion, this condition allowed the OCP values to reach steady state. Electrochemical impedance spectroscopy measurements were carried at corrosion potentials (E_{corr}) using a frequency range of 100 kHz - 0.1 Hz, the signal amplitude of perturbation was maintained at 5 mV. The potentiodynamic polarization measurements were performed at a potential range of ±250 mV versus corrosion potential within a scan rate of 0.333 mV/s [22]

2.4 Theoretical Modeling

All quantum chemical calculations were done with the density functional theory (DFT) electronic structure workstation with DMol3 [23] as found in the Materials Studio 7.0 simulation software (Accelrys, Inc.).

III. Results and discussion

3.1 Gravimetric Results

To ascertain the effect of concentration and time on the corrosion inhibition of mild steel in the presence of the inhibitor, gravimetric experiments were conducted. The results are presented in Figures 1 and 2 as well as Tables 1 and 2.

Table 1 Concentration and weight loss values for mild steel corrosion at different immersion times.

Conc. (mg/L)	Average weight loss (g)									
	24 h		48 h		72 h		96 h		120 h	
	1 M HCl	0.5 M H ₂ SO ₄	1 M HCl	0.5 M H ₂ SO ₄	1 M HCl	0.5 M H ₂ SO ₄	1 M HCl	0.5 M H ₂ SO ₄	1 M HCl	0.5 M H ₂ SO ₄
0	0.1502	0.283	0.3774	0.6345	0.4731	0.9051	0.5667	1.2351	0.7442	1.8957
200	0.0468	0.0982	0.1366	0.2392	0.2064	0.3557	0.2700	0.6114	0.3827	1.0388
400	0.0360	0.0835	0.1084	0.1961	0.165	0.3241	0.2095	0.4928	0.3169	0.7943
600	0.0281	0.0784	0.0766	0.1891	0.1160	0.2815	0.1706	0.4483	0.3018	0.7564
800	0.0159	0.0682	0.0508	0.1707	0.0967	0.2670	0.1627	0.4236	0.2954	0.7261
1000	0.0092	0.0416	0.0406	0.1009	0.0854	0.1530	0.978	0.2495	0.1843	0.4758

Table 2 Concentration and inhibition efficiency values for mild steel corrosion at different immersion times

Conc. (mg/L)	Inhibition efficiency (%)									
	24 h		48 h		72 h		96 h		120 h	
	1 M HCl	0.5 M H ₂ SO ₄	1 M HCl	0.5 M H ₂ SO ₄	1 M HCl	0.5 M H ₂ SO ₄	1 M HCl	0.5 M H ₂ SO ₄	1 M HCl	0.5 M H ₂ SO ₄
200	68.8	65.3	64.6	62.3	52.4	60.7	48.4	50.5	65.3	45.2
400	76.0	70.5	71.3	69.1	63.9	64.2	57.3	60.1	70.5	58.1
600	81.3	72.3	79.8	70.2	63.0	68.2	59.3	63.7	72.3	60.1
800	89.4	75.9	86.6	73.1	71.3	70.5	60.2	65.7	75.9	61.7
1000	93.4	85.3	89.2	84.1	82.7	83.1	75.2	79.8	85.3	74.9

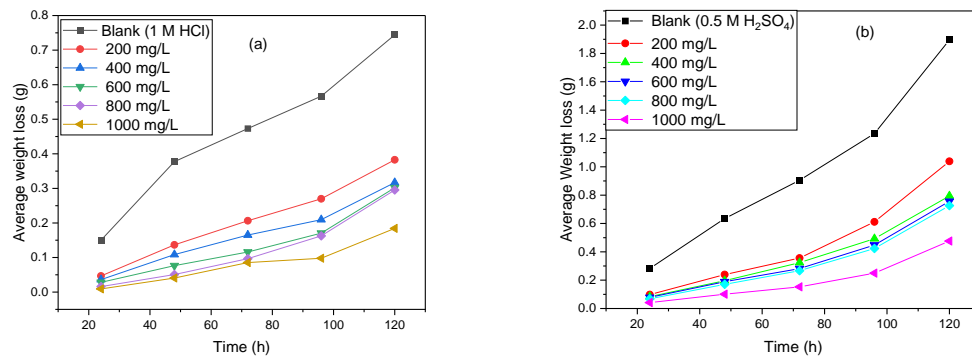


Figure 1. Weight losses of mild steel in (a) 1 M HCl and (b) 0.5 M H₂SO₄ solutions in the absence and presence of PP extract, as a function of immersion time.

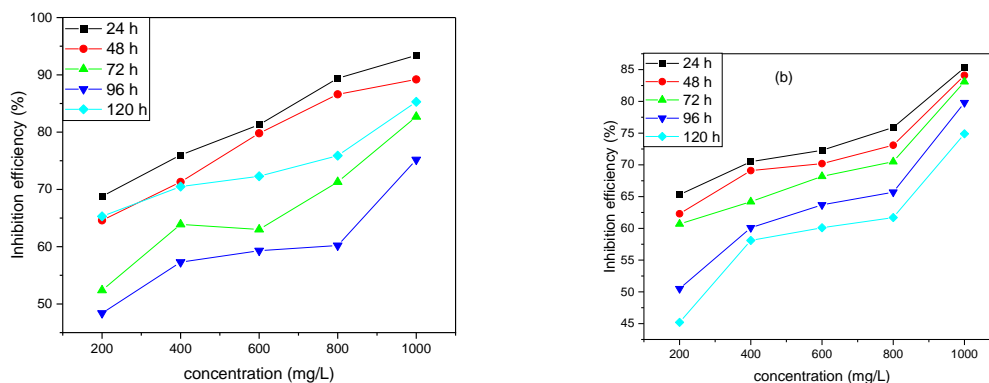


Figure 2. Relationship between inhibition efficiency of PP extracts and concentration for mild steel corrosion in (a) 1 M HCl and (b) 0.5 M H₂SO₄ solutions.

The results in Figure 1 (a&b) shows the plot of average weight loss against time for mild steel corrosion in the presence different concentrations of *Pennisetum purpureum* while Figure 2 (a &) shows the plot of inhibition efficiency against concentration for mild steel corrosion in the presence of the inhibitor. Figure 1 and Table 1 show that average weight loss increases with time while

Figure 2 and the corresponding Table 2 show that inhibition efficiency increased with concentration of the inhibitor showing that the rate of mild steel corrosion was significantly retarded in the presence of the inhibitor for all immersion times, The inhibition efficiency from the gravimetric data was calculated by the equation below: [24]

$$IE(\%) = \left(1 - \frac{w_1}{w_2}\right) \times 100 \tag{1}$$

Where w_1 and w_2 represent the weight losses of mild steel in the inhibited and uninhibited solutions respectively.

3.2 Electrochemistry Results

(a) Electrochemical Impedance Spectroscopy (EIS) Results

To ascertain the kinetics of the electrochemical reaction and also the corrosion mechanisms at the Fe/0.5-M H₂SO₄ and Fe/1-M HCl interfaces without and with the inhibitor, electrochemical impedance spectroscopy studies were undertaken. Figure 3 and 4 show the impedance responses of mild steel in the absence and presence of PP extract in (a) 1 M HCl and (b) 0.5-M H₂SO₄ solutions, the electrochemical parameters from the impedance experiments are presented in Table 3. The Nyquist plots show a depressed capacitive semicircle over the frequency range that was studied; this reveals a one-time constant in the Bode plots. In the Nyquist semicircles, the high frequency that intercept with the real axis is called the solution resistance (R_s) whereas the low frequency that intercept the real axis is called the charge transfer resistance (R_{ct}). The Nyquist plots were analyzed using an equivalent circuit $R_s(Q_{dl}R_{ct})$ [25-27] which has been used to model different mild steel/acid interfaces. In the equivalent circuit used, R_s is shorted by a constant phase element (CPE) placed parallel with the R_{ct} . The use of CPE instead of a capacitor is to account for deviations from dielectric behavior that may arise from the inhomogeneous nature of the surface of the electrodes. The impedance of the constant phase element is given as below:

$$Z_{CPE} = Q^{-1}(j\omega)^{-n} \tag{2}$$

Where Q and n stand respectively for CPE constant and element [28-29] respectively, j is an imaginary number with value $j = (-1)^{1/2}$ while ω is the angular frequency in $rad\ s^{-1}$ with value ($\omega = 2\pi f$) and f is the frequency in Hz. Table 3 shows that the addition of the inhibitor increased the value of the R_{ct} showing that the inhibitor reduced the corrosion of mild steel in both 1 M HCl and 0.5-M H₂SO₄ respectively. The inhibition efficiency (IE %) from the impedance results was estimated by comparing the values of the charge transfer resistance without the inhibitor ($R_{ct,bl}$) and with the inhibitor ($R_{ct,inh}$) as follows: [30].

$$IE\% = \left[\frac{R_{ct,inh} - R_{ct,bl}}{R_{ct,inh}}\right] \times 100 \tag{3}$$

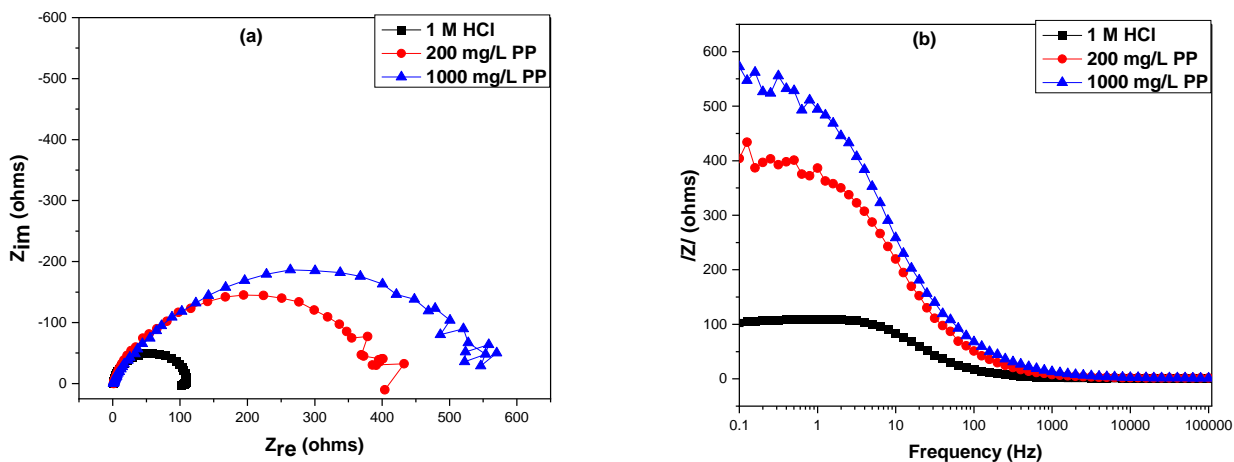


Figure 3. Electrochemical impedance spectra for mild steel in 1 M HCl solution without and with PP extract: (a) Nyquist and (b) Bode plots

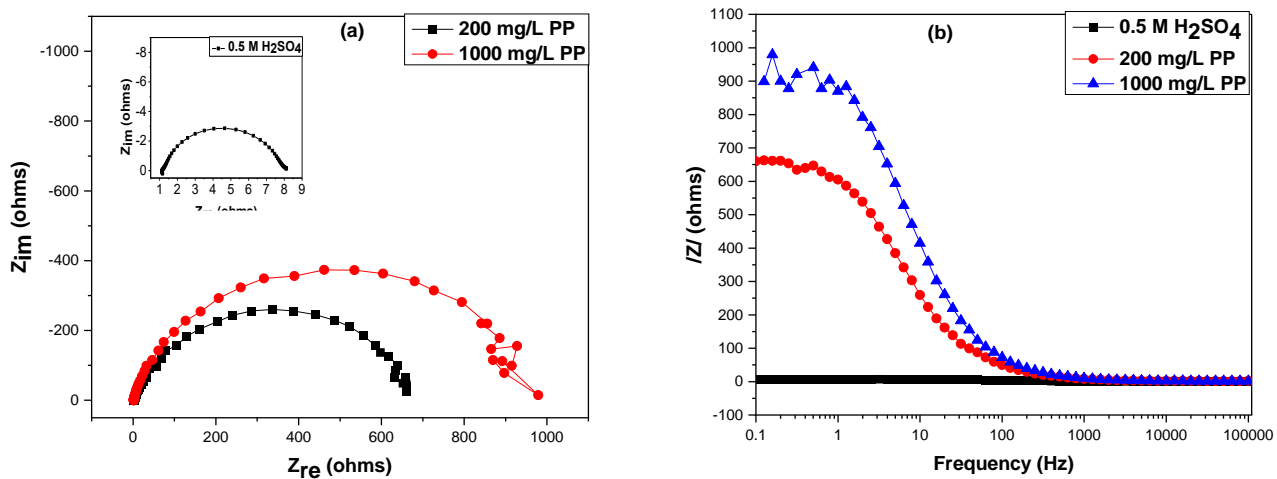


Figure 4. Electrochemical impedance spectra of mild steel in 0.5 M H₂SO₄ solution without and with PP extract: (a) Nyquist and (b) Bode plots. Inset in panel in the Nyquist plot is the plot of the uninhibited acid.

Table 3. Impedance Results for Mild Steel Corrosion in 1 M HCl and 0.5 M H₂SO₄ without and with PP Extract.

System	R _{ct} (Ω cm ²)	n	Q _{dl} (μΩ ⁻¹ S ⁿ cm ⁻²)	IE%
1 M HCl	104	0.88	91.7	
200 mg/L PP	382	0.89	32.4	72.8
1000 mg/L PP	525	0.89	23.3	80.1
0.5 M H₂SO₄	8.4	0.84	258.8	
200 mg/L PP	640	0.89	20.4	98.7
1000 mg/L PP	910	0.89	13.7	99.1

(b) Potentiodynamic polarization Results

To understudy the effects of the inhibitor on the anodic metal dissolution as well as the cathodic hydrogen evolution reactions, potentiodynamic polarization measurements were carried out. Figure 5 presents the potentiodynamic polarization curves for mild steel in (a) 1 M HCl and (b) 0.5 M H₂SO₄ solutions. Rapid dissolution of the metal was observed in the absence of PP extract. The corrosion parameters [corrosion current densities (*i_{corr}*) and corrosion potential (*E_{corr}*), calculated from the polarization curves are presented in Table 4. The results showed that the presence of the PP extract reduced the corrosion current densities in both 1 M HCl and 0.5 M H₂SO₄ solutions. The corrosion potential was also shifted slightly towards the negative value by the inhibitor, all these indicate that the extract inhibited the corrosion of mild steel in the solutions studied in line with the results reported elsewhere [31]. The inhibition efficiency was estimated from the polarization data by comparing the corrosion current density in the uninhibited solution (*i_{corr,inh}*) and the corrosion current density in the presence of the inhibitor (*i_{corr,bl}*) as follows: [32]

$$IE (\%) = \left(\frac{i_{corr,bl} - i_{corr,inh}}{i_{corr,bl}} \right) \times 100 \tag{4}.$$

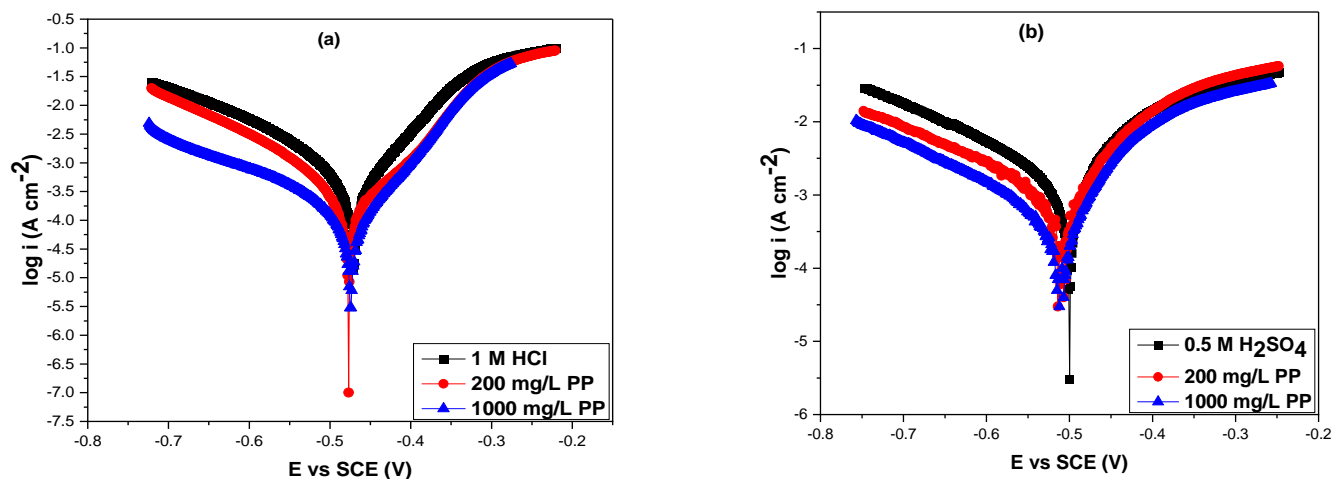


Figure 5. Potentiodynamic polarization curves of mild steel in (a) 1 M HCl and (b) 0.5 M H₂SO₄ solution without and with PP extract

Table 4 Potentiodynamic polarization parameters for mild steel corrosion without and with PP extract in acidic media

System	E _{corr} (mV vs SCE)	I _{corr} (μA/cm ²)	IE%
1 M HCl	-471.3	649.4	
200 mg/L PP	-449.8	588	9.4
1000 mg/L PP	-491.4	360.5	44.5
0.5 M H₂SO₄	-500	2940	
200 mg/L PP	-480.1	165.4	94.4
1000 mg/L PP	-505.7	88.3	97

3.3 Computational Studies

To determine the correlation between inhibitive efficiency of the inhibitor and molecular structure, two chemical constituents of the inhibitor (15-Hydroxypentadecanoic acid and 3,7,11,15-Tetramethyl-2-hexadecen-1-ol) were sourced from literature [33] and simulated. Density functional theory (DFT) was used to evaluate information relating the molecular geometries and electron distributions of the constituents which influenced the inhibition performance. DFT calculations were achieved using the electronic structure program DMol3 with a Mulliken population analysis [34]. Electronic parameters for such simulation are the Perdew–Wang (PW) local correlation density functional as well as the restricted spin polarization using the DND basis set. Geometric optimization was achieved using COMPASS force field as well as Smart minimization methods, in order to predict the activity of the inhibitor towards the metal surface, the quantum chemical parameters such as the lowest unoccupied molecular orbital (E_{LUMO}), the highest occupied molecular orbital energy (E_{HOMO}) and energy gap (ΔE) were calculated and presented in Table 5 while the geometrically optimized 3D structures, HOMO and LUMO orbitals, Fukui functions, and the total electron density are presented in Figure 6 as estimated from the simulations. The movement of electrons which leads to the adsorption of the inhibitor on the surface of the metal is due to the interaction between the frontier molecular orbitals (HOMO and LUMO orbitals) as indicated by the molecular orbital theory. HOMO energy indicates the susceptibility of the constituent toward attack by electrophile, corresponding to tendency for electron acceptance and is directly related to the ionization energy ($I = -E_{HOMO}$) while LUMO energy indicates the susceptibility of the molecule towards a nucleophilic attack and corresponds to a tendency to donate electron

and is directly related to the electron affinity ($A = -E_{LUMO}$). High values of E_{HOMO} show great ability of a molecule to release electrons to an appropriated acceptor with unfilled molecular orbital and this facilitates adsorption process, and it indicates good performance of the corrosion inhibitor. Lower values of E_{LUMO} indicate the ability of the constituent to accept electron and low values of the energy gap ($\Delta E = E_{LUMO} - E_{HOMO}$) indicate good inhibition properties. The absolute electronegativity of the molecule (χ), absolute hardness (η) and softness (δ) were estimated using the equations below:

$$\chi = \frac{I+A}{2} \quad (5)$$

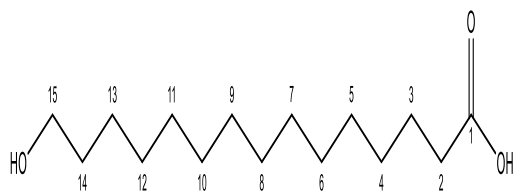
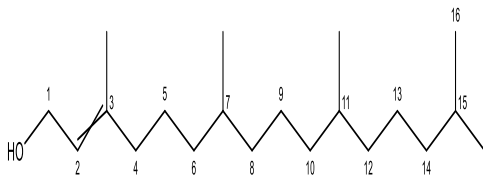
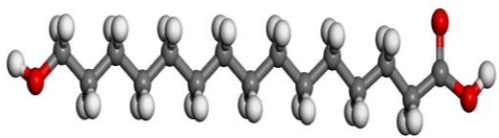
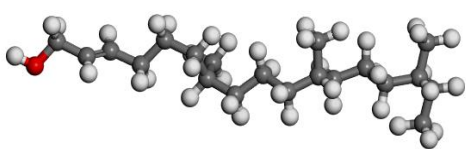
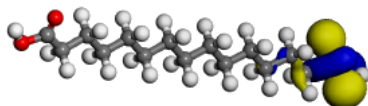
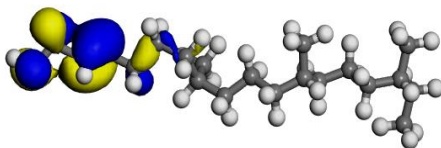
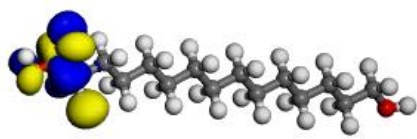
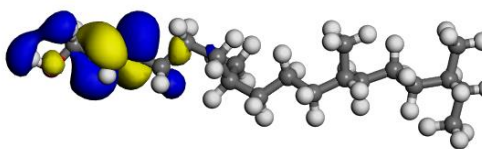
$$\eta = \frac{I-A}{2} \quad (6)$$

$$\delta = \frac{1}{\eta} \quad (7)$$

The charge transfer (ΔN) from the inhibitor to the metal surface was estimated from the equation:

$$\Delta N = \frac{\chi_m - \chi_i}{2(\eta_m + \eta_i)} \quad (8)$$

χ_m and χ_i are the absolute electronegativity of the mild steel and inhibitor respectively whereas η_m and η_i are the absolute hardness of the mild steel and the inhibitor. The values of ΔN shown in Table 5 were estimated using the theoretical values of 7 eV/mol and 0 eV/mol for the χ_m and η_m respectively [35]. The values of the simulation parameters presented in Table 5 show that the PP inhibitor functioned effectively for mild steel in the acidic media studied.

Electronic parameter	15-Hydroxypentadecanoic acid	3,7,11,15-Tetramethyl-2-hexadecen-1-ol
Molecular structure		
Optimized 3D structure		
HOMO orbital		
LUMO orbital		

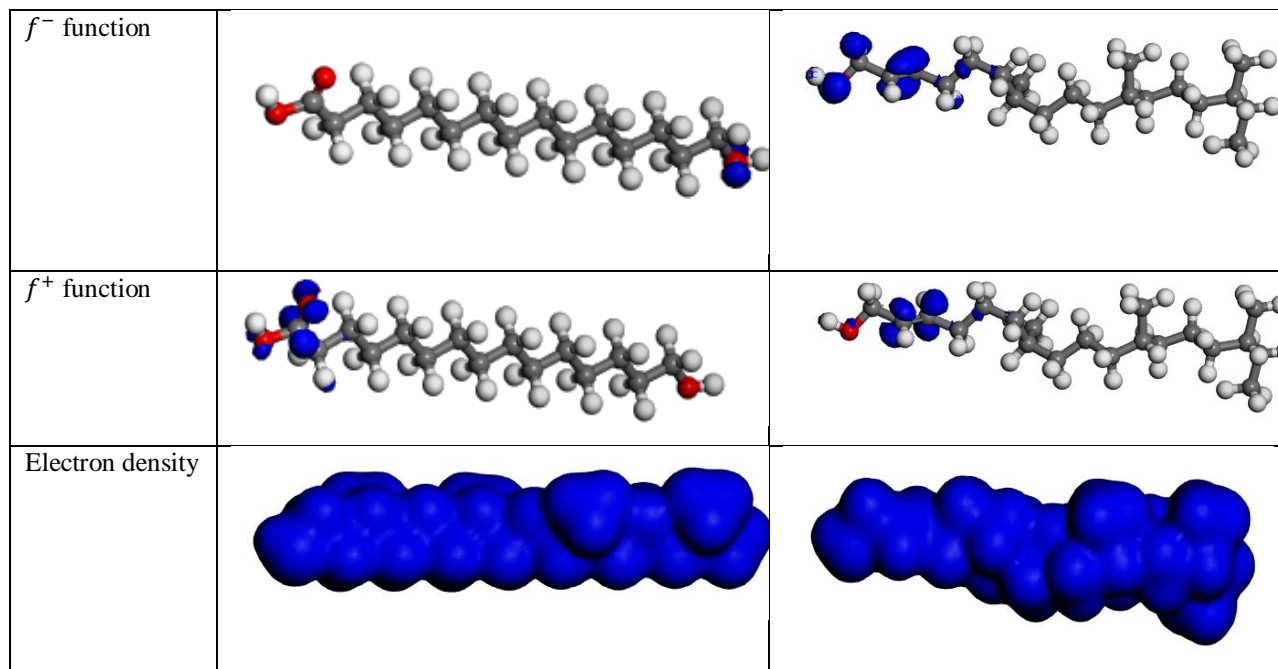

 Figure 6. Electronic properties of **15-Hydroxypentadecanoic acid** and **3,7,11,15-Tetramethyl-2-hexadecen-1-ol**

Table 5 Calculated values of some quantum chemical parameters of 15-Hydroxypentadecanoic acid and 3,7,11,15-Tetramethyl-2-hexadecen-1-ol

Parameter	15-Hydroxypentadecanoic acid	3,7,11,15-Tetramethyl-2-hexadecen-1-ol
E_{HOMO} (eV)	-0.218	-0.209
E_{LUMO} (eV)	-0.008	-0.019
ΔE (eV)	0.210	0.190
I (eV)	0.218	0.209
A (eV)	0.008	0.019
χ	0.113	0.114
η	0.105	0.100
\square	9.520	10.000
ΔN	32.790	34.430

IV. Conclusion

The results obtained from this work revealed that ethanol extract of *Pennisetum purpureum* (PP) leaves inhibited the corrosion of mild steel in both 1 M HCl and 0.5 M H₂SO₄ solutions. The gravimetric results showed that inhibition efficacy increased with concentration of the extract but decreased gradually with exposure time. The potentiodynamic polarization results showed that the extract functioned via mixed type mechanism, and this was confirmed by the electrochemical impedance spectroscopy and theoretical simulations to be accomplished by the adsorption of the extract constituents on the mild steel surface.

Conflict of interest

The author declares no conflict of interest.

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