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Corrosion inhibition performance of *hibiscus sabdariffa* leaf extract on mild steel in acidic medium

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Abstract
The performance of *Hibiscus sabdariffa (HB)* leaf extract as a corrosion inhibitor in the corrosion of mild steel in 0.5 M H_2SO_4 medium was investigated at room temperature. The experimental approaches used were gravimetric (weight loss) analysis, Potentiodynamic Polarization (PDP) technique and Fourier Transform Infra-Red Spectroscopy. The inhibitor concentrations were 5ml/L, 10ml/L, and 15ml/L for the gravimetric analysis and for the PDP, the inhibitor concentrations were 5mg/L and 15mg/L. while that of FTIR was 10ml/L. The study showed that both corrosion rate and inhibition efficiency (IE) were influenced by inhibitor concentration and exposure time. While corrosion rate increased with time of exposure, it decreased with inhibitor concentration. The gravimetric analysis gave IE values upto 95% while the PDP approach gave IE values of 96%. The Artificial Neural Network (ANN) and Multiple Line Regression (MLR) predictive models were used to analyse experimental data. They showed that inhibitor concentration affected corrosion rate more than exposure time and ANN matched experimental data more closely than MLR.

Keywords: Mild steel, *Hibiscus sabdariffa*, corrosion rate, Inhibition efficiency, Predictive models

1. Introduction

Corrosion which is the alteration or destruction of the physicochemical properties of a metal or alloy because of its reaction with its immediate environment, is a natural process. A refined metal or metal alloy is in metastable energy state (Okafor and Ebenso, 2007). The corrosion process converts the refined metal or alloy which is in a metastable energy state to a more stable (low energy) state in the form of oxides or hydroxides (called ores) in which forms the metal or alloy existed before their refining. The service environment affords the refined metal component the opportunity to recombine with O-atoms, O-H groups, S-atoms to form oxides, hydroxides, sulphides which were their original compounds when they existed as ores. The corrosion reaction facilitates the return of the metal or alloy to those original compounds (Mbah, Onah & Nnakwo, 2020)

These corrosion reactions take place when metals or alloys are exposed in the air, in solutions or buried in the ground. The process degrades the physical, chemical, electrical properties of the metals or alloys (Ameer & Ferky, 2015). The corrosion on the metal components can be localized to form a pit or crack or can be uniformly experienced on the corroding surface. It is essentially an electrochemical process, and it causes a lot of damage to assets and property, injury to people and loss of time and other resources in the industry. The cost of corrosion has been estimated by the Central Intelligence Agency (CIA), world fact book on Nigeria, to be $3.2b annually (Anyakwo, 2007). Corrosion leads to loss of man-hours, unscheduled shutdown, component maintenance and replacement costs etc. In fact, corrosion is the single largest cause of plant and equipment breakdown in the Oil and Gas industries (Akinyemi, Nwokocha & Adesanya, 2012). The World Corrosion Organization has posited that the annual cost of corrosion globally is approximately 2.2 trillion US dollars. This represents over 3% of world's Gross Domestic Product (GDP) (Kochi et al., 2002).

Corrosion control, as a result, becomes imperative and no level or number of resources and technologies invested in corrosion control is wasted. Approaches to corrosion control include but not limited to materials selection, design, cathodic protection, coating, galvanic protection, and inhibition. Inhibition approach uses inhibitors to retard either the anodic or cathodic reaction in a corrosion process or both. Inhibitors are of two major types, namely, organic and inorganic inhibitors. some of the available corrosion control technologies and approaches are expensive., Inorganic inhibitors are toxic/harmful to the environment and not bio-degradable thus causing more nuisance in the ecosystem while organic inhibitors offset these shortcomings (Okafor & Ebenso, 2007).

Nature, luckily, has provided the source/raw materials for organic inhibitors through leaves and herbs. These materials are available, non-toxic mostly, biodegradable unlike the inorganic inhibitors and other corrosion control materials and approaches. These organic inhibitors retard corrosion process by adsorbing on the surface of the metal and thus prevent direct contact between metal and corrosive environment.

The efficiency of the organic inhibitor is a measure of the coverage it offers to the surface of the metal (Chidiebere, et al., 2016). Several investigations by researchers have shown that organic inhibitors have efficiency at inhibiting corrosion. Chicken nail extract has been successfully used to inhibit mild steel corrosion in 2M H_2SO_4. Process parameters like inhibitor concentration, time of exposure and temperature. Inhibition Efficiency increased with inhibitor concentration while rate of corrosion increased with increase in time and temperature (Nwoye, Nwambu, & Emekwisia, 2023). Studies of aqueous extracts of leaves of *Delonix regia* (DR) in both 1MHCl and 0.5M H_2SO_4 showed good Inhibition Efficiency of DR as well as the fact that the extract adsorbed on the metal-environment interface

(Chidiebere, et al., 2016). Leaf extracts of HB used on mild steel corrosion proved efficient on HCl solution (Ameer & Ferky, 2015).

The present investigation evaluates the inhibition efficiency of HB in 0.5M H_2SO_4 solution using the approaches of Gravimetric analysis, PDP, FITR.

2. Materials and Methods

Three different approaches were employed in this study to evaluate the inhibition performance of HB in acidic medium. They are gravimetric (weight loss) approach, the Potentiodynamic Polarization (PDP) technique and the Fourier Transform Infra-Red (FTIR) technique. The acidic solutions are 0.5MH_2SO_4 and 1M HCl.

2.1 Gravimetric Analysis

The gravimetric (weight loss) method is the most widely method of corrosion inhibition assessment (. In this study, the mild steel coupons were mechanically press-cut into dimensions of 40mm×30mm×20mm with a hole 1.5mm diameter at the top centre of the coupon. They were cleaned with emery paper of increasing mesh size until mirror surface and kept in a desicator. The leaves of Hibiscus Sabdariffa were sun-dried and a mass of 200g was soaked in one litre of ethanol for 48hrs. The filtrate (containing inhibitor and ethanol) was decanted into an open conical flask and the residue, after squeezing out the liquid, was discarded. The filtrate was allowed open with slight heating for ethanol evaporate at 78°c. The 0.5m H_2SO_4 was prepared as well as the 1M HCl.

Corrosion rate, expressed in mm/yr, was determined by

$CR = kw/\varrho At$ _______ (1)

Where k = constant (8.76 x 10^4 to convert rate to mm/yr)
W= weight loss (final weight loss – initial weight loss) (W_0-W_1)
ϱ = density (7.85 g/cm^3 for mild steel)
t = time of exposure
Surface Area $A = 2(LB+LT+BT) - 2(\pi d^2/4) + \pi dT$ (2)
Where L= length
B= breadth
T= thickness
d= diameter

The weighing was done with the 4-digit HAUS electronic weighing machine.
The inhibition Efficiency (IE) expressed in percentages was determined using the formula:

$$IE\ (\%) = \frac{(CRu - CRi)}{CRu} \times 100\% \quad \times 100/1 \qquad (3)$$

Where CR_u = corrosion rate of uninhibited reaction
CR_i = corrosion rate of inhibited reaction

2.1 The Potentiodynamic Polarization (PDP) measurement

The mild steel used in the PDP technique under this study were cut into 2cm×2cm dimensions. The coupon after cleaning to mirror surface, was covered leaving only 1cm^2 of its surface area exposed to the corrodent 0.5M H_2SO_4 and 1M HCl. The experiment was carried out with the three-electrode corrosion cell using a 263 Potentiostat/Galvanostat electrochemical workstation. A graphite rod was used as counter electrode (SCE) was used as the reference electrode. The experiment was performed in aerated and unstirred solutions at the end of 1800s, 30^0 ±+- 1^0C, in the potential range of ±+- 250 mV versus corrosion potential at a scan rale of 0.333 mV/s. The test was run in duplicate to verify reproducibility of the data and with freshly prepared solution. The corrosion of the inhibitor were 5mls, 10mls and 15mls of the inhibitor per litre of acid solution. The machine gave plots of potential against the logarithm of current and also gave IE in percentage.

2.2 The Fourier Transform Infra-Red (FTIR)

The coupons were prepared as they were in gravimetric measurements. The degreased and cleaned coupons were immersed in freshly prepared corrosive environments of 0.5M H_2SO_4 and 1M HCl into which the inhibitor has been put. The coupons were allowed to stay immersed in the solutions for 72hrs after which they were removed and air dried. The surfaces of the coupons were then scrapped, and the scrape-off particles were used for the FTIR analysis.

3.0 Result and Discussion

Table 1. Corrosion rate (mm/yr) and inhibition efficiency (IE) of HB in 0.5M H_2SO_4

Time	Weight loss (g)				Corrosion rate (mm/yr)				I E (%)		
(hrs)	blank	5mls	10mls	15mls	blank	5mls	10mls	15mls	5mls	10mls	15mls
24	0.541	.0503	.0488	.0407	9.3651	.8707	.8448	.7045	91	91	93
48	1.1912	.0998	.0799	.0701	10.319	.8638	.6916	.6067	92	93	94
72	1.9152	.1785	.1358	.1186	11.0512	1.0300	.7836	.6844	91	93	94
96	2.5323	.2548	.1881	.1458	10.959	1.1027	.8140	.6440	90	93	94
120	3.1428	.328	.2178	.1792	10.8809	1.1356	.7541	.6214	90	93	94
144	3.9218	.4669	.2456	.2182	11.3149	1.3471	.7086	.6295	88	94	94
168	4.7335	.5803	.2979	.2463	11.7058	1.4351	.7367	.6095	88	94	95

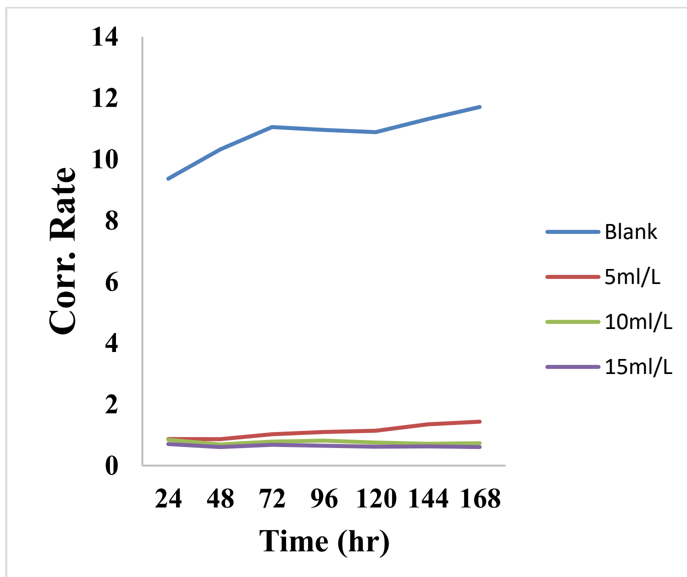


Figure 1: Plot of corrosion rate (mm/yr) vs time

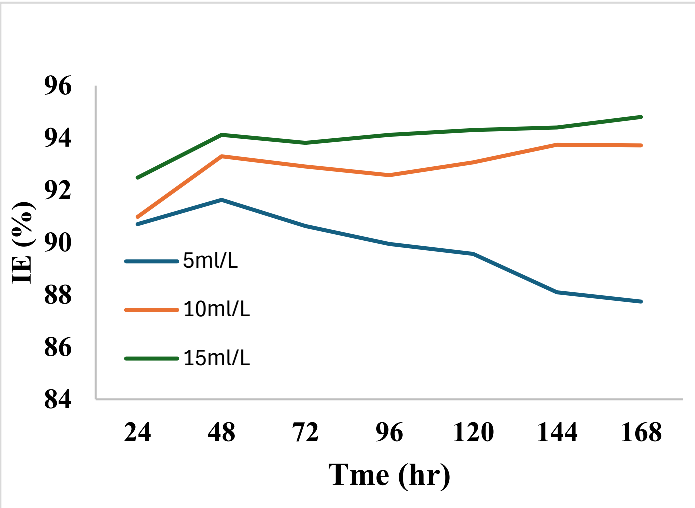


Figure 2: Plot of inhibition efficiency (IE) vs time

Table 2: PDP results of HB leaf extract in 0.5M H_2SO_4

Concentration	Icorr ($\mu A/cm^2$)	Ecorr (mV vs SCE)	IE (%)
Blank	164	-484.8	
5 mg/L	18.2	-483.2	88.9
15 mg/L	6.5	-478.5	96

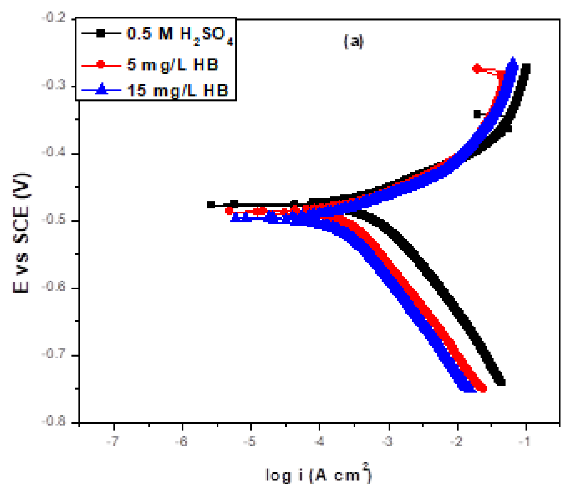
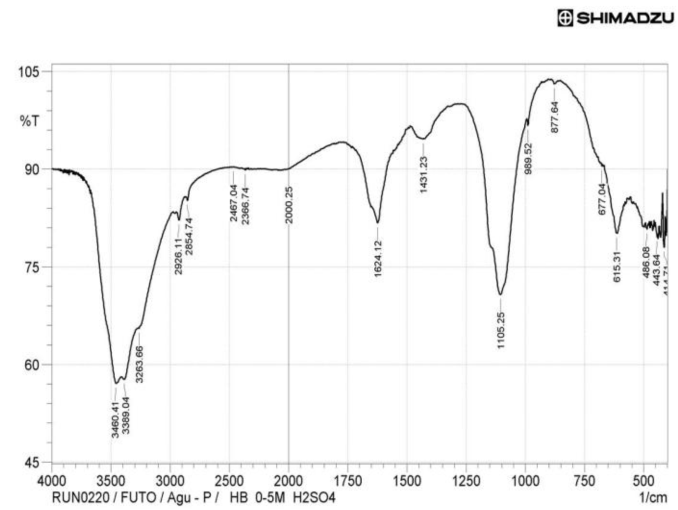


Figure 3: The PDP plot of HB leaf extract in 0.5M H_2SO_4



	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	414.71	77.964	6.579	420.5	408.92	1.093	0.248
2	443.64	79.577	0.834	451.36	441.71	0.912	0.026
3	486.08	80.799	0.842	491.86	482.22	0.876	0.027
4	615.31	80.142	6.94	667.39	580.59	6.536	1.526
5	677.04	90.46	0.402	680.89	673.18	0.329	0.008
6	877.64	103.095	0.611	889.21	862.21	-0.393	0.032
7	989.52	96.716	1.38	993.37	931.65	-0.236	-0.09
8	1105.25	70.73	27.955	1259.56	993.37	17.254	15.926
9	1431.23	94.64	0.073	1435.09	1427.37	0.183	0.001
10	1624.12	81.733	4.92	1649.19	1564.32	5.352	0.79
11	2000.25	90	0.093	2004.11	1915.38	3.652	0.031
12	2366.74	89.873	0.24	2372.52	2362.88	0.442	0.006
13	2467.04	90.309	0.026	2470.9	2463.18	0.341	0
14	2854.74	85.192	1.025	2866.32	2810.38	3.5	0.066
15	2926.11	82.2	1.978	2949.26	2877.89	5.451	0.26
16	3263.66	65.56	0.293	3267.52	2976.26	36.041	-2.069
17	3389.04	57.685	1.351	3406.4	3269.45	28.678	0.425
18	3460.41	57.076	6.19	3666.8	3414.12	42.98	5.001

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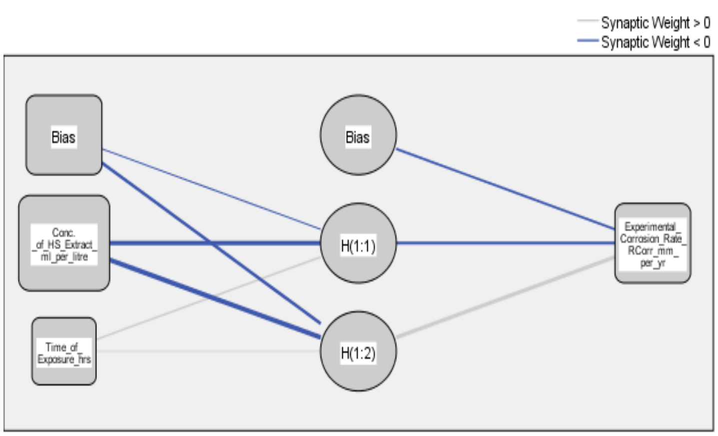
Apodization;

Figure 4 and Table 3: FTIR Results of HB in 0.5 M H_2SO_4

Table 4: ANN and MLR predictions of the experimental corrosion rates corrosion inhibition of mild steel in 0.5 M sulphuric acid using HB extract

Case	Time of Exposure (hrs)	Conc. of H_2SO_4 (M)	Con. of HS Extract (ml/L)	Experimental orrosion rate, R_{Corr} (mm/yr) (Control)	Prediction of R_{Corr}			
					By ANN	Error in prediction	By MLR	Error in prediction
1	24	0.50	0.00	9.4909	.4618	-.03	79822	-.69
2	48	0.50	0.00	10.4576	.8156	-.64	96558	-1.49
3	72	0.50	0.00	11.1997	.1448	-1.05	13294	-2.07
4	96	0.50	0.00	11.1062	.4489	-.66	30030	-1.81
5	120	0.50	0.00	11.0270	.7279	-.30	46766	-1.56
6	144	0.50	0.00	11.4670	.9825	-.48	63502	-1.83
7	168	0.50	0.00	11.8670	1.2133	-.65	80238	-2.06
8	192	0.50	0.00	12.8641	1.4218	-1.44	96974	-2.89
9	24	0.50	5.00	0.8824	8243	-.06	10938	4.23
10	48	0.50	5.00	0.8754	8647	-.01	27674	4.40
11	72	0.50	5.00	1.0438	9307	-.11	44410	4.40
12	96	0.50	5.00	1.1175	.0280	-.09	61146	4.49
13	120	0.50	5.00	1.1508	.1620	.01	77882	4.63
14	144	0.50	5.00	1.3652	.3370	-.03	94617	4.58

15	168	0.50	5.00	1.4543	.5560	.10	11353	4.66
16	192	0.50	5.00	1.5375	.8208	.28	28089	4.74
17	24	0.50	0.00	9.2717	.4618	.19	79822	-.47
18	48	0.50	0.00	10.3988	.8156	-.58	96558	-1.43
19	72	0.50	0.00	11.1061	).1448	-.96	13294	-1.97
20	96	0.50	0.00	10.5778	).4489	-.13	30030	-1.28
21	120	0.50	0.00	10.8414	).7279	-.11	46766	-1.37
22	144	0.50	0.00	11.1444	).9825	-.16	63502	-1.51
23	168	0.50	0.00	11.4087	1.2133	-.20	80238	-1.61
24	192	0.50	0.00	11.7953	1.4218	-.37	96974	-1.83
25	24	0.50	10.00	0.8561	6765	-.18	42053	.56
26	48	0.50	10.00	0.7009	6618	-.04	58789	.89
27	72	0.50	10.00	0.7941	6433	-.15	75525	.96
28	96	0.50	10.00	0.825	6204	-.20	92261	1.10
29	120	0.50	10.00	0.7642	5921	-.17	08997	1.33
30	144	0.50	10.00	0.7181	5576	-.16	25733	1.54
31	168	0.50	10.00	0.7466	5160	-.23	42469	1.68
32	192	0.50	10.00	0.7329	4661	-.27	59205	1.86
33	24	0.50	0.00	8.5629	.4618	.90	79822	.24
34	48	0.50	0.00	8.6725	.8156	1.14	96558	.29
35	72	0.50	0.00	9.2833	).1448	.86	13294	-.15
36	96	0.50	0.00	9.2927	).4489	1.16	30030	.01
37	120	0.50	0.00	10.1415	).7279	.59	46766	-.67
38	144	0.50	0.00	10.1795	).9825	.80	63502	-.54
39	168	0.50	0.00	10.1724	1.2133	1.04	80238	-.37
40	192	0.50	0.00	10.2806	1.4218	1.14	96974	-.31
41	24	0.50	15.00	0.7140	7170	.00	.26831	-2.98
42	48	0.50	15.00	0.6149	7163	.10	.10095	-2.72
43	72	0.50	15.00	0.6935	7154	.02	.93359	-2.63
44	96	0.50	15.00	0.6526	7142	.06	.76623	-2.42
45	120	0.50	15.00	0.6288	7128	.08	.59887	-2.23
46	144	0.50	15.00	0.6380	7110	.07	.43151	-2.07
47	168	0.50	15.00	0.6173	7088	.09	.26415	-1.88
48	192	0.50	15.00	0.6335	7060	.07	.09679	-1.73



Hidden layer activation function: Sigmoid

Output layer activation function: Identity

Table 5: Independent Variable Importance and Parameter Estimates of the prediction of corrosion inhibition of mild steel in 0.5 M H_2SO_4 medium by the HB Extract using ANN

Independent Variable Importance

	Importance
Conc._of_HS_Extract_ml_per_litre	0.896
Time_of_Exposure_hrs	0.104

Parameter Estimates

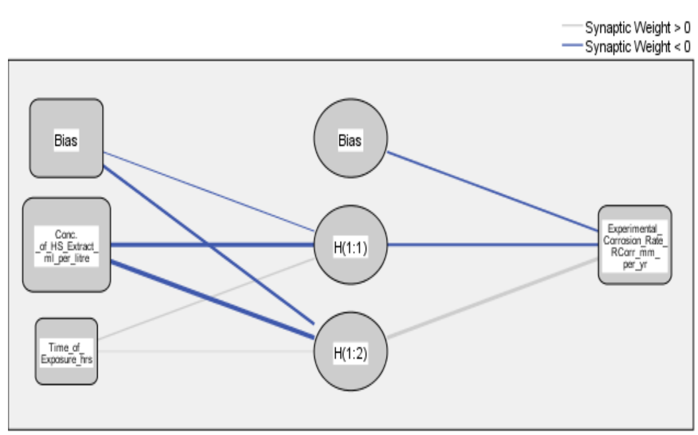
Predictor		Predicted: Hidden Layer 1 H (1:1)	Predicted: Hidden Layer 1 H (1:2)	Predicted: Output Layer Experimental_Corrosion_Rate_RCorr_mm_per_yr
Input Layer	(Bias)	-.369	-2.317	
	Conc._of_HS_Extract_ml_per_litre	-3.911	-3.959	
	Time_of_Exposure_hrs	0.461	0.321	
Hidden Layer 1	(Bias)			-0.813
	H (1:1)			-0.882
	H (1:2)			3.395

Predictive equation by MLR

Predicted-RCorr$_{HS \text{ in } H2SO4 \text{ by MLR}}$ = 8.631 + 0.007 (time of exposure) – 0.738 (conc. of HS leaf-extract)

Table 6: ANN and MLR predictions of the experimental corrosion rates corrosion inhibition of mild steel in 0.5 M sulphuric acid using HS extract

	Time of Exposure (hrs)	Conc. H_2SO_4 (M)	Conc. HS Extract (/L)	Experimental Corrosion rate, R_{Corr} (mm/yr) (Control)	Prediction of R_{Corr} By ANN	Error prediction	By MLR	Error in prediction
1	24	0.50	0.00	9.4909	9.4618	-.03	8.79822	-.69
2	48	0.50	0.00	10.4576	9.8156	-.64	8.96558	-1.49
3	72	0.50	0.00	11.1997	10.1448	-1.05	9.13294	-2.07
4	96	0.50	0.00	11.1062	10.4489	-.66	9.30030	-1.81
5	120	0.50	0.00	11.0270	10.7279	-.30	9.46766	-1.56
6	144	0.50	0.00	11.4670	10.9825	-.48	9.63502	-1.83
7	168	0.50	0.00	11.8670	11.2133	-.65	9.80238	-2.06
8	192	0.50	0.00	12.8641	11.4218	-1.44	9.96974	-2.89
9	24	0.50	5.00	0.8824	.8243	-.06	5.10938	4.23
10	48	0.50	5.00	0.8754	.8647	-.01	5.27674	4.40
11	72	0.50	5.00	1.0438	.9307	-.11	5.44410	4.40
12	96	0.50	5.00	1.1175	1.0280	-.09	5.61146	4.49
13	120	0.50	5.00	1.1508	1.1620	.01	5.77882	4.63
14	144	0.50	5.00	1.3652	1.3370	-.03	5.94617	4.58
15	168	0.50	5.00	1.4543	1.5560	.10	6.11353	4.66
16	192	0.50	5.00	1.5375	1.8208	.28	6.28089	4.74
17	24	0.50	0.00	9.2717	9.4618	.19	8.79822	-.47
18	48	0.50	0.00	10.3988	9.8156	-.58	8.96558	-1.43
19	72	0.50	0.00	11.1061	10.1448	-.96	9.13294	-1.97
20	96	0.50	0.00	10.5778	10.4489	-.13	9.30030	-1.28
21	120	0.50	0.00	10.8414	10.7279	-.11	9.46766	-1.37
22	144	0.50	0.00	11.1444	10.9825	-.16	9.63502	-1.51
23	168	0.50	0.00	11.4087	11.2133	-.20	9.80238	-1.61
24	192	0.50	0.00	11.7953	11.4218	-.37	9.96974	-1.83
25	24	0.50	0.00	0.8561	.6765	-.18	1.42053	.56
26	48	0.50	0.00	0.7009	.6618	-.04	1.58789	.89
27	72	0.50	0.00	0.7941	.6433	-.15	1.75525	.96
28	96	0.50	0.00	0.825	.6204	-.20	1.92261	1.10
29	120	0.50	0.00	0.7642	.5921	-.17	2.08997	1.33
30	144	0.50	0.00	0.7181	.5576	-.16	2.25733	1.54
31	168	0.50	0.00	0.7466	.5160	-.23	2.42469	1.68
32	192	0.50	0.00	0.7329	.4661	-.27	2.59205	1.86
33	24	0.50	0.00	8.5629	9.4618	.90	8.79822	.24
34	48	0.50	0.00	8.6725	9.8156	1.14	8.96558	.29
35	72	0.50	0.00	9.2833	10.1448	.86	9.13294	-.15
36	96	0.50	0.00	9.2927	10.4489	1.16	9.30030	.01
37	120	0.50	0.00	10.1415	10.7279	.59	9.46766	-.67
38	144	0.50	0.00	10.1795	10.9825	.80	9.63502	-.54
39	168	0.50	0.00	10.1724	11.2133	1.04	9.80238	-.37
40	192	0.50	0.00	10.2806	11.4218	1.14	9.96974	-.31
41	24	0.50	5.00	0.7140	.7170	.00	-2.26831	-2.98
42	48	0.50	5.00	0.6149	.7163	.10	-2.10095	-2.72
43	72	0.50	5.00	0.6935	.7154	.02	-1.93359	-2.63
44	96	0.50	5.00	0.6526	.7142	.06	-1.76623	-2.42
45	120	0.50	5.00	0.6288	.7128	.08	-1.59887	-2.23
46	144	0.50	5.00	0.6380	.7110	.07	-1.43151	-2.07
47	168	0.50	5.00	0.6173	.7088	.09	-1.26415	-1.88
48	192	0.50	5.00	0.6335	.7060	.07	-1.09679	-1.73



Hidden layer activation function: Sigmoid

Output layer activation function: Identity

Predictive equation by MLR

Predicted-RCorr$_{\mathbf{HS}\ \text{in H2SO4 by MLR}}$ = 8.631 + 0.007 (time of exposure) – 0.738 (conc. of HS leaf-extract)

4.0 Discussion

From the gravimetric analysis result, corrosion rate increased with time for both inhibited and uninhibited reactions. For the inhibited reactions the inhibitor covered the surface of the mild steel coupons by adsorption creating a film/barrier between the metal surface and the acidic environment. This accounts for the reduction in the corrosion rate of inhibited reaction compared to the blank. For the inhibited reaction, corrosions rate decreased with increase in inhibitor concentration at any given time while corrosion rate increased with time for each inhibitor concentration. This is attributed to the fact that the film created by the inhibitor on the surface of the metal gets weak with time and the corrodent penetrates to attack the metal.

Inhibitor Efficiency (IE) decreased with increase in time of exposure with 5mls/L inhibitor concentration but increased with increase in time of exposure for both 10mls/L and 15mls/L of inhibitor concentration respectively. For any given time of exposure, IE increased with increase in inhibitor concentration. This is because higher inhibitor concentration offers more coverage of the mild steel surface preventing it from the attack of the corrodent. In 2022, (Shahen, Abdel-Karim & Gaber), using 1M HNO_3 and Cu-Zn alloy, concluded that *Hibiscus Sabdarifa* (HB) is an excellent inhibitor and that its IE increased with increase in the inhibitor concentration. Also, Amabua et al, (in 2024), concluded that *Hibiscus rose-sinensis* gave a higher IE value than commercial (inorganic) inhibitors in all the concentrations used.

For the Potentiodynamic Polarization (PDP) approach, HB gave IE of 96% at 15ml/L of inhibitor concentration. Here, IE also increased with inhibitor concentration. The PDP also shows HB as a mixed inhibitor, retarding both anodic and cathodic reactions Hibiscus sabdariffa as an inhibitor using 0.1M HCl and X-ray photoelectron spectroscopy approach, was confirmed to be a mixed inhibitor (Nguyen, Xuan-To, Gervasi, Paint, Gonon & Olivier, 2018).

5.0 Conclusion

Hibiscus sabdariffa leaf extract is an excellent corrosion mixedinhibitor for mild steel, offering an IE value of 96%. The IE is influenced by both inhibitor concentration and exposure time. It can be employed as an organic inhibitor in the mitigation of corrosion in oil pipelines. The concentration of inhibitor influenced the corrosion rate more than the exposure time. ANN predictive model agreed more closely with experimental data than MLR.

Declaration Statement

The authors agreed with total interest to submit the manuscript entitled, 'Corrosion inhibition performance of hibiscus sabdariffa leaf extract on mild steel in acidic medium' for publication in your reputable Institution without conflict of interest be it design and implementation, respect towards society, resources and research output and conduct without deceptive acts.

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Conflict of Interest

The authors declare no conflict of interest.

Author Contribution

Paulinus Chukwudi Agu: Writing – original draft, Methodology. C. N. Anyakwo, A. I. Ndukwe and C. Onuoha: Supervision. N. A. Nnodum: Formal analysis.

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