https://doi.org/10.30799/jaec.052.18040102 J. Adv. Electrochem. - Volume 4 Issue 1 (2018) 158–161

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Journal of Advanced Electrochemistry

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ISSN: 2455-0205

Inhibition Efficiency of Eco-friendly Green Inhibitors (*Ocimum tenuiflorum* Phytocompounds) on Corrosion of High Carbon Steel in HCl Environment using Thermometric and Electrochemical Methods

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ARTICLE DETAILS

Article history:
Received 07 May 2018
Accepted 15 May 2018
Available online 16 May 2018

Keywords:
Polarization
Thermometric Method
Corrosion Rate
Impedance Spectroscopy

ABSTRACT

Corrosion inhibition behavior of crude alkaloids and flavonoids extracts of *Ocimum tenuiflorum* roots on the corrosion of high carbon steel in hydrogen chloride acid environment was studied using thermometric, electrochemical impedance spectroscopy and potentiodynamic polarization techniques. The phytocompounds showed excellent corrosion retardation ability on the high carbon steel metal in HCl acid environment. The alkaloids demonstrated a more reliable inhibition efficacy compared to flavonoids extracts with maximum inhibition efficiency of 98.1% and 93.6% respectively at a maximum concentration of 2.0 g/L. There was a noticeable increase in inhibition mitigation potential of the inhibitors with increasing concentration of extracts. A physical adsorption mechanism was recorded by the inhibitor and the Langmuir adsorption isotherm was well obeyed with correlation coefficient (R^2) at approximately 0.999. Results of electrochemical impedance spectroscopy and potentiodynamic polarization confirmed the strong adsorption of the inhibitors on the steel surface through the values of charge transfer resistance and corrosion current densities. The inhibitors proved to be stable ones on the metal and a spontaneous reaction process was recorded from the data derived from the Gibbs free energy of adsorption.

1. Introduction

Metals and alloys can have spontaneous failure resulting from the combined effects of corrosion and stress. Aside specific corrosion, even general corrosion leads to the reduction in the cross section of metal or alloy structure to the point where it can no longer support applied load. The consequences of corrosion are enormous ranging from economic, health and safety to environmental standpoints amongst others [1-3]. The major problem associated with carbon steel is its susceptibility to corrosion when it comes in contact with corrosive environments such as acids (HCl, H_2SO_4, and H_3PO_4), chloride rich solutions and aqueous hydrogen sulphide medium.

Corrosion mitigation strategies adopted by some industries include materials selection, coatings and linings, cathodic protection and the use of corrosion inhibitors amongst others. Corrosion control of metals is of technical, economic, environmental, and aesthetical importance. The use of corrosion inhibitors is the most practical and cost effective method in fighting corrosion.. Corrosion inhibitors retard corrosion by adsorbing onto the low carbon steel surface and blocking one or more of the electrochemical reactions occurring at the solution/metal interface. Well known corrosion inhibitors are organic compounds; these organic compounds typically contain nitrogen, sulfur and oxygen, and hydrophobic hydrocarbon chains in their structures [1].

The environmental toxicity of organic corrosion inhibitors has promoted the search for green corrosion inhibitors as they are biodegradable; do not contain heavy metals, or other toxic compounds. As in addition to being environmentally friendly and ecologically acceptable, plants products are inexpensive, readily available and renewable. Investigation of corrosion inhibiting abilities of tannins, alkaloids, organic, amino acids, and organic dyes of plant origin are of interest. Although substantial research has been devoted to corrosion inhibition by plant extracts, report on the detail mechanism of the adsorption process and identification of active ingredient are still scares [3-5].

However, inhibitors are usually effective only for a particular material in a certain environment, but the corrosion environments are highly variable; therefore, an inhibitor that works in one well may not work in another. Thus, it is necessary to continuously develop new formulas for different environment were numerous compounds are designed which gave rise to this research on inhibition efficiency of eco-friendly green inhibitors (*Ocimum tenuiflorum* phyto - compounds) on corrosion of high carbon steel in HCl environment [1, 4, 5].

2. Experimental Methods

2.1 Metal Preparation

The metal substrate utilized in the investigation was high carbon steel with carbon percentage of 98%. Prior to corrosion studies, the metal substrate was cut into coupons of dimensions 2.0 cm x 2.0 cm x 0.08 cm for thermometric measurements and 1 cm x 1 cm x 1 cm for electrochemical measurements. The carbon steel sheets were abraded using successive grades of silicon carbide paper (#120 – #800), degreased with ethanol to remove the residues arising from the abraded process, rinsed with acetone and air dried. Carbon steel coupons for electrochemical studies were completely insulated leaving one side of the steel surface (area ¼ 1 cm^2) exposed. The prepared metal substrates were stored in desiccator prior to use.

2.2 Pre-Preparation Extraction of Agrimonia eupatoria Sample

The fresh roots of *Ocimum tenuiflorum* obtained from an abandoned piece of land in Becheve, Ranch in Cross River were washed and cut into small cheeps and dried in oven at 60 °C. After drying, they were put through grinding to obtain a powdered sample. The powdered sample was extracted in methanol using soxhlet extraction process. The combination of crude root extract and methanol was placed in a water bath for 24 hours to evaporate the methanol leaving a paste (crude methanol extract) for alkaloid and flavonoid extraction.

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https://doi.org/10.30799/jaec.052.18040102

Cite this Article as: B.U. Ugi, M.E. Obeten, A.I. Ikeuba, Inhibition efficiency of eco-friendly green inhibitors (*Ocimum tenuiflorum* phytocompounds) on corrosion of high carbon steel in HCl environment using thermometric and electrochemical methods, J. Adv. Electrochem. 4(1) (2018) 158–161.

2.3 Preparation of Crude Flavonoid and Alkaloid Inhibitor Solutions

For alkaloid fraction, dilute hydrochloric acid (35.5% w/w) and ammonia solution was used. 35 g of the ethanol extract was partitioned between 100 cm^3 of chloroform and 100 cm^3 of 0.1 M HCl solution using a separating funnel. The HCl solution in the float fraction was carefully basify with ammonia solution and this was taken well above pH 7. Chloroform was immediately added into the basic solution in the separating funnel to obtain two nice layers with the lower one containing the alkaloids. The chloroform layer was eventually separated from mixture and put aside, the chloroform distilled off, and a small quantity of the crude alkaloids was obtained. 10 g of the alkaloid extract was soaked in 250 cm^3 of 0.5 M HCl solution and kept overnight. The solutions obtained were filtered and stored.

In another experiment, 30 g of the dried powdered sample was weighed into a beaker and extracted with 50 cm^3 of 80% methanol at room temperature for one hour. The solution was filtered through filter paper. The filtrate was evaporated to dryness over water bath at 60 °C. The weight of the dried extract was taken and the amount of flavonoid present was calculated. 10 g of flavonoids extract of *Ocimum tenuiflorum* was digested in 250 cm^3 of 1.0 M HCl solution. The resultant solution was kept for 24 hours and filtered. The stock solution (10 g/L) obtained was used in preparing test solution of different concentrations of the inhibitor; 0.1, 0.5, 0.7, 1.0 and 2.0 g/L. The test solutions for the inhibitor were prepared by serial dilution of the stock solution. The prepared solutions were then used to study the corrosion inhibition abilities of the extract.

2.4 Thermometric Measurements

100 cm^3 of the corrodent (1 M HCl) was introduced into a beaker. Thereafter, a carbon steel coupon of dimension 2.0 cm x 2.5 cm x 0.08 cm already weighed was dropped into the corrodent and the beaker immersed in a water bath. The volume of the hydrogen gas evolved from the corrosion reaction was regulated by temperature changes in the water bath. At different temperatures of 303 K, 318 K and 333 K, the metal was removed, degreased with ethanol, rinsed with acetone and air dried and then weighed. This procedure was repeated for a set of fresh coupons at different concentrations of *Ocimum tenuiflorum* root extracts (0.1, 0.5, 0.7, 1.0 and 2.0 g/L).

2.5 Electrochemical Determination

Electrochemical measurements were undertaken in a three-electrode cell using Gamry Instrument Potentiostat/Galvanostat/ZRA with a Gamry framework system based on ESA410. High carbon steel samples, graphite rod and silver/silver chloride (Ag/AgCl) were used as working, counter and reference electrodes, respectively. All the measurements were taken after the working electrode was immersed for 30 minutes in the different test solutions at room temperature in order to attain a steady-state open-circuit potential (OCP). The frequency range from 100 kHz to 0.01 Hz with amplitude of 10 mV was used in electrochemical impedance experiments. The potentiodynamic polarization curves were recorded from cathodic potential of -150 mV to anodic potential of +100 mV at a scan rate of 0.5 mV s^{-1} with respect to free corrosion potential (E_{corr}). The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential to obtain the corrosion current densities (icorr) and other electrochemical parameters of interest. Inhibition efficiency from electrochemical impedance spectroscopy (EIS), and potentiodynamic polarization (PDP) was computed using Eqs.(1) and (2) respectively.

$$IE_{EIS} = 1 - \left(\frac{R^0_{ct}}{R_{ct}}\right) x\ 100 \qquad (1)$$

where R^0_{ct} and R_{ct} are the charge transfer resistances in the absence and presence of the inhibitors respectively.

$$IE_{PDP} = 1 - \left(\frac{i_{corr}}{i^0_{corr}}\right) x\ 100 \qquad (2)$$

where i^0_{corr} and i_{corr} are the corrosion current densities in the absence and presence of inhibitor respectively.

3. Results and Discussion

3.1 Thermometric Results

The influence of temperature on the corrosion inhibitive behavior of the two tested inhibitors were investigated by thermometric method in the temperature range 303, 318 and 333 K. Results obtained as shown in Figs. 1-3 revealed that corrosion rate increased with increase in temperature in the absence and presence of the inhibitors but decreased with increased inhibitor concentration. On the other hand, inhibition efficiency of the two inhibitors decreased as temperature was increased and increase with inhibitor concentration. The apparent decrease in % IE as temperature was raised from 303, 318 and 333 K for AEOTR and FEOTR (Tables 1 and 2) could be related to change of adsorption mode from chemisorption to physisorption caused by desorption of adsorbed inhibitor as a result of increased solution agitation due to higher rate of hydrogen gas evolution [3, 6-10]. This is verified from the higher values of inhibition efficiency noticed at the lowest temperature (303 K) against the highest (333 K).

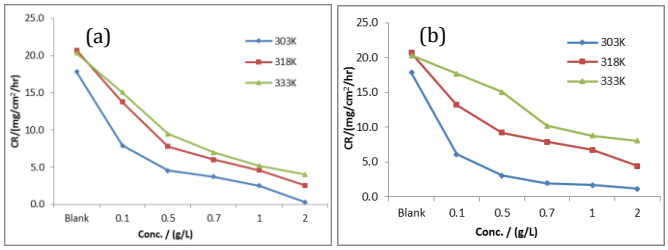

Fig. 1 Plots of corrosion rate of metal against concentration of (a) AEOTR and (b) FEOTR in HCl solution

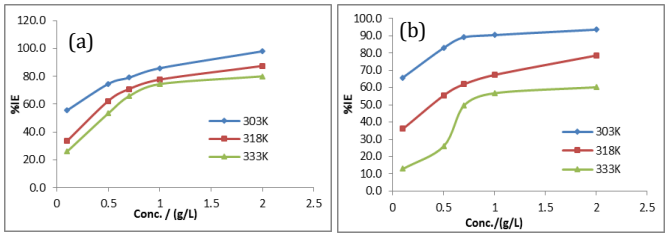

Fig. 2 Plots of inhibition efficiency of inhibitor against concentration of (a) AEOTR and (b) FEOTR in HCl solution

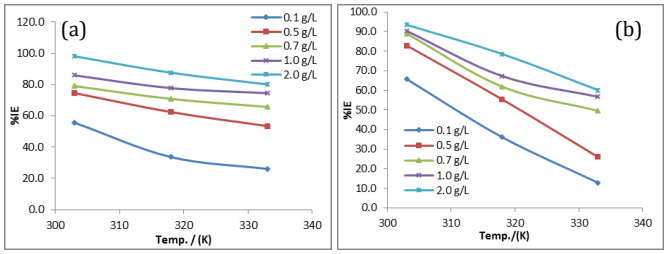

Fig. 3 Plots of inhibition efficiency of inhibitor against concentration of (a) AEOTR and (b) FEOTR in HCl solution

Table 1 Results of corrosion rate of metal, surface coverage and inhibition efficiency of inhibitor (AEOTR) in HCl

Conc.	CR (mg/cm²/hr)			θ			%IE		
(g/L)	303 K	318 K	333 K	303 K	318 K	333 K	303 K	318 K	333 K
Blank	17.839	20.716	20.326	-	-	-	-	-	-
0.1	7.919	13.752	15.042	0.556	0.336	0.260	55.6	33.6	26.0
0.5	4.532	7.790	9.467	0.746	0.624	0.534	74.6	62.4	53.4
0.7	3.734	6.037	6.978	0.791	0.709	0.657	79.1	70.9	65.7
1.0	2.516	4.616	5.187	0.859	0.777	0.745	85.9	77.7	74.5
2.0	0.338	2.575	4.044	0.981	0.876	0.801	98.1	87.6	80.1

Table 2 Results of corrosion rate of metal, surface coverage and inhibition efficiency of inhibitor (FEOTR) in HCl

Conc.	CR (mg/cm²/hr)			θ			%IE		
(g/L)	303 K	318 K	333 K	303 K	318 K	333 K	303 K	318 K	333 K
Blank	17.84	20.72	20.33	-	-	-	-	-	-
0.1	6.105	13.24	17.71	0.658	0.361	0.129	65.8	36.1	12.9
0.5	3.049	9.243	15.04	0.829	0.554	0.260	82.9	55.4	26.0
0.7	1.956	7.901	10.25	0.890	0.619	0.496	89.0	61.9	49.6
1.0	1.721	6.781	8.801	0.904	0.673	0.567	90.4	67.3	56.7
2.0	1.163	4.443	8.098	0.935	0.786	0.602	93.5	78.6	60.2

3.2 EIS Results

Figs. 4(a) and (b) show the electrochemical impedance plots for AEOTR and FEOTR respectively. Electrochemical impedance parameters produced from the Nyquist plots are presented in Table 3. From Table 3, it is clearly seen that the charge transfer resistance exhibits an increasing trend with increasing AEOTR and FEOTR concentration up to 2.0 g/L within the range of concentration studied. Similar observation has been

https://doi.org/10.30799/jaec.052.18040102

reported earlier [11-15] and other workers which was attributed to an increase in heterogeneity resulting from the adsorption of the inhibitors on the steel surface.

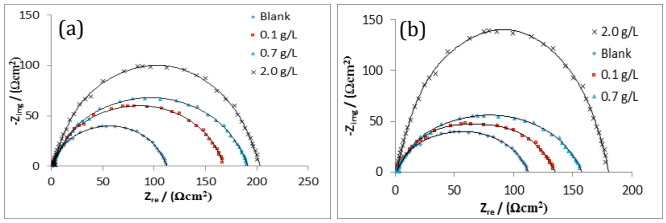

Fig. 4 Nyquist plots of showing effect of (a) AEOTR and (b) FEOTR on high carbon steel corrosion in HCl

Table 3 Data obtained from the Nyquist plots of AEOTR and FEOTR on high carbon steel corrosion in HCl

	Conc. (g/L)	Rct (Ωcm^{-2})	IE %
	Blank (1 M HCl)	115	-
AEOTR	0.1	177	40.0
	0.7	198	72.2
	2.0	220	91.3
FEOTR	0.1	148	28.7
	0.7	169	47.0
	2.0	180	56.5

3.3 PDP Results

Electrochemical parameters obtained from the analysis of the polarization curves (Figs. 5(a) and (b)) using extrapolation method are displayed in Table 4. It is clear from the table that corrosion current density decreased while the inhibition efficiency increased with increase in concentration of AEOTR and FEOTR concentration up to 2.0 g/L. A reduction in corrosion current density and an increase in inhibition efficiency is clearly visible from Table 4. This result is in agreement with that of thermometric measurements. These observations are indicative of the fact that addition of AEOTR and FEOTR to 1 M HCl solution has reduced the anodic dissolution and the cathodic hydrogen evolution reaction of stainless steel in the acid medium, hence functioning as mixed type corrosion inhibitors [1, 5, 14-17].

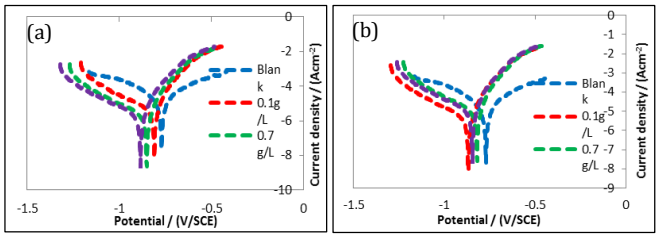

Fig. 5 Arrhenius plots of showing effect of (a) AEOTR and (b) FEOTR on high carbon steel corrosion in HCl

Table 4 Data obtained from the potentiodynamic plots of AEOTR and FEOTR on high carbon steel corrosion in HCl

	Conc. (g/L)	I_{corr} (mAcm^{-2})	IEi (%)
	Blank (1 M HCl)	1.355	-
AEOTR	0.1	0.811	40.2
	0.7	0.524	61.3
	2.0	0.087	93.6
FEOTR	0.1	0.925	31.7
	0.7	0.647	52.3
	2.0	0.325	76.1

3.4 Thermodynamic Properties

The temperature of the system was varied across the inhibitor concentrations from which the activation energy for the corrosion of high carbon steel in solutions of HCl was evaluated using the Arrhenius equation given by Eq.(3) [11-15].

$$\ln R_c = \ln A - \frac{E_o}{RT} \qquad (3)$$

where Rc is the corrosion rate, Ea is the apparent effective activation energy, R is the general gas constant, and A is the Arrhenius pre-exponential factor [10, 14-19]. Calculated values of activation energy between 303 K, 318 K and 333 K were obtained from the slope of Fig. 6 and presented in Table 5. The values obtained are greater than the value obtained for the blank solution indicating that alkaloid and flavonoid extracts of *Ocimum tenuiflorum* leaves retards the corrosion of high carbon steel in HCl solution [1-3, 5, 15-21]. Since the activation energy which is the energy required to oxidize metal is increased with inhibitor concentration, it implies that more energy has to be supplied to the system for the corrosion to take place, thus the observed decrease in corrosion rate. The values are also consistent with the data expected for the mechanism of physical adsorption (<80 kJmol^{-1}) [3-7, 22-25].

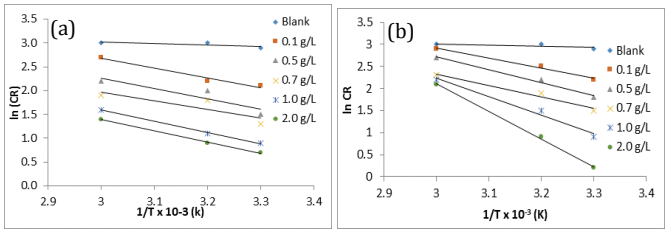

Fig. 6 Arrhenius plots showing effect of (a) AEOTR and (b) FEOTR on high carbon steel on high carbon steel corrosion in HCl

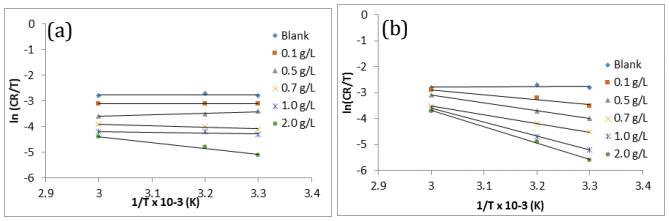

Fig. 7 Transition state plots showing effect of (a) AEOTR and (b) FEOTR on high carbon steel on high carbon steel corrosion in HCl

Thermodynamic parameters; Enthalpy and ΔH_{ads} , Entropy ΔS_{ads} of alkaloid and flavonoid extracts of *Ocimum tenuiflorum* leaves on high carbon steel were obtained from Figs. 7(a) and (b) respectively and calculated using Eq.(4) (transition state equation) [8 -15, 23]

$$\frac{CR}{T} = log\ \frac{R}{Nh} + \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303RT}\log \qquad (4)$$

From Eq.(4), values of log (C_R/T) were plotted against 1/T as shown in Fig. 5 and from the slope and intercept of the plot, values of enthalpy and entropy of adsorption were calculated as shown in Table 5. From the calculated values of ΔH_{ads} (Table 5), it can be deduced that the adsorption of the inhibitor on high carbon steel surface is exothermic and the reaction becomes less exothermic with increase in inhibitor concentration [1, 5 – 11, 17, 21-25]. The negative values for ΔS_{ads} shows the non-spontaneous dissolution of the high carbon steel and the increase in its value suggests decrease in disordering in the rate determining step, hence increase stability of inhibitors on steel surface [5, 11, 19-20].

Table 5 Data obtained from the Arrhenius and transition state plots of AEOTR and FEOTR on high carbon steel corrosion in HCl

Conc. (g/L)	AEOTR			FEOTR		
	Ea kJmol^{-1}	ΔH kJmol^{-1}	ΔS kjmol^{-1}	Ea kJmol^{-1}	ΔH kJmol^{-1}	ΔS kjmol^{-1}
Blank	62.86	71.41	-119.73	62.860	71.41	-119.73
0.1	25.12	64.29	-112.61	42.140	62.86	-111.18
0.5	23.57	43.28	-91.60	29.290	53.57	-101.89
0.7	21.43	38.11	-86.43	28.570	33.57	-81.89
1.0	20.71	28.57	-76.89	25.710	30.00	-78.32
2.0	17.86	22.86	-71.18	22.860	19.29	-67.32

3.5 Adsorption Isotherm Investigations

The values obtained were theoretically fitted into Langmuir adsorption isotherm model and the correlation coefficient (R^2) value was used to select the best fit isotherm. Langmuir isotherm was found to be one of the best fit adsorption isotherm model to describe the adsorption of both AEOTR and FEOTR onto the high carbon steel surface. Langmuir isotherm is characterized by the following expression [15, 26]:

$$\frac{C}{\theta} = \frac{1}{Kads} + C \qquad (5)$$

where C is the inhibitor concentration and Kads is the equilibrium constant of the adsorption–desorption process. Figs. 8(a) and (b) presents the plot of C/θ against C using θ values obtained for thermometric technique at various temperatures. In all cases, linear plots were obtained

https://doi.org/10.30799/jaec.052.18040102

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and the R^2 values are close to one (Table 6) suggesting that the adsorption of AEOTR and FEOTR molecules onto the carbon steel surface obeys Langmuir isotherm [16, 22, 26-29]. Although the plots are linear with good correlation coefficient values, the slopes are more than unity indicating a deviation from ideal Langmuir adsorption equation. This suggests that there is an interaction between adsorbed species of the inhibitor molecules on the metal surface [1, 15]. From the values of Kads, the standard free energy of adsorption for both AEOTR and FEOTR at different temperatures were obtained as follows:

$$\Delta Gads = -RTln(55.5Kads) \quad (6)$$

where R is the universal constant, T is the absolute temperature and 55.5 is the concentration of water molecules expressed in mg/L. The computed values of ΔG^0_{ads} listed in Table 3 are less negative than -40 kJmol^{-1} and more negative than -20 kJmol^{-1}. In the literature, the value of ΔG^0_{ads} equal to -20 kJmol^{-1} or less is taken to indicate physisorption involving electrostatic interaction between charged molecules whereas those in the order of -40 kJmol^{-1} or more is interpreted as chemisorption involving charge sharing or transfer from the inhibitors to the metal surface to form a kind of co-ordinate bond [11, 5, 15, 29-32]. From the values of ΔG^0_{ads} obtained in this present work, it can be deduced that adsorption mechanism of both inhibitors on high carbon steel surface may involve physisorption [14-26]. As pointed out by Umoren [1] and Okafor [15] a consequence of strong adsorption of water molecules on the steel surface, it may be assumed that adsorption occurs due to the physical forces.

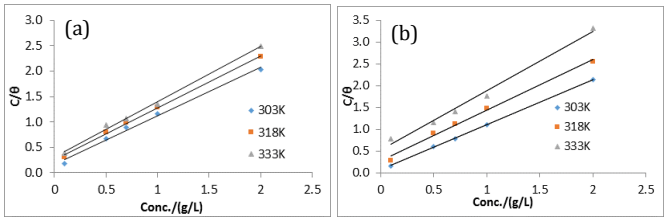

Fig. 8 Langmuir plots showing effect of (a) AEOTR and (b) FEOTR on high carbon steel on high carbon steel corrosion in HCl

Table 6 Data obtained from the Langmuir plots of AEOTR and FEOTR on high carbon steel corrosion in 1 M HCl

	AEOTR				FEOTR			
Temp. (K)	k (g/L)	R^2	Slope	ΔGads (kJ/mol)	k (g/L)	R^2	Slope	ΔGads (kJ/mol)
303	6.127	0.9930	0.9588	-14.68	7.974	0.9997	1.0407	-15.35
318	4.055	0.9976	1.0289	-14.32	3.766	0.9921	1.1666	-14.12
333	3.239	0.9955	1.0889	-14.37	1.950	0.9897	1.3659	-12.97

4. Conclusion

From the results obtained, the following conclusions could be drawn: The two extracted compounds (alkaloid and flavonoid) acted as corrosion inhibitors for high carbon steel in 1 M HCl but with AEOTL showing superior performance. Corrosion inhibition performance is found to depend on the concentration of the inhibitors and temperature. Gradual increase in amount of inhibitor extracts improves the inhibition efficiency of the two inhibitors due to the strong adsorption of inhibitor molecules and the on the steel surface. Results of potentiodynamic polarization studies revealed that the two inhibitors functions as a mixed type corrosion inhibitor. Result of electrochemical impedance spectroscopy revealed increase charge transfer resistance values depicting reduction in corrosion attack. Corrosion inhibition occurs by virtue of adsorption of the inhibitor molecules on the steel surface which was found to accord with Langmuir adsorption isotherm model.

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https://doi.org/10.30799/jaec.052.18040102

Cite this Article as: B.U. Ugi, M.E. Obeten, A.I. Ikeuba, Inhibition efficiency of eco-friendly green inhibitors (*Ocimum tenuiflorum* phytocompounds) on corrosion of high carbon steel in HCl environment using thermometric and electrochemical methods, J. Adv. Electrochem. 4(1) (2018) 158–161.