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Comparative study of mild steel corrosion inhibition performance of *hibiscus sabdariffa* leaf extract in different acid media

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Abstract

A comparative study of mild steel corrosion inhibition performance of *Hibiscus sabdariffa* (HB) leaf extract on mild steel in the acidic media of 0.5M H₂SO₄ and 1M HCl was conducted over a duration of 168 hours (7 days). Gravimetric Analysis (GA) and Potentio-Dynamic Polarization (PDP) were employed as the experimental techniques. The Prediction Models (PM) of Artificial Neural Network (ANN) and Multiple Linear Regression (MLR) were used to predict the experimental data. For the GA technique, inhibitor concentrations of 5ml, 10ml, 15ml per litre (mL/L) of corrodent were used. HB leaf extract of concentration 5mL/L of corrodent yielded Inhibition Efficiency (IE) of 92% in 0.5M H₂SO₄ and 93% in 1M HCl. In 10ml/l concentration, IE was 94% in 0.5M H₂SO₄ and 93% in 1MHCl while with 15ml/l concentration, IE of 95% in 0.5M H₂SO₄ and 94% in 1M HCl were recorded. The PDP approach showed IE of 77.9% and 82.8% in 1M HCl with 5 mg/L and 15mg/L respectively while IE 88.9% and 96% were achieved with 0.5M H₂SO₄ in 5mg/L and 15mg/L respectively. The PMs confirmed that inhibitor concentration affected IE more than exposure time and the ANN model agreed more closely with the experimental data than the MLR model and therefore can be used to model the experimental data, namely, corrosion rate and IE, in this study.

Keywords: Mild steel; Corrosion inhibition; *Hibiscus sabdariffa*; Inhibition efficiency; Prediction models

1. Introduction

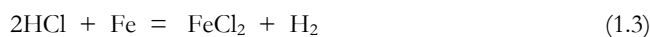
Mild steel, due to its relative cheapness and appreciable mechanical strength, enjoys versatile structural applications in the oil and gas industries (Anyakwo, 2007). Most of the environments where these mild steel structures are used are corrosive hence the imperativeness of corrosion mitigation processes to prolong the economic life of the structures. Metals corrode by oxidation process, at the anode, according to the equation below.



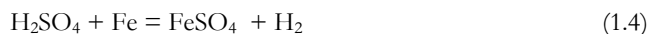
For mild steel, the equation becomes



When iron (mild steel) corrodes in acid solutions which have high concentrations of H^+ ions, the H^+ ions are reduced in the cathodic reaction with evolution of hydrogen gas according to equation (1.3)



In sulphuric acid, the reaction is as shown below



The reactions described in equations (1.3) and (1.4) above take place at the cathode.

Corrosion can be mitigated through several approaches including the use of inhibitors. Inhibitors can be organic or inorganic, anodic or cathodic. Inhibitors alter the rate of anodic reaction or that of cathodic reaction or both. While anodic inhibitors retard anodic (corrosion) reaction and cathodic inhibitors retard cathodic reaction, mixed inhibitors retard the reaction at both electrodes (Ndukwe & Anyakwo, 2017)

Inorganic (synthetic) inhibitors, some of which have proved efficient, are expensive, non-biodegradable and inimical to health and environment. Organic (green) inhibitors on the other hand, are biodegradable, eco-friendly, and cheaper to acquire hence the rising awareness and importance of green inhibitors (Gunavathy & Murugaavel, 2012).

The corrosion of mild steel in the media under study was affected mainly by such variables as exposure time in environment and inhibitor concentration. Any prediction models that accommodate these variables are relevant in predicting corrosion rate and IE.

2.0 Materials and Methods

Two different techniques were employed in this study to evaluate the inhibition performance of HB in acidic medium. They are GA (weight loss) and PDP techniques. The acidic solutions are 0.5M H_2SO_4 and 1M HCl.

2.1 Gravimetric Analysis

The gravimetric (weight loss) method is the most widely method of corrosion inhibition assessment (Musa et al., 2010). 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-like surface was achieved and kept in a desiccator. The leaves of *Hibiscus Sabdariffa* were sun-dried and a mass of 200g of ground HB leaf powder 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 to evaporate at 78°C. The 0.5M H_2SO_4 solution was prepared as well as the 1M HCl.

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

$$CR = (k \times w) / (\rho \times A \times t) \quad (2.1)$$

Where k = constant (8.76×10^4 to convert rate to mm/yr)

W= weight loss (final weight loss – initial weight loss)

ρ = density (7.85 g/cm³ for mild steel)

t = time of exposure

$$\text{Surface Area } A = 2(LB+LT+BT) - 2(\pi d^2/4) + \pi dT \quad (2.2)$$

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:

$$IE (\%) = [(CR_u - CR_i) / CR_u] \times 100 \quad (2.3)$$

Where CR_u = corrosion rate of uninhibited reaction

CR_i = corrosion rate of inhibited reaction

2.2 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-like surface, was covered leaving only 1cm² of its surface area exposed to the corrodent 0.5M H₂SO₄ 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 while the Saturated Calomel Electrode (SCE) was used as the reference electrode. The experiment was performed in aerated and unstirred solutions at the end of 1800s, 30⁰ ±1⁰C, in the potential range of ± 250 mV versus corrosion potential at a scan rate 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 was 5mg/L, and 15mg/L of the inhibitor per litre of acid solution, as minimum and maximum values respectively. The machine gave plots of potential against the logarithm of current and the IE in percentage.

3.0 Result and Discussion

Table 1 Corrosion rate (mm/yr) and inhibition efficiency (IE) of HB in 0.5M H₂SO₄

Time (hrs)	Corrosion rate (mm/yr)				IE (%)		
	Blank	5mls	10mls	15mls	5mls	10mls	15mls
24	9.3651	0.8707	0.8448	0.7045	91	91	93
48	10.319	0.8638	0.6916	0.6067	92	93	94
72	11.0512	1.0300	0.7836	0.6844	91	93	94
96	10.959	1.1027	0.8140	0.6440	90	93	94
120	10.8809	1.1356	0.7541	0.6214	90	93	94
144	11.3149	1.3471	0.7086	0.6295	88	94	94
168	11.7058	1.4351	0.7367	0.6095	88	94	95

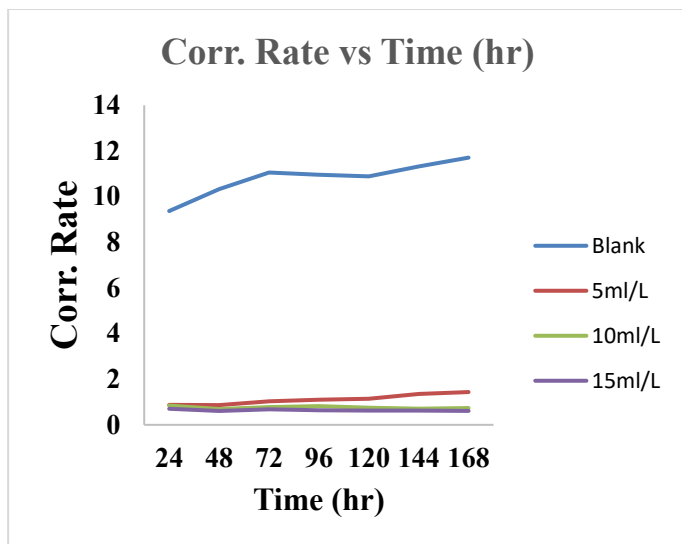


Figure 1(a) Plot of corrosion rate Vs time for HB in 0.5MH₂SO₄

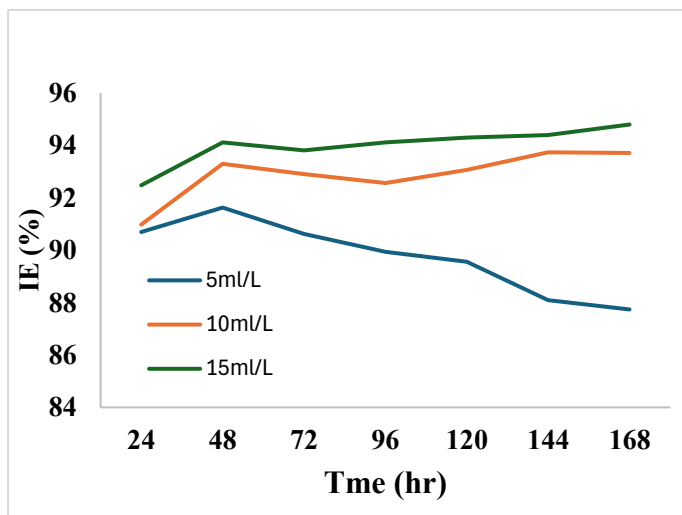


Figure 1 (b) Plot of (IE) Vs time for HB in 0.5M H₂SO₄

Table 2 Corrosion rate (mm/yr) and Inhibition Efficiency (IE) for HB in 1MHCl

Time (hrs)	Corr. Rate (mm/yr)				IE (%)		
	Blank	5mls	10mls	15mls	5mls	10mls	15mls
24	3.3843	0.2908	0.2770	0.2614	91	92	92
48	3.7218	0.2510	0.2441	0.2328	93	93	74
72	3.3537	0.4714	0.4530	0.3376	86	86	90
96	3.1601	0.4509	0.4293	0.3536	86	86	89
120	3.2524	0.4546	0.4321	0.4030	86	87	87
144	4.0842	0.4694	0.4201	0.4056	89	90	90
168	4.3408	0.4916	0.4404	0.4231	89	90	90

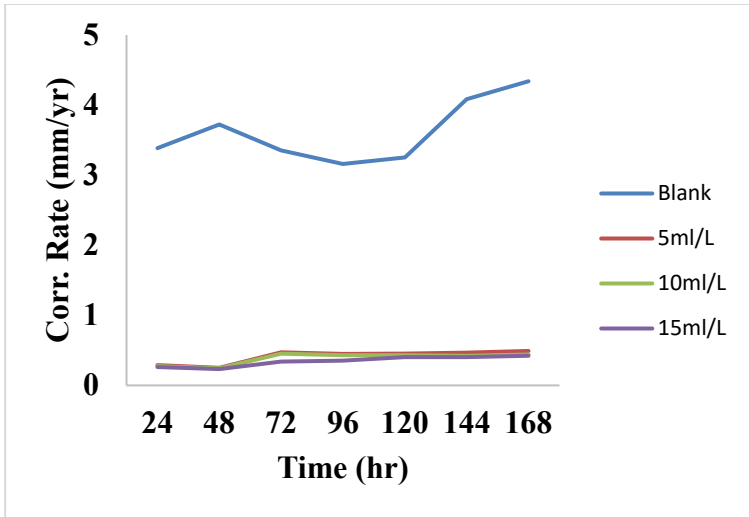


Figure 2 (a) Plot of Corrosion rate Vs time for HB in 1M HCl

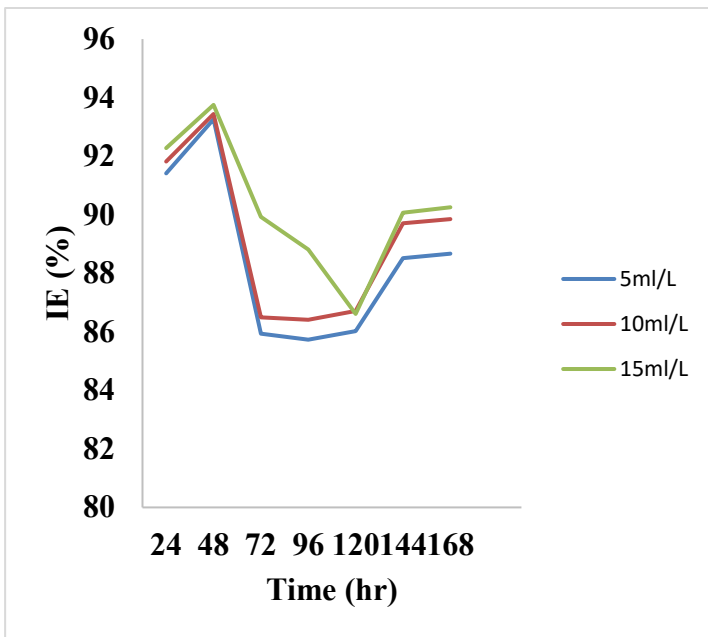


Figure 2 (b) Plot of IE Vs time for HB in 1M HCl

Table 3: Potentiodynamic polarization Parameters for mild steel in 1 M HCl and 0.5 M H₂SO₄ in the Absence and Presence of HB.

System	I_{corr} ($\mu\text{A}/\text{cm}^2$)	E_{corr} (mV vs SCE)	IE%
1 M HCl	183	-483.4	
5 mg/L HB	40.5	-492.7	77.9
15 mg/L HB	31.4	-476.4	82.8
0.5 M H₂SO₄	164	-484.8	
5 mg/L HB	18.2	-483.2	88.9
15 mg/L HB	6.5	-478.5	96

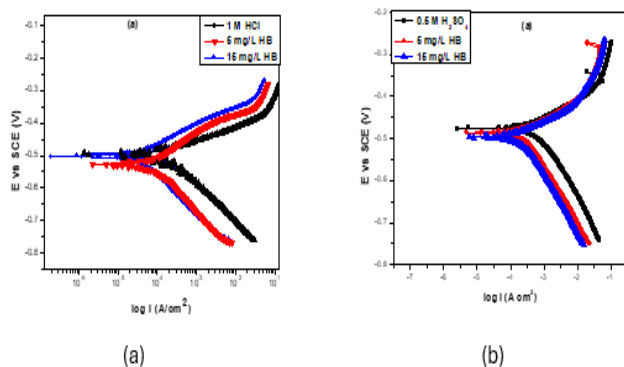


Figure 3 The PDP plot of HB leaf extract in (a) 1M HCl and (b) 0.5M H₂SO₄

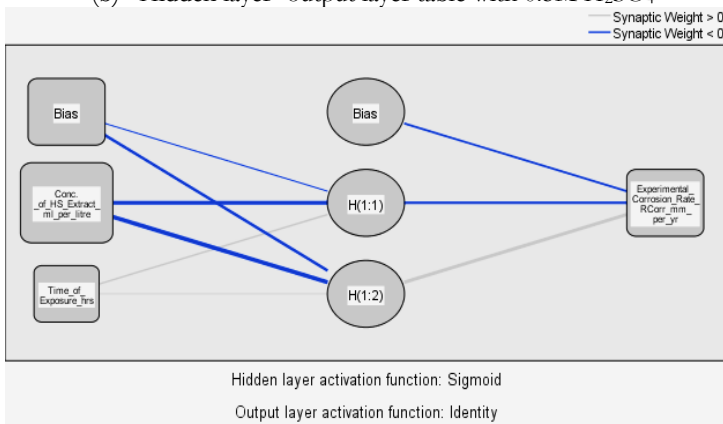
Table 4: ANN and MLR predictions of the experimental corrosion rates in corrosion inhibition of mild steel in 0.5 M H₂SO₄ using HB extract

(a) Prediction values of ANN and MLR in 0.5M H₂SO₄

Case	Time of Exposure (hrs)	Conc. of H ₂ SO ₄ (M)	Conc. of HB Extract (ml/L)	Experimental Corrosion 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	9.4618	-.03	9822	-.69
2	48	0.50	0.00	10.4576	9.8156	-.64	6558	-1.49
3	72	0.50	0.00	11.1997	10.1448	-1.05	3294	-2.07
4	96	0.50	0.00	11.1062	10.4489	-.66	0030	-1.81
5	120	0.50	0.00	11.0270	10.7279	-.30	6766	-1.56
6	144	0.50	0.00	11.4670	10.9825	-.48	3502	-1.83
7	168	0.50	0.00	11.8670	11.2133	-.65	0238	-2.06
8	192	0.50	0.00	12.8641	11.4218	-1.44	6974	-2.89
9	24	0.50	5.00	0.8824	.8243	-.06	0938	4.23
10	48	0.50	5.00	0.8754	.8647	-.01	7674	4.40
11	72	0.50	5.00	1.0438	.9307	-.11	4410	4.40
12	96	0.50	5.00	1.1175	1.0280	-.09	51146	4.49
13	120	0.50	5.00	1.1508	1.1620	.01	7882	4.63
14	144	0.50	5.00	1.3652	1.3370	-.03	4617	4.58
15	168	0.50	5.00	1.4543	1.5560	.10	1353	4.66
16	192	0.50	5.00	1.5375	1.8208	.28	8089	4.74
17	24	0.50	0.00	9.2717	9.4618	.19	9822	-.47
18	48	0.50	0.00	10.3988	9.8156	-.58	6558	-1.43
19	72	0.50	0.00	11.1061	10.1448	-.96	3294	-1.97
20	96	0.50	0.00	10.5778	10.4489	-.13	0030	-1.28
21	120	0.50	0.00	10.8414	10.7279	-.11	6766	-1.37
22	144	0.50	0.00	11.1444	10.9825	-.16	3502	-1.51
23	168	0.50	0.00	11.4087	11.2133	-.20	0238	-1.61
24	192	0.50	0.00	11.7953	11.4218	-.37	6974	-1.83

25	24	0.50	10.00	0.8561	.6765		-.18	2053	.56
26	48	0.50	10.00	0.7009	.6618		-.04	18789	.89
27	72	0.50	10.00	0.7941	.6433		-.15	5525	.96
28	96	0.50	10.00	0.825	.6204		-.20	22261	1.10
29	120	0.50	10.00	0.7642	.5921		-.17	8997	1.33
30	144	0.50	10.00	0.7181	.5576		-.16	5733	1.54
31	168	0.50	10.00	0.7466	.5160		-.23	2469	1.68
32	192	0.50	10.00	0.7329	.4661		-.27	9205	1.86
33	24	0.50	0.00	8.5629	9.4618		.90	9822	.24
34	48	0.50	0.00	8.6725	9.8156		1.14	6558	.29
35	72	0.50	0.00	9.2833	10.1448		.86	3294	-.15
36	96	0.50	0.00	9.2927	10.4489		1.16	4030	.01
37	120	0.50	0.00	10.1415	10.7279		.59	6766	-.67
38	144	0.50	0.00	10.1795	10.9825		.80	3502	-.54
39	168	0.50	0.00	10.1724	11.2133		1.04	40238	-.37
40	192	0.50	0.00	10.2806	11.4218		1.14	6974	-.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	143151	-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	409679	-1.73

(b) Hidden layer -output layer table with 0.5M H₂SO₄



© Independent Variable table

Independent Variable Importance and Parameter Estimates of the prediction of corrosion inhibition of mild steel in 0.5 M H₂SO₄ medium by the HS Extract using ANN

Independent Variable Importance		Importance		
Conc. of HB Extract ml per litre		0.896		
Time of Exposure hrs		0.104		
Parameter Estimates				
Predictor		Predicted		Output Layer
		Hidden Layer 1	H (1:2)	
		H (1:1)		Experimental Co rosion Rate RC orr mm per yr
Input Layer	(Bias)	-3.69	-2.317	
	Conc. of HB 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} in H₂SO₄ by MLR = 8.631 + 0.007 (time of exposure) – 0.738 (conc. of HS leaf-extract)

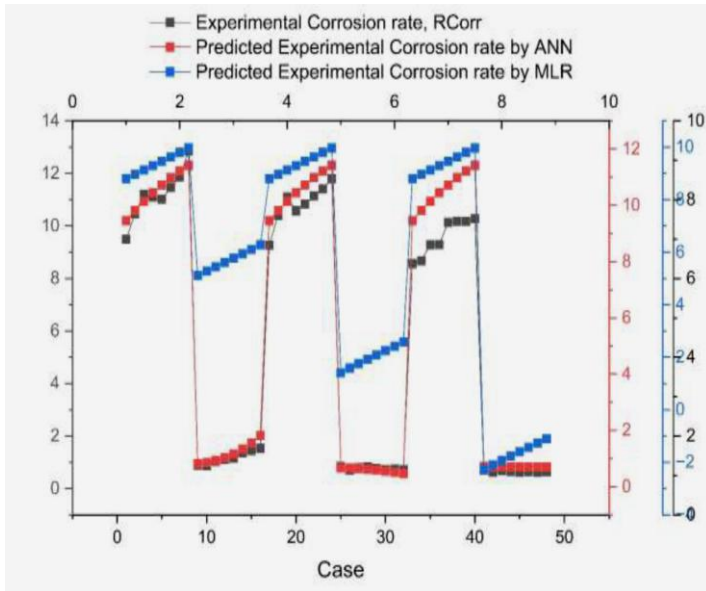


Figure 4: Comparative Analysis of Multiple Linear Regression (MLR) and Artificial Neural Network (ANN) Models for Corrosion Inhibition Effectiveness of leaves of (HB) Extract on Mild Steel in 0.5 M H₂SO₄ Solution

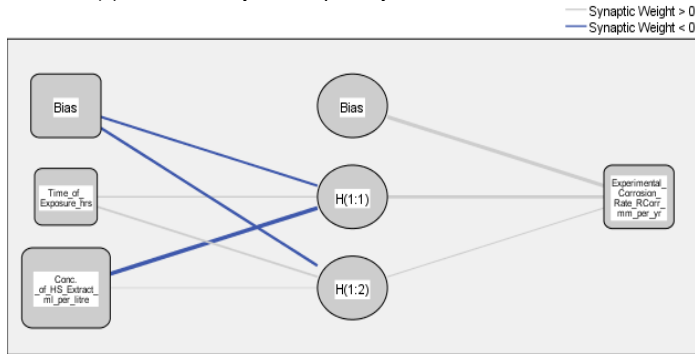
Table 5 ANN and MLR predictions of the experimental corrosion rates of corrosion inhibition of mild steel in 1 M hydrochloric acid using HB leaf extract

(a) Prediction values of ANN and MLR in 1MHCl

Case	Time of Exposure (hrs)	Conc. of HCl (M)	Con. of HS Extract (ml/L)	Experimental Corrosion rate, R _{Corr} (mm/yr) (Control)	Prediction of R _{Corr}			
					y ANN	Error in prediction	y MLR	Error in prediction
1	24	1.00	0.00	3.4297	2.9765	-.02	2.78657	-.45
2	48	1.00	0.00	3.7718	3.1566	-.31	2.92469	-.72
3	72	1.00	0.00	3.3987	3.3348	.11	3.06282	-.27
4	96	1.00	0.00	3.2025	3.5103	.36	3.20094	-.01
5	120	1.00	0.00	3.296	3.6827	.33	3.33907	-.03
6	144	1.00	0.00	4.1391	3.8511	-.46	3.47719	-.80
7	168	1.00	0.00	4.3991	4.0147	-.66	3.61531	-.99
8	192	1.00	0.00	4.4284	4.1725	-.64	3.75344	-2.74
9	24	1.00	5.00	0.2947	.7973	.07	1.60429	1.50
10	48	1.00	5.00	0.2544	.9204	-.09	1.74241	1.36
11	72	1.00	5.00	0.4778	1.0582	-.05	1.88054	1.43
12	96	1.00	5.00	0.4570	1.2111	.01	2.01866	1.50
13	120	1.00	5.00	0.4607	1.3788	.10	2.15679	1.62
14	144	1.00	5.00	1.8333	1.5606	.14	2.29491	1.67
15	168	1.00	5.00	2.6087	1.7553	.20	2.43304	1.76
16	192	1.00	5.00	2.5019	1.9609	.27	2.57116	2.50
17	24	1.00	0.00	3.1385	2.9765	.27	2.78657	-.15
18	48	1.00	0.00	3.5069	3.1566	-.05	2.92469	-.45
19	72	1.00	0.00	3.2765	3.3348	.24	3.06282	-.15
20	96	1.00	0.00	3.128	3.5103	.44	3.20094	.07
21	120	1.00	0.00	3.1059	3.6827	.52	3.33907	.16
22	144	1.00	0.00	3.8031	3.8511	-.12	3.47719	-.47
23	168	1.00	0.00	4.0099	4.0147	-.28	3.61531	-.60

24	192	1.00	0.00	4.0161	4.1725	-0.22	3.75344	-3.56
25	24	1.00	10.00	0.2807	.2493	-0.30	.42201	.29
26	48	1.00	10.00	0.2474	.3009	-0.30	.56014	.30
27	72	1.00	10.00	0.4591	.3599	-0.20	.69826	.41
28	96	1.00	10.00	0.4351	.4266	-0.14	.83638	.49
29	120	1.00	10.00	0.4379	.5008	-0.14	.97451	.49
30	144	1.00	10.00	0.4257	.5819	-0.13	1.11263	.52
31	168	1.00	10.00	0.4464	.6691	-0.08	1.25076	.58
32	192	1.00	10.00	0.6693	.7612	-0.03	1.38888	2.54
33	24	1.00	0.00	3.0876	2.9765	.32	2.78657	-1.10
34	48	1.00	0.00	3.3499	3.1566	.11	2.92469	-0.30
35	72	1.00	0.00	3.2063	3.3348	.31	3.06282	-0.08
36	96	1.00	0.00	3.021	3.5103	.55	3.20094	.17
37	120	1.00	0.00	2.9922	3.6827	.62	3.33907	.27
38	144	1.00	0.00	3.5093	3.8511	.17	3.47719	-0.17
39	168	1.00	0.00	3.7896	4.0147	-0.05	3.61531	-0.38
40	192	1.00	0.00	3.9565	4.1725	-0.16	3.75344	-4.73
41	24	1.00	15.00	0.2649	.2084	-0.19	-.76027	-.93
42	48	1.00	15.00	0.236	.2534	-0.12	-.62214	-.84
43	72	1.00	15.00	0.3421	.3043	-0.07	-.48402	-.79
44	96	1.00	15.00	0.3581	.3610	.01	-.34589	-.70
45	120	1.00	15.00	0.4084	.4229	.07	-.20777	-.62
46	144	1.00	15.00	0.4111	.4889	.10	-.06964	-.58
47	168	1.00	15.00	0.4288	.5577	.08	.06848	-.58
48	192	1.00	15.00	0.5017	.6279	.12	.20660	2.66

(b) Hidden layer- output layer table



Hidden layer activation function: Hyperbolic tangent

Output layer activation function: Identity

© Independent Variable table

Independent Variable Importance and Parameter Estimates of the prediction of corrosion inhibition of mild steel in 1M HCl medium by the HS Extract using AN

Independent Variable Importance			
	Importance		
Time of Exposure_hrs	0.229		
Conc. of HS Extract ml_per_litre	0.771		
Parameter Estimates			
Predictor	Predicted		
	Hidden Layer 1	Hidden Layer 2	Output Layer
	H (1:1)	H (1:2)	Experimental Corrosion Rate RCORR_mms_per_yr
Input Layer	(Bias)	-0.458	-0.487
	Time of Exposure_hrs	0.185	0.279
	Conc. of HS Extract ml_per_litre	-1.305	0.063
Hidden Layer 1	(Bias)		.602
	H (1:1)		.516
	H (1:2)		.142

Predictive equation by MLR

Predicted-RCorr_{HS} in HCl by MLR = 2.648 + 0.006 (time of exposure) – 0.236 (conc. of HS leaf-extract)

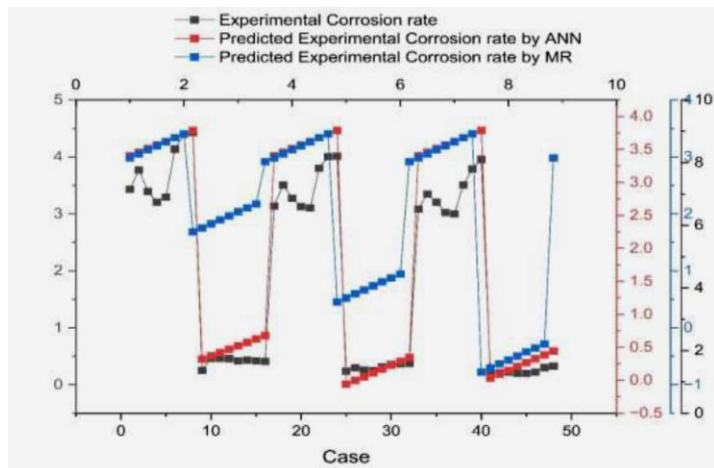


Figure 5: Comparative Analysis of Multiple Linear Regression (MLR) and Artificial Neural Network (ANN) Models for Corrosion Inhibition Effectiveness of leaves of Hibiscus sabdariffa (HS) Extract on Mild Steel in 1 M HCl

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 (Anyakwo, 2007). This accounts for the reduction in the corrosion rate of inhibited reaction compared to the blank. For the inhibited reaction, corrosion rate decreased with increase in inhibitor concentration at any given period 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, Shahena et al., using 1M HNO₃ and Cu-Zn alloy, concluded that HB is an excellent inhibitor and that its IE increased with increase in the inhibitor concentration. Also, Amabua et al., (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 in 0.5M H₂SO₄. Here, IE also increased with inhibitor concentration. The PDP also shows HB as a mixed inhibitor, retarding both anodic and cathodic reactions. Nguyen et al., (2018), studied HB as an inhibitor using 0.1M HCL and X-ray photoelectron spectroscopy, confirmed that HB is a mixed inhibitor.

The ANN and MLR prediction models are reliable in predicting corrosion rates and IE and ANN showed closer margin to the results of the study (Ndukwe, 2017).

The models employed in the study revealed that while inhibitor concentration and time of exposure of mild steel to the corrodent affect the IE, inhibitor concentration has a greater effect on IE than exposure time. HB leaf extract showed higher IE values with 15mL/L inhibitor concentration with 0.5M H₂SO₄ environment than that of 1M HCl in both techniques of GA and PDP.

5.0 Conclusion

Hibiscus sabdariffa leaf extract is an excellent corrosion mixed inhibitor for mild steel, offering an IE value of 94% in HCl and 95% in H₂SO₄ with weight loss approach while PDP approach showed IE of 82.8% in HCl and 96% in H₂SO₄. The HB leaf extract proved a little more efficient in corrosion inhibition of mild steel in H₂SO₄ environment than in HCl. 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 in both acid media. ANN predictive model agreed more closely with experimental data than MLR in both media.

Declaration Statement

The authors agreed with total interest to submit the manuscript entitled, 'Comparative study of mild steel corrosion inhibition performance of *hibiscus sabdariffa* leaf extract in different acid media' 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.

Conflict of Interest

The authors declare no conflict of interest.

Author Contribution

P. C. Agu – Original draft, Conceptualization and Methodology.

A. I. Ndukwe and C. Onuoha: Supervision, review and editing. N. A. Nnodum, K. Okon, and C. C. Ozoh: Formal analysis.

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