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Application of Neem Leaf as an Inhibitor for Mild Steel Corrosion Control in Hydrochloric Acid Medium

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ABSTRACT

This study focused on the application of Neem (*Azadirachta indica)* leaf as an inhibitor of mild corrosion in HCl medium. The inhibitor was extracted from ground neem leaf. Phytochemicals and functional groups of the inhibitor were examined. Stock solution of 10 g/L of the extract was used to obtain various inhibitor concentrations (0.2 g/L - 1.0 g/L) through standard process of dilution. The inhibition process parameters were determined by gravimetric method. It included optimization of the efficiency of neem leaf using response surface methodology (RSM). Alkaloid was found as the most predominant phytochemical with a value of 218.62 ±0.03 mg/100g, followed by cardiac glycoside of 112.41 ±0.01 mg/100g value. It was also revealed that C-H, N-H, C-F, O-H, C=H, and C=O are the functional groups. These characteristics show that neem leaf is a potential antioxidant with abundant medicinal and inhibitive properties. For the revealed physical adsorption mechanism, Frumkin isotherm was found to be the best fitted isotherm. Optimum inhibition efficiency of 98.38% was attained at temperature of 310.96 K, time of 3.46 h and inhibitor concentration of 0.82 g/L. Therefore, neem leaf is recommended for application in corrosion control of mild steel structure in HCl environment.

Keywords: Neem leaf, inhibitor, mild steel, corrosion, HCl

INTRODUCTION

Mild steel is a useful alloy with strong tensile strength. It is simple to produce, and widely available. These explain why it is largely employed in various sectors [1]. However, when alloys (such as mild steel) come in close contact with hostile media, their surfaces corrode. Temperature and other variables, such as the concentration of the aggressive medium, affect how quickly and how much corrosion occurs. When metal comes into touch with acid solutions during acid cleaning, acid de-scaling and other chemical activities, it corrodes severely [2, 3]. Corrosion of metal has negative impacts on the integrity of metallic structures. Its consequences are huge: the safety, reliability and efficiency of equipment or structures are compromised [4]. In several cases, corrosion necessitates expensive replacements though the amount of metal destroyed is small. As such, efforts are being made to control corrosion.

Researchers from a variety of industrial sectors are now involved in studies on corrosion processes and control [2, 5]. One of the most practical methods of metal corrosion control is application of inhibitors. The majority of corrosion inhibitors are expensive and environmentally risky synthetic compounds. Synthetic corrosion inhibitors such as sodium molybdate and sodium chromate are not environmentally friendly. They are non-renewable and hazardous chemicals; which are directly and indirectly harmful to the eco-system. Sodium molybdate and allied inhibitors used in previous research works were reported as anodic inhibitors. Their usage requires the presence of oxygen or other oxidizing agents in solution. Little and contradicting research data are available about the mechanism of action of MoO_4^{2-} as corrosion inhibitor. As such, it is necessary to investigate corrosion inhibitors that are eco-friendly from renewable plant extracts of rightful phytochemicals and effective inhibitive properties [6, 7]. Plant material of interest is neem leaf. There is need to convert the plant leaf (which is often regarded as waste) to useful product (corrosion inhibitor). Such inhibitor may be used to control mild steel corrosion in hydrochloric acid medium.

Therefore, emphasis has shifted to sourcing and development of corrosion inhibitors that are safe to the environment [8]. In the 21st century, there are several research reports on plant leaves acting as efficient corrosion inhibitors in acidic conditions [9]. Phytochemicals found in extracts from plant materials include alkaloids, tannins, flavonoids, saponins and phenolic acids, which have electronic structures similar to those of traditional organic corrosion inhibitors. It has been reported that corrosion of metals can be inhibited by plant extracts [10, 11]. There is need for extensive use of biomass extract for the prevention of metallic corrosion. Study of inhibitory effect of neem leaf on HCl corrosion in mild steel falls within such scope. Though its anti-oxidizing property is fairly known, its discrete inhibitive capability (based on the roles of the phytochemicals and functional groups) for mitigating mild steel corrosion in HCl medium has not been investigated. Therefore, this study aims to develop and apply neem leaf as corrosion inhibitor in HCl medium. The study will establish optimum parameters for the corrosion inhibition process.

MATERIALS AND METHOD

The materials used include neem leaves, ethanol, acetone, hydrochloric acid, mild steel coupon, emery papers and electronic weighing balance. The mild steel has the following composition; Si, 0.02%; S, 0.02%; C, 0.23%; Cu, 0.01%; Mn, 0.11%; P, 0.02%; Cr, 0.01%, Ni, 0.02 and Fe, 99.56 %). The mild steel was authenticated at the Department of Chemical Engineering, Nnamdi Azikiwe University, Awka, Nigeria, while the neem leaf was authenticated by at the Faculty of Agriculture, Southern Delta University, Ozoro, Delta State, Nigeria.

Preparation of 1M HCl

Medium of 1M HCl was prepared using distilled water. In the process, 85.91 mL HCl (36% pure, specific gravity of 1.18) was put in to 1 L measuring cylinder containing 600 mL of distilled water. Then, the medium was made up (with additional distilled water) to 1L.

Characterization of *Azadirachta indica* Leaf

Standard method used by previous report was implemented in the phytochemical analysis of the *Azadirachta indica* leaf [12]. It involved qualitative and quantitative analyses, were the presence and quantities of alkaloids, cardiac glycosides, flavonoids, phenols, phytate, saponins and tannins were determined.

Spectroscopic Analysis of *Azadirachta indica* Leaf Functional Groups

Fourier transform infrared (FTIR) spectrophotometer (Cary 630, Agilent Technologies USA) was employed to determine the functional groups of the *Azadirachta indica* leaf. FTIR spectrum was obtained in transmission mode using the Br pellet method [8].

Weight Loss Technique

Weight loss (gravimetric) technique used by Omotioma and Onukwuli [13] was adopted in this study. Mild steel used was divided into corrosion coupons of 3cmx3cm sizes. They were polished with varied grades of emery paper. It was followed by degreasing them with acetone, and then washed with distilled water. The inhibitor was obtained by soaking 45.0 g of the ground *Azadirachta indica* leaf in 1 litre of ethanol (99.7% v/v) for two days. Thereafter, filtration and subsequent separation of the extract from the ethanol followed. Then, 10 g of the extract was dissolved in 1000 ml of the 1M HCl. The stock solution of 10 g/L was used to obtain various concentrations of *Azadirachta indica* leaf (0.2 g/L - 1.0 g/L) through standard process of dilution. The weighed mild steel was put into 250 ml separate open beakers containing 150 ml of 1M HCl (blank). Also, mild steel samples were put into 250 ml of separate open beakers containing 150 ml of 1M HCl with varied concentrations of *Azadirachta indica* leaf**.**

There was periodic evaluation of the corrosion inhibition at various temperatures, times and inhibitor concentration. At regular interval, the mild steel samples were withdrawn from the medium, degreased, washed, dried and weighed. Then the corrosion inhibition parameters we determined. The weight loss, corrosion rate, inhibition efficiency and degree of surface coverage were appraised by Equations (1), (2), (3) and (5) respectively [7, 10, 14]. Arrhenius equation was deployed to ascertain the activation energy of the control process (6), while Equation (7) was used to evaluate the corresponding heat of adsorption Q_{ads} ($kJmol^{-1}$). Furthermore, mechanism of the corrosion inhibition was tested using Langmuir, Frumkin, Temkin and Flory-Huggins of Equations (7), (8), (9) and (10) respectively.

$$\Delta w = w_i - w_f \qquad (1)$$

$$CR = \frac{w_i - w_f}{At} \qquad (2)$$

$$IE\% = \frac{\omega_0 - \omega_1}{\omega_0} * 100 \qquad (3)$$

$$\Theta = \frac{\omega_0 - \omega_1}{\omega_0} \tag{4}$$

$$\text{In(CR)} = \text{InA} - \left({}^{E_a}/_{R}\right)\left(\frac{1}{\text{T}}\right) \tag{5}$$

$$Q_{ads} = 2.303R\left[\log\left(\frac{\theta_2}{1-\theta_2}\right) - \log\left(\frac{\theta_1}{1-\theta_1}\right)\right] * \frac{T_2.T_1}{T_{2-T_1}} \tag{6}$$

$$log\frac{C}{\theta} = \log C - logK \tag{7}$$

$$\log\left((C) * \left(\frac{\theta}{1-\theta}\right)\right) = 2.303logK + 2\alpha\theta \tag{8}$$

$$\theta = -\frac{2.303logK}{2a} - \frac{2.303logC}{2a} \tag{9}$$

$$\log\left(\frac{\theta}{C}\right) = \log K + xlog(1-\theta) \tag{10}$$

where w_i and w_f are the initial and final weight of mild steel samples respectively; ω_1 and ω_0 are the weight loss values in presence and absence of *Azadirachta indica* leaf, respectively. A is the mild steel area and t is the time of immersion. In addition, CR is the corrosion rate, A is the frequency factor and $E_{a.}$ is the activation energy. More so, α is the lateral interaction term, '*a*' is the attractive parameter, *C* is the concentration and *x* is the size parameter, R is the gas constant, θ_1 and θ_2 are the degrees of surface coverage at temperatures T_1 and T_2 respectively.

Design of Experiment for the Optimization Process

On the response surface methodology (RSM), Design Expert software was used to design the experiment. Interactive effects of inhibitor concentration, temperature and time on the inhibition efficiency were determined in with previous report [8]. Design matrix is presented in Table 1.

Table 1. Factors of the matrix of the Experiment

Factor	Name	Units	Minimum	Maximum	Mean	Std. Dev.
A	Inhibitor concentration	g/L	0.6	1	0.8	0.1451
B	Temperature	K	303	323	313	7.25
C	Time	hr	1	5	3	1.45

RESULTS AND DISCUSSION

Characteristics of *Azadirachta indica* Leaf

Phytochemicals of neem leaf are presented in Table 2. They are shown in terms of qualitative and quantitative forms. From the analysis, alkaloid is the most predominant phytochemical with a value of 218.62 ±0.03 mg/100g. It is followed by cardiac glycoside of 112.41 ±0.01 mg/100g value. Presence of the phytochemicals shows that *Azadirachta indica* leaf is a potential antioxidant of great medicinal and inhibitive properties [5, 12, 15]. The corresponding functional groups as characterized using Fourier transform infrared spectrophotometer are shown in the spectrum (Fig. 1). The infrared chart was used to identify the functional groups as C-H stretch, N-H bend, C-F stretch, O-H stretch, C=H stretch, and C=O stretch. Presence of the heteroatoms of N and O confirms it as a potential corrosion inhibitor [16, 17].

Table 2: Phytochemical analysis of *Azadirachta indica* leaf

Names of the phytochemicals	Units	Qualitative status	Quantitative data
Tannin	mg/100g	+	60.31 ±0.02
Alkaloid	mg/100g	+++	218.62 ±0.03
Phytate	mg/100g	+	46.90 ±0.07
Flavonoid	mg/100g	-	9.05 ±0.10
Cardiac glycoside	mg/100g	++	112.41 ±0.01
Saponin	mg/100g	+	38.67 ±0.13
Phenolics	GAE/g	+	51.52 ±0.11

+ (in traces), ++ (concentrated), –. (little) and +++ (highly concentrated)

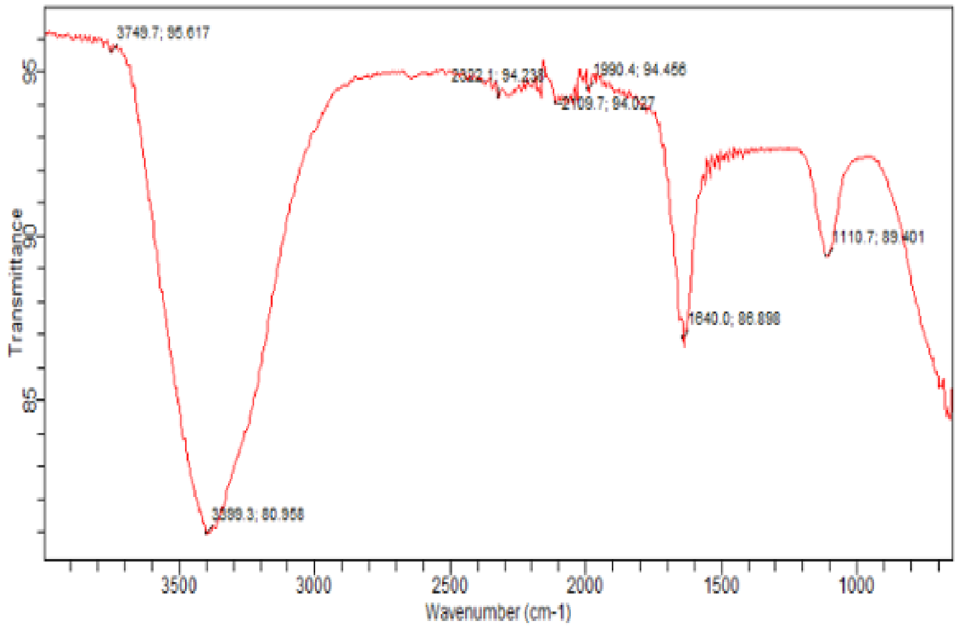

Figure 1. Spectrum of *Azadirachta indica* Leaf

Mechanism of Tte Inhibition Process

Graphs of the tested Langmuir, Frumkin, Temkin Temkin and Flory-Huggins isotherms are presented in Figs. (2), (3), (4), and (5) respectively. On the average, Frumkin isotherm was found to be the best fitted adsorption isotherm because it has the highest values of correlation coefficient, R^2 (closest to 1.00). It recorded 0.981 ad 0.989 R^2 values. Linkage of Frumkin isotherm to the adsorption of the *Azadirachta indica* leaf on surface of the mild steel shows that there is no chemical reaction between the mild steel and the *Azadirachta indica* leaf [8, 10, 11, 18]. Hence, it is a physical adsorption mechanism.

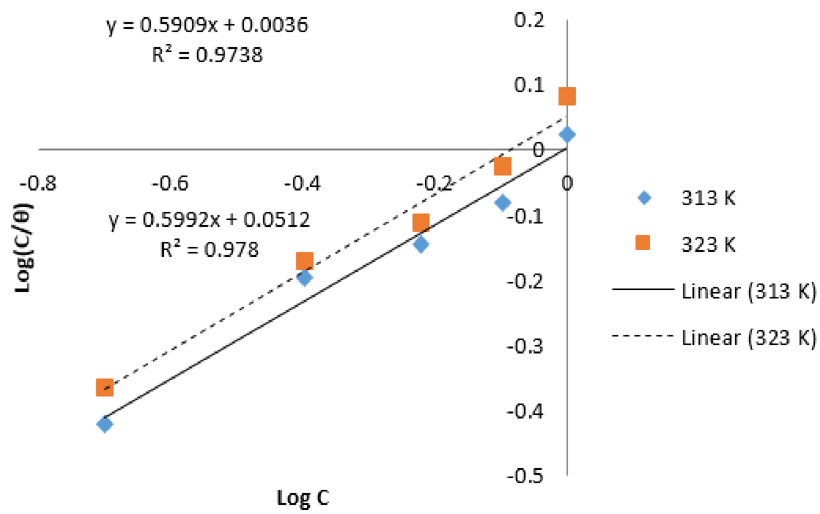

Figure 2. Fitting of Langmuir isotherm

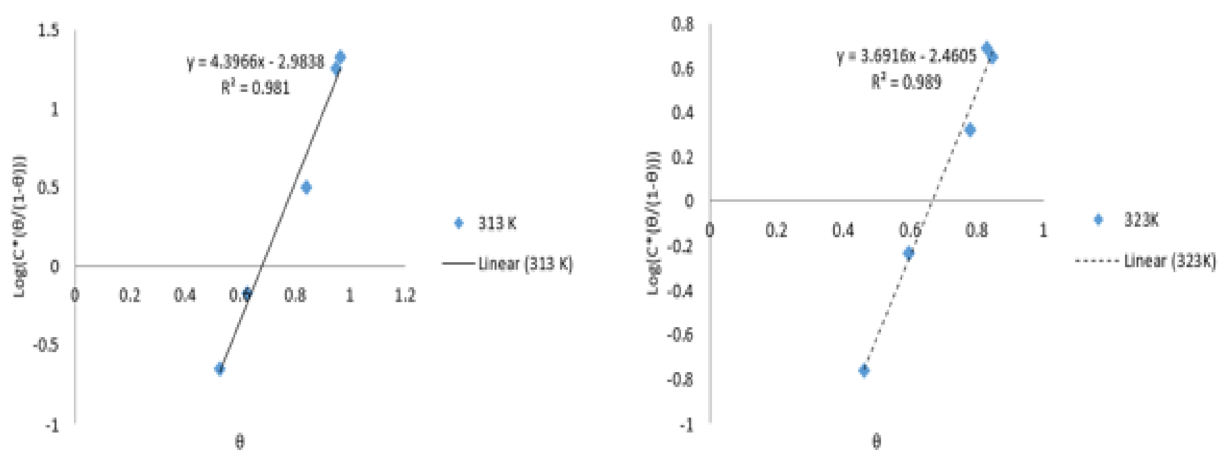

Figure 3. Fitting of Frumkin isotherm

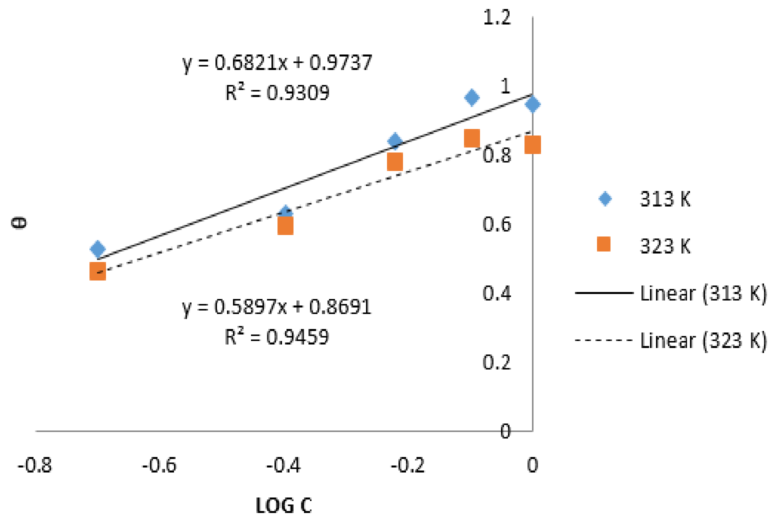

Figure 4. Fitting of Temkin isotherm

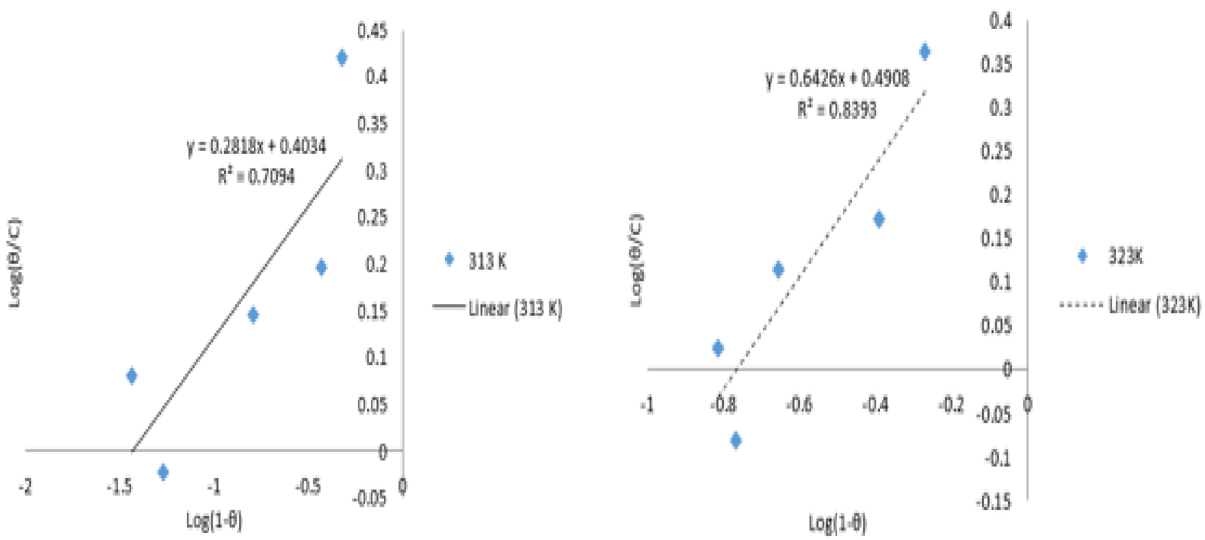

Figure 5. Fitting of Flory-Huggins isotherm

Analysis of the RSM

Result of the RSM of the efficiency of *Azadirachta indica* leaf is shown in Table 3. The experimental data stem from the interactive influence of the corrosion inhibition factors. Maximum efficiency of *Azadirachta indica* leaf was recorded as 96.45% temperature of 313 K, time of 3 h and inhibitor concentration of 0.8 g/L. Inhibitor of such magnitude of efficiency is highly recommended for corrosion control process [3, 6, 7]

Table 3: Result of the RSM of the inhibition efficiency of *Azadirachta indica* leaf

Std	Run	A - Inhibitor conc. g/L	B - Temperature K	C - Time h	Inhibition efficiency %
12	1	0.8	323	3	81.44
10	2	1	313	3	90.82
14	3	0.8	313	5	94.29
17	4	0.8	313	3	96.45
13	5	0.8	313	1	87.47
11	6	0.8	303	3	90.23
7	7	0.6	323	5	55.93
20	8	0.8	313	3	96.45
9	9	0.6	313	3	81.26
15	10	0.8	313	3	96.45
16	11	0.8	313	3	96.45
3	12	0.6	323	1	48.72
4	13	1	323	1	64.61
8	14	1	323	5	79.56
1	15	0.6	303	1	63.71
19	16	0.8	313	3	96.45
2	17	1	303	1	72.72
5	18	0.6	303	5	66.66
6	19	1	303	5	81.58
18	20	0.8	313	3	96.45

Fit summary of the efficiency of *Azadirachta indica leaf*

Fit summary of the model for the inhibition efficiency of *Azadirachta indica* leaf is presented in Table 4. Four models were tested (linear, 2 factor interaction (2FI), quadratic and cubic models). The quadratic model was suggested as the best fitted model because of the values and relationship between the predicted and adjusted R^2 [17]. Model of inhibition efficiency of *Azadirachta indica* leaf is shown in Equation 12. It is a quadratic model capable of predicting the given levels of each factor.

Table 4. Summary of fitness for the efficiency of *Azadirachta indica* leaf

Model	Adjusted R^2	Predicted R^2	Sequential p-value	
Linear	0.0589	-0.4512	0.2802	
2FI	-0.1357	-5.0558	0.9663	
Quadratic	0.9904	0.9630	< 0.0001	Suggested
Cubic	0.9968	-0.2429	0.0275	Aliased

$$\text{Inhibition efficiency} = +96.91 + 7.30A - 4.46B + 4.08C + 1.95A*B + 1.71A*C + 1.29B*C - 11.56A^2 - 11.77B^2 - 6.72C^2 \quad (12)$$

Result of the Diagnostic Analysis of the RSM

Table 5 presents the report of the diagnostic analysis for the corrosion control using *Azadirachta indica* leaf. Results of the statistical analyses in terms of predicted data, residual, cook's distance, and difference in fit (DFFIT) validate the generated model and overall RSM result. Further analysis in terms of graphical analysis of the predicted versus actual efficiency of *Azadirachta indica* leaf is presented in Figure 6, which displayed a linear graph. It shows that the experimentally-based generated model sufficiently predicted the actual data [13].

Table 5. Report of the diagnostic data of the RSM result

Actual data	Predicted data	Residual	Leverage	Internally studentized residuals	Externally studentized residuals	Cook's distance	Influence on fitted value DFFITS
81.44	80.68	0.7604	0.491	0.721	0.703	0.050	0.690
90.82	92.65	-1.83	0.491	-1.736	-1.970	0.291	-1.935
94.29	94.27	0.0224	0.491	0.021	0.020	0.000	0.020
96.45	96.91	-0.4609	0.118	-0.332	-0.317	0.001	-0.116
87.47	86.11	1.36	0.491	1.291	1.341	0.161	1.317
90.23	89.61	0.6224	0.491	0.591	0.570	0.034	0.560
55.93	56.81	-0.8818	0.793	-1.313	-1.369	0.661	-2.681
96.45	96.91	-0.4609	0.118	-0.332	-0.317	0.001	-0.116
81.26	78.05	3.21	0.491	3.048	10.850	0.896	10.655
96.45	96.91	-0.4609	0.118	-0.332	-0.317	0.001	-0.116
96.45	96.91	-0.4609	0.118	-0.332	-0.317	0.001	-0.116
48.72	49.48	-0.7588	0.793	-1.130	-1.147	0.489	-2.247
64.61	64.57	0.0442	0.793	0.066	0.062	0.002	0.122
79.56	78.72	0.8362	0.793	1.245	1.285	0.594	2.516

63.71	64.89	-1.18	0.793	-1.759	-2.009	1.187	-3.933
96.45	96.91	-0.4609	0.118	-0.332	-0.317	0.001	-0.116
72.72	72.18	0.5362	0.793	0.798	0.783	0.244	1.532
66.66	67.05	-0.3898	0.793	-0.580	-0.560	0.129	-1.097
81.58	81.17	0.4132	0.793	0.615	0.595	0.145	1.165
96.45	96.91	-0.4609	0.118	-0.332	-0.317	0.001	-0.116

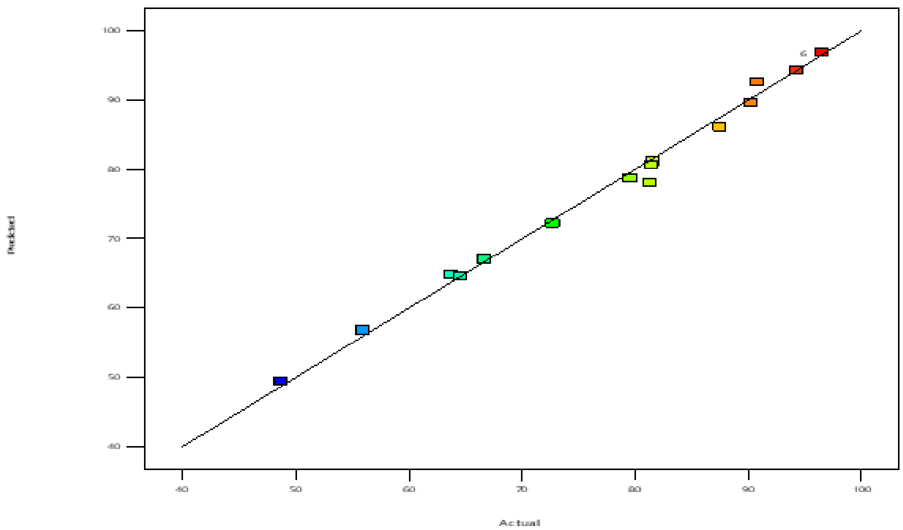

Figure 6. Relationship between actual and predicted efficiency of *Azadirachta indica* leaf

Three-dimensional Graphs of the RSM

Three-dimensional plots of the RSM analysis of the efficiency of *Azadirachta indica* leaf are presented in Figures 7 - 9. Interactive effects of the independent factors on the dependent variable display parabolic curves in all the cases. The nature of graphs (parabolic curves) supports the earlier explanation that quadratic model fits the relationship between the independent factors and the response. In addition, the 3-D plots showcased the optimum inhibition efficiency of 98.38%, which was attained at temperature of 310.96 K, time of 3.46 h and inhibitor concentration of 0.82 g/L. It competed favourably with contemporary green inhibitors [4, 10, 11, 17].

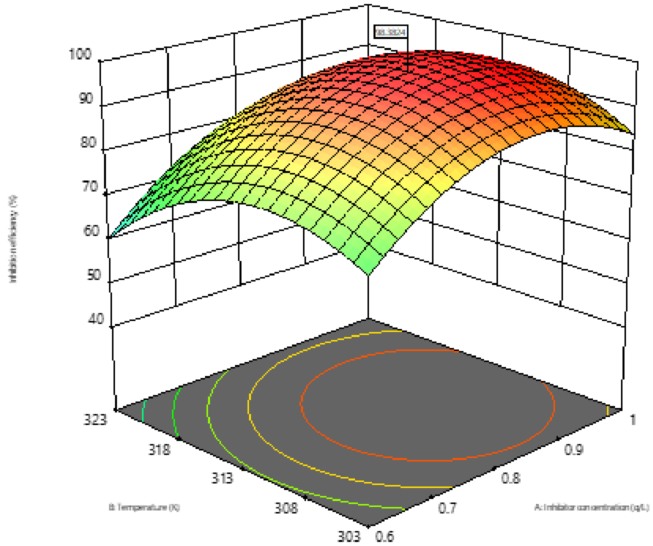

Figure 7. Inhibition efficiency versus inhibitor concentration and temperature

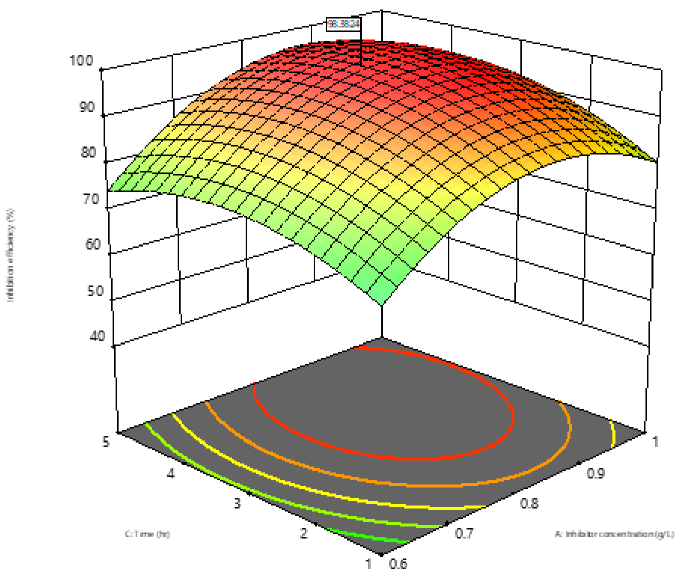

Figure 8. Efficiency versus inhibitor concentration and time

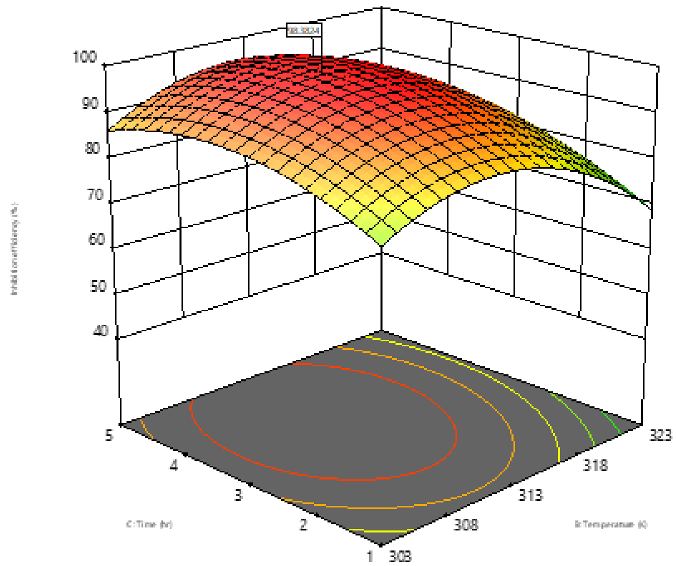

Figure 9. Efficiency versus temperature and time

CONCLUSION

Neem leaf was successfully deployed in corrosion inhibition of mild steel in HCl medium. It was found that alkaloid is the most predominant phytochemical of *Azadirachta indica* leaf with a value of 218.62 ±0.03 mg/100g; followed by cardiac glycoside of 112.41 ±0.01 mg/100g value. The functional groups were revealed as C-H stretch, N-H bend, C-F stretch, O-H stretch, C=H stretch, and C=O stretch. These characteristics presented *Azadirachta indica* leaf as a potential antioxidant with viable inhibitive properties. For the revealed physical adsorption mechanism, Frumkin isotherm was found to be the best fitted isotherm. The optimum inhibition efficiency of 98.38% was obtained at temperature of 310.96 K, time of 3.46 h and inhibitor concentration of 0.82 g/L. It is recommended that *Azadirachta indica* leaf should be used for corrosion control of mild steel structure in HCl solution.

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