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Corrosion control of mild steel in hydrochloric acid solution using extract of sweet potato leaf as green inhibitor

Omotioma Monday*, Okolo Wilfred Nkemjika, Onuora Okorie, Ekete Juliet Azuka, Aliozo Sandra Ogechukwu

Department of Chemical Engineering, Enugu State University of Science and Technology, PMB 01660, Enugu State, Nigeria

*E-mail address: monday.omotioma@esut.edu.ng

ABSTRACT

This study investigated extract of sweet potato leaf as green corrosion inhibitor of mild steel in hydrochloric acid solution. Qualitative and quantitative analyses of the extract were carried out to identify and quantify its phytochemicals, while Fourier transforms infrared (FTIR) spectroscopy was used to detect the functional groups. Gravimetric method was employed in the corrosion control study. Effects of inhibitor concentration, temperature, and time on the weight loss, corrosion rate, inhibition efficiency, and degree of surface coverage were determined Analyses of the experimental results showed that phytochemicals of alkaloids, cardiac glycosides, flavonoids, phenolics, phytates, saponins, and tannins were present in the sweet potato leaf extract at various degrees. The extract contains more alkaloids (334.1±0.04 mg/100g) and saponins (320.2±0.31 mg/100g), indicating good corrosion inhibitive properties. The predominant functional groups include; ≡C-H stretch, C-H stretch, C-F stretch, N=O stretch, C-O stretch, C-H out of the plane, and C-H deformation. Quadratic model adequately defined the connection between inhibition efficiency of the extract and factors of the corrosion inhibition; inhibitor concentration, temperature and time. The optimum Inhibition efficiency of 82.58% was derived at a time of 3.91hrs, the temperature of 316.39 K, and inhibitor concentration of 0.80 g/L. Thermodynamic and adsorption parameters revealed that the adsorption of molecules of sweet potato leaf on the mild steel surface is exothermic and it transpired by physical attachment mechanism. Based on the performance evaluation, the sweet potato leaf extract should be applied as green (eco-friendly) corrosion inhibitor of mild steel structure in hydrochloric acid environment.

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***Keywords*:** Sweet potato leaf, Green corrosion inhibitor, Phytochemicals

1. INTRODUCTION

Corrosion causes tremendous damage to the nation's economy. About 10% of iron, aluminum as well as mild steel and its alloys produced annually are lost to corrosion. As such, maintenance operations (cleaning, descaling and pickling) are required to elongate shelf-life of metals. Hydrochloric acid is widely used in pickling, especially in maintaining equipment (such as metallic pipe and heat exchanger). It has side effect of corroding the metal. As such, pickling solution is enriched with inhibitor. Mixed inhibitor is a well-recognized inhibitor for corrosion control process [1]. Refineries and petrochemical industries employ a variety of film-forming inhibitors to control wet corrosion. Most of the inhibitors are long-chain nitrogenous organic materials, including amines and amides.

Several studies on corrosion inhibition of metals using synthetic substances have been reported [2, 3]. It has been noted that synthetic substances are expensive and hazardous to the environment. Though plant extracts have been mentioned to be eco-friendly, they have not been adequately exploited and developed for the corrosion control process. Attention has been focused on the corrosion inhibiting properties of plant extract because plant extracts serve as incredibly rich sources of natural chemical compounds [4-7]. They are environmentally acceptable, readily available, and renewable sources of materials and can be extracted by simple procedures. In this study, potato leaf extract was selected as an inhibitor to examine its ability to prevent corrosion of mild steel in HCl medium. Potato leaf is a leguminous shrub plant growing mainly in the tropical regions of Asia, Africa, and Australia. Sweet potato leaves are in abundance but are usually treated as waste. There is a need for its conversion to the new eco-friendly value-added product such as corrosion inhibitor. Thus, the aim of this study is to control the corrosion of mild steel in HCl solution using sweet potato leaf extract as an inhibitor.

2. MATERIALS, CHEMICALS AND EQUIPMENT USED

The materials used during this project work include: mild steel coupon, glass measuring cylinder, electronic weighing balance, conical flask and cork, emery papers, water bath, retort stand, beakers, spatula, volumetric flask, hydrochloric acid, distilled water, ethanol, acetone, $FeCl_3$, H_2SO_4, NH_4OH, filter cloth, spectrophotometer, stopwatch, and sweet potato leaf. The mild steel used for this study was cut into corrosion coupons of sizes 3×3 cm. The surface preparation of the mechanically polished specimens were carried out using different grades of emery paper and then degreased with acetone after washing with distilled water. Sweet potato leaves were obtained, washed thoroughly with distilled water (to remove impurities), dried and ground in to fine particles. 30 grams of inhibitor were soaked in 1000 ml of ethanol for 48 hours. Then, the mixture was filtered, and the extract obtained was concentrated by evaporating the ethanol from the filtrate. The obtained extract was stored in a plastic container (from where samples were taken for characterization and corrosion control investigation). Phytochemicals and functional groups of the extract (inhibitor) were determined. Methods used by previous research work [8] were adopted for the phytochemical analysis of the leaf extract.

FTIR spectrophotometer (Model: Cary 630, Agilent Technologies USA) was used to identify functional groups of the sweet potato leaf.

2. 1. Weight loss procedure

The weight loss (gravimetric) procedure applied by previous reports [5, 9] was used in this experimental work. The corrosion control studies were carried out using 150 ml of 1M HCl solution in 250 ml beakers. Loss in weight was monitored at various temperatures and time of immersion in 1.0M HCl medium, in the absence and presence of varied sweet potato leaf extract concentration. The mild steel samples were taken out at regular time intervals, cleaned and reweighed. Calculations of weight loss (Δw), corrosion rate (CR), inhibition efficiency (IE) and degree of surface coverage were done by employing Equations (1), (2), (3) and (4) respectively:

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

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

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

$$\theta = \frac{\omega_0 - \omega_1}{\omega_0} \quad (4)$$

where w_i and w_f are the respective initial and final weight of mild steel coupons; ω_1 and ω_0 represent the respective weight loss values in presence and absence of sweet potato leaf extract. A is the total area of the mild steel coupon and t is the time of immersion.

2. 2. Determination of effects of corrosion control variables

Effects of corrosion control variables (inhibitor concentration, temperature, and time) on weight loss, corrosion rate, inhibition efficiency) were determined. Interactive effects of inhibitor concentration, temperature, and time on the inhibition efficiency were determined using response surface methodology (RSM).

Design-Expert software 11 (tool option: central composite design) was used to design the experiment. The experimental results were analyzed using mathematical modeling and optimization.

2. 3. Determination of thermodynamic and adsorption properties

Activation energy, the heat of adsorption, and physical adsorption isotherms were considered in line with the method used by report [5, 10, 11]. Activation energy and heat of adsorption of the corrosion inhibition process were determined using Equations (5) and (6) respectively. To ascertain the adsorption parameters, data of the experimental study were put to adsorption isotherms of Langmuir and Temkin; Equations (7) and (8) respectively.

$$\ln(CR) = lnA - ({}^{E_a}/_{R})(\tfrac{1}{T}) \quad (5)$$

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

$$log\,\frac{C}{\theta} = \log C - logK \quad (7)$$

$$\theta = -\frac{2.303logK}{2a} - \frac{2.303logC}{2a} \quad (8)$$

3. RESULTS AND DISCUSSION

3. 1. Characterization of the sweet potato leaf extract

3. 1. 1. Phytochemical analysis of sweet potato leaf extract

Qualitative and quantitative analyses of the phytochemicals of sweet potato leaf extract are presented in Table 1. The qualitative results of the phytochemicals are denoted with symbols; +++ (highly concentrated), ++ (concentrated), + (in traces), and – (absent or too little to be observed qualitatively). It showed that phytochemicals of alkaloids, cardiac glycosides, flavonoids, phenolics, phytates, saponins, and tannins were present in the extract at various degrees. Plant extract is known to contain phytochemicals suitable for medicinal purpose [12, 13]. In this case, the sweet potato leaf contains more alkaloids (334.1±0.04 mg/100g) and saponins (320.2±0.31 mg/100g), indicating that it has proper corrosion inhibitive properties. Alkaloids are well recognized phytochemical species for corrosion inhibition efficiency [8].

Table 1. Phytochemical analyses of sweet potato leaf extract.

Phytochemicals	Qualitative analysis	Quantitative analysis
Alkaloids (mg/100g)	+++	334.1±0.04
Cardiac glycosides (mg/100g)	+	75.50±0.31
Flavonoids (mg/100g)	++	203.5±0.22
Phenolics (GAE/g)	+	50.40±0.01
Phytates (mg/100g)	++	171.4±017
Saponins (mg/100g)	+++	320.2±03
Tannins (mg/100g)	-	12.30±0.15

+++ (highly concentrated), ++ (concentrated), + (in traces), - (absence or too little to be observed qualitatively).

3. 1. 2. FTIR results (functional groups)

FTIR spectrum of the sweet potato leaf extract is shown in Figure 1. The predominant functional groups of the extract include; ≡C-H stretch, C-H stretch, C-F stretch, N=O stretch, C-O stretch, C-H out of plane and C-H deformation. According to [11], the nature of the functional groups (structural factor) affects the actions of the inhibitor. Hence, these functional groups showed that they can be adsorbed and used to block active centre for metal dissolution. These functional groups can impede metallic corrosion by forming protective surface film that separates the metal from the corrosive environments. Polar functional groups and the structural multiple bonds (double and triple) can act as adsorption centres during metal inhibitor interactions [6, 11].

Sample ID:SWEET POTATO
Sample Scans:30
Background Scans:16
Resolution:8
System Status:Good
Method Name:ABSORBANCE
User:Admin
Date/Time:2021-03-26T04:33:09.432-07:00
Range:4000 - 650
Apodization:Happ-Genzel
File Location:C:\Program Files\Agilent\MicroLab PC\Results\SWEET POTATO_2021-03-26T04-33-09.a2r

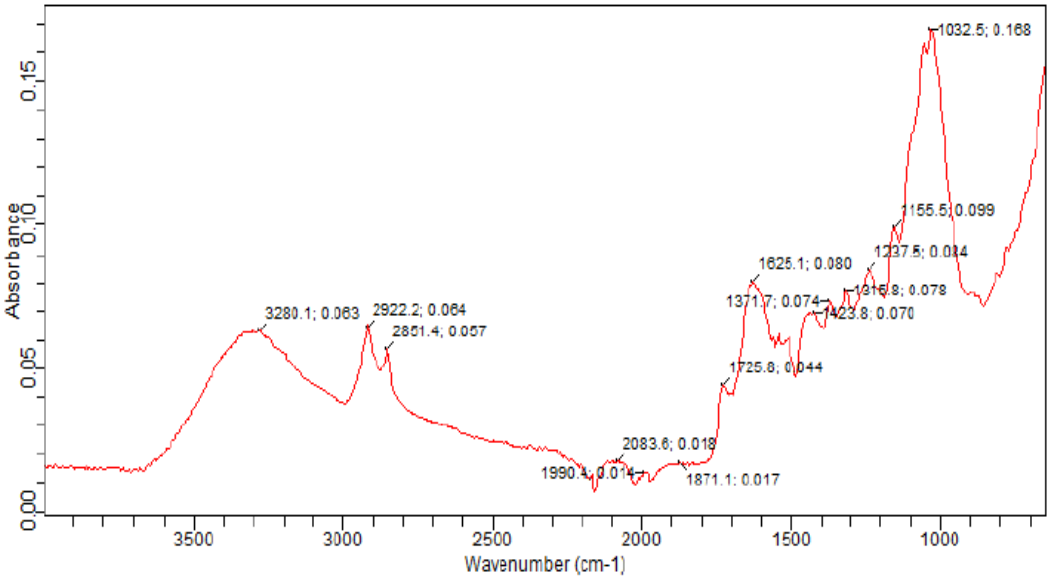

Figure 1. Functional groups of the sweet potato leaf extract

3. 2. Effects of process variables on the corrosion inhibition process

Effect of process variables of time, inhibitor concentration (at 313K), temperature and inhibitor concentration (at 323K) on the corrosion control are shown in Tables 2, 3 and 3 respectively. ΔW_0 represents loss in weight in the absence of inhibitor; ΔW_1 represents loss in weigh in the presence of inhibitor; CR_0 represents corrosion rate in the absence of inhibitor;

CR_1 represents corrosion rate in the presence of inhibitor; Θ represents Degree of surface coverage; and IE represents Inhibition efficiency. For process variable time, it was observed that increase in time increased inhibition efficiency until it got to the peak at time 4hrs from where a decrease in the inhibition efficiency was noticed from 84.48% (at immersion time of 4hrs) to 80%. Similarly, inhibition efficiency increased with increase in inhibitor concentration. But increase in temperature decreased inhibition efficiency. It corroborates with the reports [8, 14-16]. Also, effect of inhibitor concentration was obtained at 4hrs and 313K, while the effect of temperature on the responses was obtained at 4hrs and 0.8 g/L. The inhibition efficiency was determined as a function of inhibitor concentration, temperature, and time. Within the saturation limits, the inhibition efficiency increased with increase in concentration of the inhibitor and time but decreased with increase in temperature. These observations corroborate with previous reports [3, 6].

Table 2. Effect of time on the responses of the inhibition proces

Time (hr)	W0 (g)	CR0 (mg/cm^2hr)	W1 (g)	CR1 (mg/cm^2hr)	IE (%)	Θ
1.00	0.14	15.556	0.06	6.667	57.14	0.5714
2.00	0.29	16.111	0.10	5.556	65.52	0.6552
3.00	0.34	12.593	0.08	2.963	76.47	0.7647
4.00	0.58	16.111	0.09	2.500	84.48	0.8448
5.00	0.60	13.333	0.12	2.667	80.00	0.8000

Table 3. Effect of concentration on the responses of the inhibition process at 313 K

Inh. Conc. (g/L)	W_0 (g)	CR_0 (mg/cm^2hr)	W_1 (g)	CR_1 (mg/cm^2hr)	IE (%)	Θ
0.00	0.58	16.111				
0.20			0.27	7.500	53.45	0.5345
0.40			0.17	4.722	70.69	0.7069
0.60			0.13	3.611	77.59	0.7759
0.80			0.09	2.500	84.48	0.8448
1.00			0.11	3.056	81.03	0.8103

Table 4. Effect of temperature on the responses of the inhibition proces

Temp. (K)	W_0 (g)	CR_0 (mg/cm^{2}hr)	W_1 (g)	CR_1 (mg/cm^{2}hr)	IE (%)	Θ
303.00	0.53	14.722	0.11	3.056	79.25	0.7925
313.00	0.58	16.111	0.09	2.500	84.48	0.8448
323.00	0.69	19.167	0.16	4.444	76.81	0.7681
333.00	0.71	19.722	0.22	6.111	69.01	0.6901
343.00	0.72	20.000	0.24	6.667	66.67	0.6667

Table 5. Effect of concentration on the responses of the inhibition process at 323 K

Inh. Conc. (g/L)	W_0 (g)	CR_0 (mg/cm^{2}hr)	W_1 (g)	CR_1 (mg/cm^{2}hr)	IE (%)	Θ
0.00	0.69	19.167				
0.20			0.35	9.722	49.28	0.4928
0.40			0.3	8.333	56.52	0.5652
0.60			0.23	6.389	66.67	0.6667
0.80			0.16	4.444	76.81	0.7681
1.00			0.19	5.278	72.46	0.7246

3. 3. Optimization results

3. 3. 1. Interactive effects of the process variables on the inhibition efficiency (IE)

RSM results are shown in Table 6. The interactive effects of inhibitor (sweet potato leaf extract) concentration (0.6 – 1.0g/L), temperature (303 -313K), and time (1-5hrs) on the IE showed that the highest value of the IE (84.48%) was obtained at the midpoint of the considered factors. This observation suggests that relationships between IE and considered factors of inhibition concentration, temperature, and time are parabolic in nature [6, 17, 18, 19, 20]. Linear, 2-factor indicator (2FI), quadratic and cubic models were used to test the inhibition efficiency data (Table 7). The quadratic model is suggested as the best-fitted model because the predicted R^2 of 0.8035 is in reasonable agreement with the adjusted R^2 of 0.9516. Table 8 presents the ANOVA of the inhibition efficiency. The model F-value of 42.47 implies the model

is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant [11]. Codes A, B, C, AC, A^2, B^2, and C^2 are significant model terms. The predicted R^2 of 0.8035 is in equitable agreement with the adjusted R^2 of 0.9516; the difference between them is not up to 0.2. Adequate precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 21.768 indicates a satisfactory signal. Generated model of Equation (9) can be used to steer the design space. The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

Table 6. RSM results of the corrosion inhibition

Std	Run	Factor 1 Inhibitor conc. g/L	Factor 2 Temperature K	Factor 3 Time hr	Response 1 Inhibition efficiency (IE) %
7	1	0.6	323	5	64.13
15	2	0.8	313	4	84.48
5	3	0.6	303	5	67.76
18	4	0.8	313	4	84.48
19	5	0.8	313	4	84.48
2	6	1.0	303	3	70.32
13	7	0.8	313	3	76.47
20	8	0.8	313	4	84.48
3	9	0.6	323	3	42.12
11	10	0.8	303	4	79.25
1	11	0.6	303	3	51.14
10	12	1.0	313	4	81.03
16	13	0.8	313	4	84.48
12	14	0.8	323	4	76.81
17	15	0.8	313	4	84.48
6	16	1.0	303	5	72.65
9	17	0.6	313	4	77.59
4	18	1.0	323	3	62.30

8	19	1.0	323	5	66.21
14	20	0.8	313	5	80.00

Table 7. Fit summary of the inhibition efficiency model

Source	Sequential p-value	Adjusted R^2	Predicted R^2	
Linear	0.2696	0.0642	-0.4723	
2FI	0.8239	-0.0768	-4.7973	
Quadratic	< 0.0001	0.9516	0.8035	Suggested
Cubic	0.0081	0.9894	-3.1081	Aliased

Table 8. ANOVA of the inhibition efficiency

Source	Sum of Squares	Df	Mean Square	F-value	p-value	
Model	2617.15	9	290.79	42.47	< 0.0001	significant
A-Inhibitor concentration	247.71	1	247.71	36.17	0.0001	
B-Temperature	87.32	1	87.32	12.75	0.0051	
C-Time	234.26	1	234.26	34.21	0.0002	
AB	0.4095	1	0.4095	0.0598	0.8118	
AC	131.14	1	131.14	19.15	0.0014	
BC	6.07	1	6.07	0.8868	0.3685	
A^2	127.89	1	127.89	18.68	0.0015	
B^2	180.41	1	180.41	26.35	0.0004	
C^2	171.39	1	171.39	25.03	0.0005	
Residual	68.48	10	6.85			
Lack of Fit	68.48	5	13.70			

Pure Error	0.0000	5	0.0000			
Cor Total	2685.63	19				
Std. Dev.	2.62		R^2		0.9745	
Mean	73.73		Adjusted R^2		0.9516	
C.V. %	3.55		Predicted R^2		0.8035	
			Adeq Precision		21.7681	

$$IE = + 85.14 + 4.98A - 2.95B + 4.84C - 4.05AC - 6.82A^2 - 8.10B^2 - 7.89C^2 \quad (9)$$

3. 3. 2. Graphical analysis of the inhibition efficiency of sweet potato leaf extract

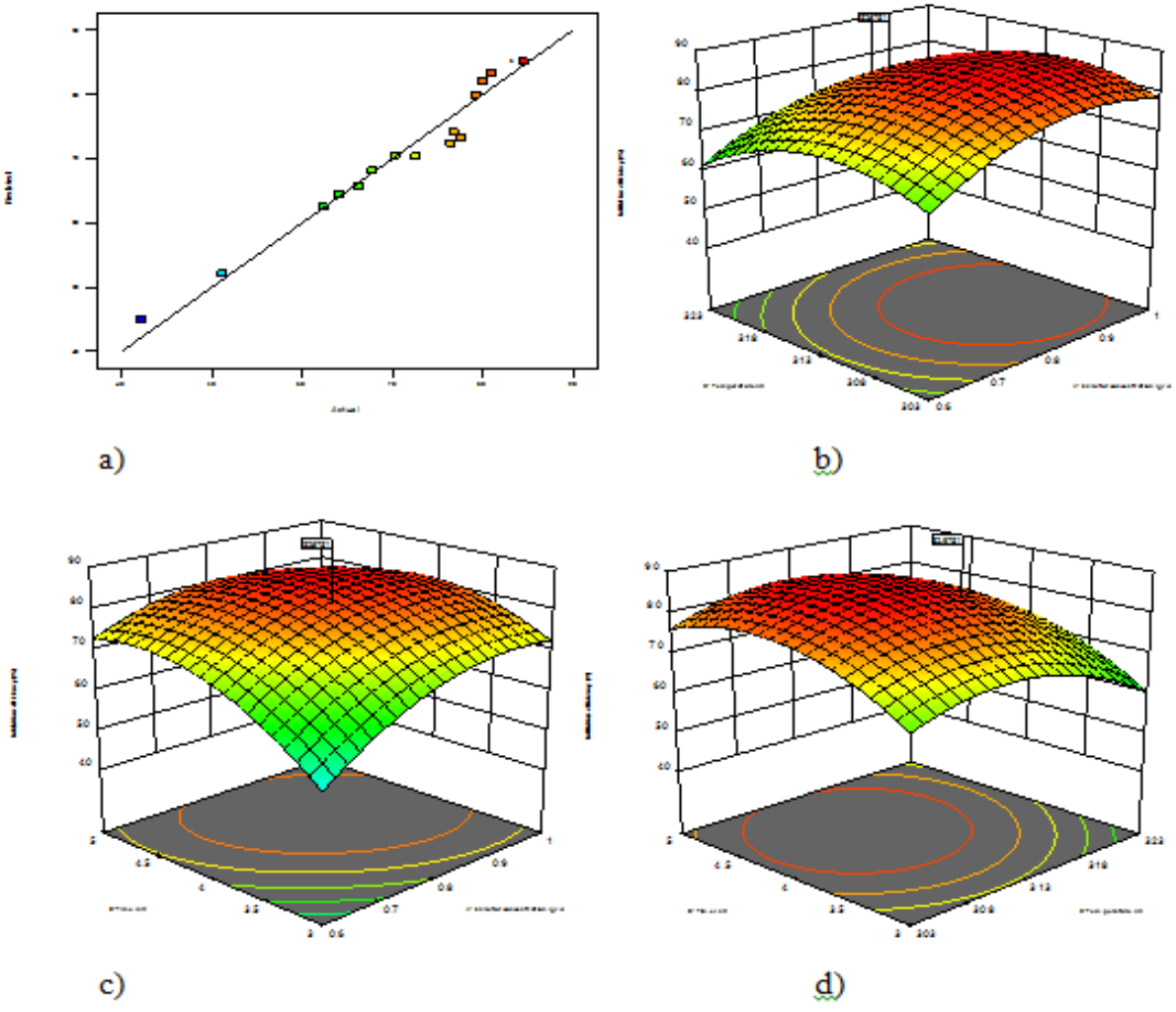

Figure 2. Graphical analysis of the inhibition efficiency (IE)
a) Predicted versus actual IE, b) IE versus inhibitor concentration and temperature, c) IE versus inhibitor concentration and time, d) IE versus temperature and time

Graphical analyses of the results for the mild steel in HCl are presented in Figures 2. In Figure 2a, the plot of predicted versus actual inhibition efficiency of inhibited displayed a linear (straight line) graph. The points clustered along the line of best fit. This is an indication that the generated model adequately described the efficiency of the corrosion control process. This observation is in line with previous research reports [19, 21]. The 3-D plots of Figures 2b - 2d are in parabolic form, typical of the quadratic model. The plots revealed optimum Inhibition efficiency of 82.58% was obtained at a time of 3.91hrs, at a temperature of 316.39 K, and at an inhibitor concentration of 0.80 g/L. The optimum inhibition efficiency was validated by comparing it with the experimental inhibition efficiency of 84.07% obtained at a time of 3.91hrs, at a temperature of 316.39 K, and at an inhibitor concentration of 0.80 g/L. The determined percentage deviation is less than the critical value of 5%. It confirmed that the model adequately described the relationship between the inhibition efficiency of sweet potato extract and the factors of time, inhibitor concentration, and temperature.

3. 4. Thermodynamic and adsorption properties

Result of the activation energy for the corrosion inhibition process is shown in Table 9. The value of the activation energy is less than the critical value of 80kJ/mol. It means that it is a physical adsorption process [17, 19]. Heat of adsorption value for the corrosion control process is presented in Table 10.

Table 9. Activation energy for corrosion inhibition process using watermelon leaf extract

T (K)	CR (mg/cm^{2}hr)	E_a (kJ/mol)
303	3.056	21.09
313	2.500	
323	4.444	
333	6.111	
343	6.667	

Table 10. Heat of adsorption for corrosion inhibition process using watermelon leaf extract

Inh. Conc. (g/L)	The heat of adsorption, Q_{ads} (J/mol)
0.2	-14041.3
0.4	-51961.4
0.6	-46123.0
0.8	-41763.0
1.0	-40736.9

The heat of adsorption is negative, which suggests that the adsorption of the molecules of the sweet potato on the mild steel is exothermic [17]. For the Langmuir adsorption isotherm, the graph of log(C/θ) versus log(C) is presented in Figures 6. The plot displaced linear graph, with a correlation coefficient close to one (1); revealed strong adherence to Langmuir isotherm. This agrees with the previous research reports [10, 19]. On the Temkin isotherm, plot of θ versus log C is presented in Figure 7. It revealed straight line graph, showing that the Temkin adsorption isotherm was obeyed. Considering the values of correlation coefficients (R^2), the Langmuir isotherm is a better model for the inhibition process because its R^2 is closest to1 (one). It agrees with reports of previous authors [16, 19].

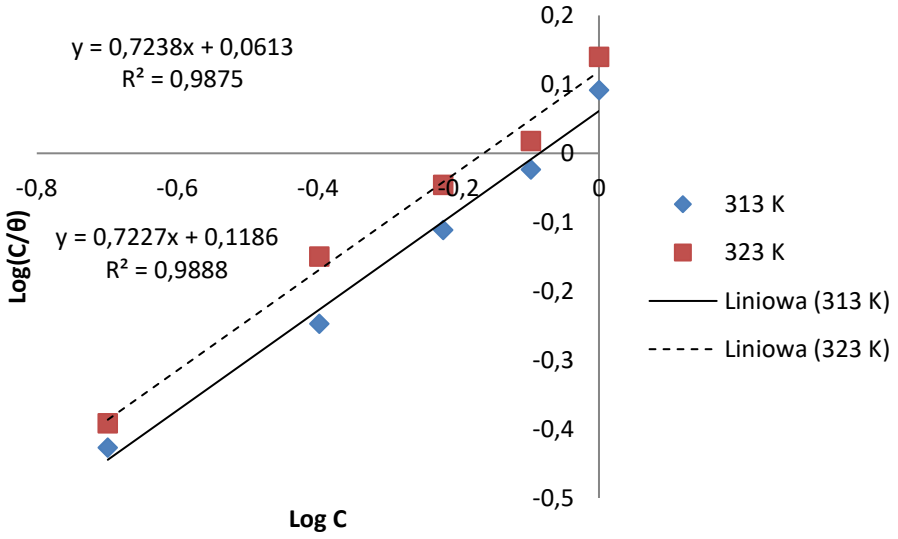

Figure 6. Langmuir isotherm plot

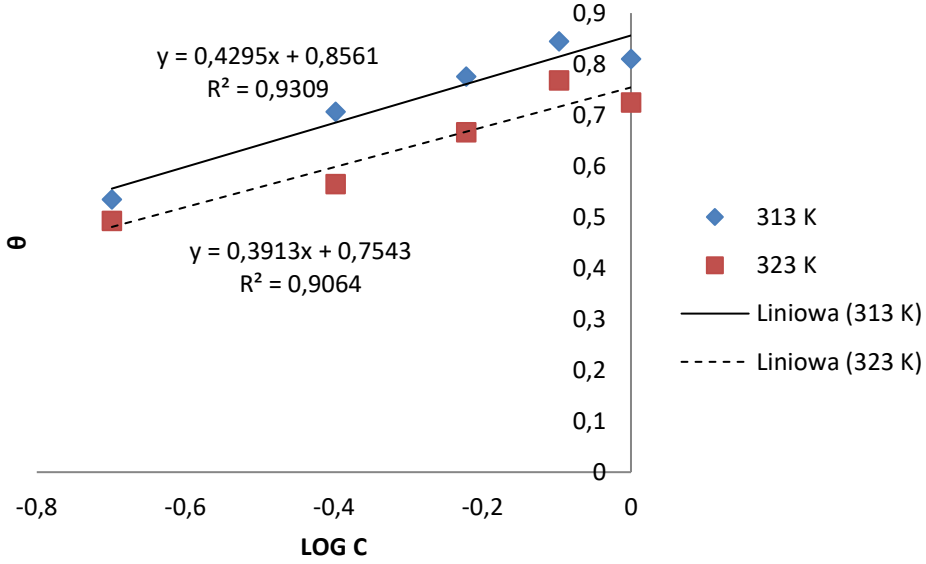

Figure 7. Temkim isotherm plot

4. CONCLUSIONS

From the analyses of the experimental results, the following conclusions were made:

i) Sweet potato leaf extract contains alkaloids and allied phytochemicals. The presence of the phytochemicals showed that the extract possesses corrosion inhibitive properties.

ii) The predominant functional groups of sweet potato leaf extract include ≡C-H stretch, C-H stretch, C-F stretch, N=O stretch, C-O stretch, C-H out of plane and C-H deformation

iii) Inhibitor concentration, temperature and time influenced the weight loss, corrosion rate, inhibition efficiency and degree of surface coverage.

iv) Quadratic model adequately expressed the relationship between inhibition efficiency and the inhibition process factors. Optimum Inhibition efficiency of 82.58% was obtained at time of 3.91hrs, temperature of 316.39 K, and inhibitor concentration of 0.80 g/L. Inhibitor of sweet potato leaf extract exhibited high inhibition efficiency. Hence, it should be used to inhibit corrosion of mild steel in HCl solution.

v) Thermodynamic and adsorption parameters revealed that the adsorption of the watermelon leaf extract on the mild steel surface is exothermic and occurred in accordance with physical adsorption mechanism.

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