



Management of aqueous corrosion of federated mild steel (Local constructional steel) at elevated temperatures employing environmentally friendly inhibitors: *Matricaria chamomilla* plant

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Abstract

Management of aqueous corrosion of federated mild steel at elevated temperatures employing environmentally friendly inhibitors of *Matricaria chamomilla* plant was carried out at temperatures of 318 K, 333 K and 347 K in 0.5 M H₂SO₄ solution. Thermometric, potentiodynamic polarization and electrochemical impedance spectroscopic measurements were adopted for the study. In all, both inhibitors demonstrated an appreciable result at the tested temperatures with the alkaloid extract of *Matricaria chamomilla* plant (AEMCP) showing a better inhibition of 99 % compared to Non alkaloid extract of *Matricaria chamomilla* plant (NAEMCP) 95 %, implying that the hetero-compounds in the alkaloid inhibitor has a stronger adsorption compared to non alkaloids. The values of the charge transfer resistance are seen to be increasing with increasing concentration of both inhibitors which reveals the strong adsorption of the inhibitors on the metal surface. The lower corrosion current density (I_{corr}) values in the presence of inhibitors without causing significant changes in corrosion potential (E_{corr}) suggested a mixed type inhibitor for both extracts. The ΔH_{ads} results obtained indicated that the inhibition reaction is endothermic which suggested that they inhibitors adsorbed heat from the surrounding to form more and stronger bonds with the mild steel. Meanwhile, values of ΔS_{ads} were negative indicating the absence of disorderliness. Hence, there was association of the inhibitor molecules. The ΔG_{ads} values followed a physical adsorption process as values are below -20 kJ/mol and the negative values indicated a more stabled and spontaneous reaction process. The reaction obeyed the Frunkim and El-Awady adsorption isotherms as their correlation coefficient was 0.99, an indication that the data fit the isotherms.

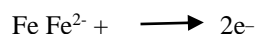
Keywords: El-Awady *et al*, adsorption, thermometric, federated steel, *Matricaria chamomilla*, polarization, hydrogen tetraoxosulphate (VI) acid

1. Introduction

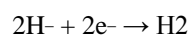
Corrosion is a process that involves chemical reactions occurring at the interface between a metal and its environment leading to the dissolution of metal atoms from metallic surfaces^[1]. One of the key factors in any corrosion situation is the environment. The definition and characteristics of this variable can be quite complex. But for practical situations, it is important to realize that the environment is a variable that can change with time and conditions. It is also important to realize that the environment that actually affects a metal corresponds to the micro environmental conditions that this metal really "sees," i.e., the local environment at the surface of the metal. It is indeed the reactivity of this local environment that will determine the real corrosion damage. Metals and alloys can have spontaneous failure resulting from the combined effects of corrosion and stress^[1, 2]. 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.

Since steels and other iron-based alloys are the metallic materials most commonly exposed to water, aqueous corrosion will be studied with a special focus on the reactions of steel^[1, 3]. Metal ions go into solution at anodic areas in an amount chemically equivalent to the reaction at cathodic

areas (Fig. 1.1). In the cases of iron-based alloys, the following reaction usually takes place at anodic areas:



This reaction is rapid in most media, as shown by the lack of pronounced polarization when iron is made an anode employing an external current. When iron corrodes, the rate is usually controlled by the cathodic reaction, which in general is much slower (cathodic control)^[4, 5]:



The chamomile flowering plant has a long tradition of using as an important herb. The plant contains a number of volatile oils including bisabolol, matricin, bisabolol A and Bisabolol B. Its blue oil consists of sesquiterpene derivatives (75 – 90%) but only traces of monoterpenes. The oil contains up to 20% polyynes. The principal components are ϵ - β -farnesene (4.9 – 8.1%), terpene alcohol (farnesol), chamazulene (2.3 – 10.9%), α -bisabolol (4.8 – 11.3%) according to study of Singh *et al.*,^[6]. Due to the limited studies in the usage of this plant's hetero compounds in corrosion studies especially on federated mild steel, this study therefore centers on the application of synthesized extracts of *Matricaria chamomilla* plant on the management of corrosion of federated mild steel in hydrogen tetraoxosulphate (IV) acid environment.

2. Material and Methods

2.1 Metal composition and preparation

Federation steel has the highest hardness value of 126.3 HV and impact value of 149 HV with an ultimate tensile and yield strength of 799.49 MPa and 660 MPa, respectively. The steel which was commercially obtained has the following chemical composition (wt%): C (0.266), Si (0.164), S (0.018), P (0.018), Mn (0.637), Ni (0.026), Cr (0.025), Mo (0.502), V (0.001), Cu (0.220) and Fe (98.123) according to the study of Alo *et al.*,^[7] in his assessment of imported and local constructional steel in Nigeria: Analysis by One – Way ANOVA. The metal was resized to 3cm x 1.5cm x 0.08cm for thermometric analysis and 1 x 1 cm² samples for the electrochemical measurements. The metals were then polished, washed with ethanol, rinsed in acetone and air dried. The aggressive acidic solution of 0.5M H₂SO₄ solutions was of analar grade.

2.2 Pre-extraction/stock solution preparation of *Matricaria chamomilla* plant

The initial stage in studying corrosion using plant is the preparation of plant samples to preserve the biomolecules in the plant prior to extraction. The fresh *Matricaria chamomilla* leaves obtained from an uncultivated land in Betereko, Boki LGA, Cross River State were washed and cut into small cheeps and dried in an oven at 50° C. After the drying of the leaves, they were put through grinding to obtain a powdered sample. This is because lowering particle size increases surfaces contact between samples and extraction solvents. Conventional mortar and pestle was used for grinding, followed by the use of electric blender to reduce particle size of sample. The grind sample was further extracted using the hot method (shoxlet extractor) with ethanol as the extracting solvent for 3 days. Evaporation of the crude extract was carried out to remove the solvent content using a water bath. The crude extract, the stock solutions were prepared.

2.3 Inhibitor preparation

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 cm³ of chloroform and 100 cm³ 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 cm³ of 0.5 M HCl solution and kept overnight. The solutions obtained were filtered and stored.

2.4 Thermometric measurements

100 cm³ of the corrodent (1 M HCl) was introduced into a

beaker. Thereafter, a carbon steel coupon of dimension 3cm x 1.5cm x 0.08cm 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 318 K, 333 K and 347 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 5 g/L, 10 g/L, 15 g/L, 20 g/L and 25 g/L concentrations of *Matricaria chamomilla* extracts.

2.5 Electrochemical determination

Electrochemical measurements were undertaken in a three-electrode cell. Federated mild 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 (i_{corr}) and other electrochemical parameters of interest.

3. Results

3.1 Thermometric analysis result

In order to evaluate the effect of increasing temperature on the inhibitors at various concentrations, thermometric analysis was employed. The data describing the effect of temperature has been shown in Tables 1 – 2 for alkaloids and non alkaloid inhibitors. The tables revealed the higher adherence of the inhibitors on the metal surface especially at lower temperatures. This is a strong indication of a physical adsorption mechanism^[1, 5, 8]. Both inhibitors demonstrated good adsorption phenomena on the metal surface even at higher temperatures of 347K which shows that a chemical mechanism may be attended with time if the process continued. There is an increase in adsorption with corresponding increase in inhibitor concentration meaning that the corrosion rate of the metal reduces with increase molecule on the metal surface^[3-4, 9]. Values of inhibition efficiency are gradually decreasing with increase temperature indicating desorption process due to strong agitation on the metal/solution interface as temperature increase^[4, 10 – 11]. In all, the AEMCP show a better inhibition of 99% compared to NAEMCP with 95 % which implies that the hetero-compounds in the alkaloid inhibitor has a stronger adsorption than the non alkaloids^[1].

Table 1: Data revealing the corrosion rate of metal, surface coverage and inhibition efficiency of AEMCP in 0.5 M H₂SO₄ solution at various temperatures effects

Inhibitor Conc. (g/L)	CR (mg/cm ² /hr)			θ			%IE		
	318K	333K	347K	318K	333K	347K	318K	333K	347K
Control	0.0370	0.0910	1.6200	-	-	-	-	-	-
5 g/L	0.0110	0.0210	0.7190	0.703	0.769	0.556	70.27	76.92	55.62
10 g/L	0.0067	0.0196	0.5251	0.819	0.785	0.676	81.89	78.46	67.59
15 g/L	0.0023	0.0121	0.3320	0.938	0.867	0.795	93.78	86.70	79.51
20 g/L	0.0013	0.0093	0.1954	0.965	0.898	0.879	96.49	89.78	87.94
25 g/L	0.0002	0.0066	0.1741	0.995	0.927	0.893	99.46	92.75	89.25

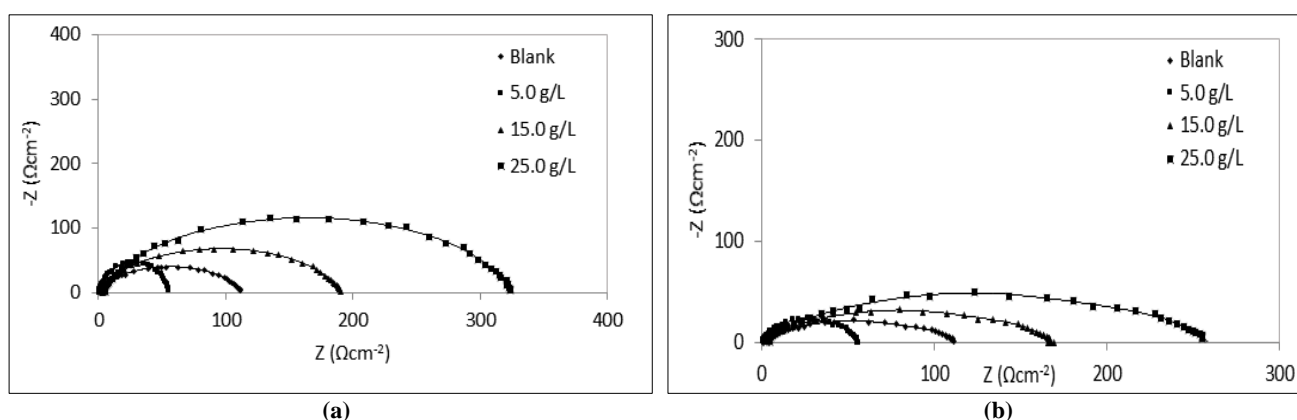
Table 2: Data revealing the corrosion rate of metal, surface coverage and inhibition efficiency of NAEMCP in 0.5 M H₂SO₄ solution at various temperatures effects

Inhibitor Conc. (g/L)	CR (mg/cm ² /hr)			θ			%IE		
	318K	333K	347K	318K	333K	347K	318K	333K	347K
Control	0.0370	0.0910	1.6200	-	-	-	-	-	-
5 g/L	0.013	0.035	0.723	0.641	0.611	0.554	64.054	61.099	55.370
10 g/L	0.011	0.031	0.573	0.697	0.659	0.646	69.730	65.934	64.630
15 g/L	0.009	0.026	0.482	0.751	0.713	0.702	75.135	71.319	70.247
20 g/L	0.005	0.022	0.439	0.873	0.763	0.729	87.297	76.264	72.901
25 g/L	0.002	0.011	0.318	0.951	0.875	0.804	95.135	87.473	80.370

3.2 Electrochemical impedance results

Figs. 1a – b are representative Nyquist plots obtained from the corrosion inhibition of (a) alkaloids and (b) Non alkaloid extracts of *Matricaria chamomilla* Plant on federated mild

steel in 0.5 M H₂SO₄ environment. They are perfect semicircles and this was attributed to charge transfer reaction [1, 10].

**Fig 1:** Graphical representation of the corrosion inhibition of (a) alkaloids and (b) Non alkaloid extracts of *Matricaria chamomilla* Plant on federated steel in 0.5 M H₂SO₄ environment using Nyquist plots

The impedance readings for the various Impedance parameters derived from Nyquist plots are tabulated in Table 3. It is observed that the various double layer capacitance values are decreasing with increase inhibitor concentration, which can result from an increase in thickness of electrical double layer, suggesting that the inhibitor molecules function

by adsorption at the metal- solution interface [11 – 15]. The values of the charge transfer resistance are seen to be increasing with increasing concentration of both inhibitors which reveals the strong adsorption of the inhibitors on the metal surface [4, 9, 15]. This is in agreement to the results obtained from the thermometric experimentation.

Table 3: Theoretical analysis result from the Nyquist plots of the corrosion inhibition of *Matricaria chamomilla* Plant extracts on federated steel in 0.5 M H₂SO₄ environment.

	Conc. (g/L)	R _{ct} (Ωcm ²)	C _{dl} (μFcm ⁻²)	Inhibition Efficiency
	Control (0.5 M H ₂ SO ₄)	51	0.0069	-
AEMCP	5.0 g/L	124	0.0052	58.9 %
	15.0 g/L	199	0.0039	74.4 %
	25.0g/L	341	0.0011	85.0 %
NAEMCP	5.0 g/L	111	0.0028	55.1 %
	15.0 g/L	169	0.0015	69.8 %
	25.0g/L	276	0.0007	81.5 %

AEMCP – Alkaloid extracts of *Matricaria chamomilla* Plant. NAEMCP – Non alkaloid extracts of *Matricaria chamomilla* Plant

R_c – charge transfer resistance. C_{dl} – Double layer capacitance

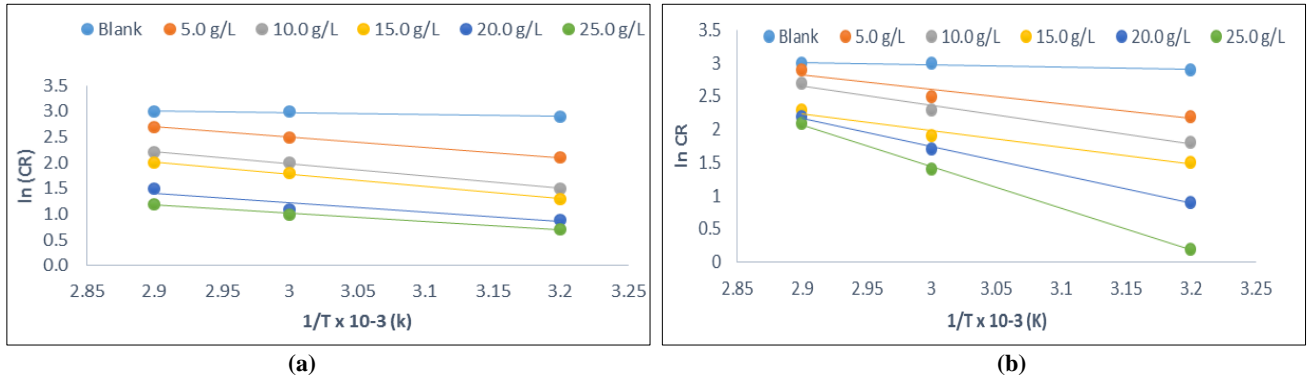


Fig 3: Plots of the Arrhenius equation for the determination of energy of activation of the adsorption of (a) AEMCP and (b) NAEMCP on federated mild steel in 0.5 M H₂SO₄.

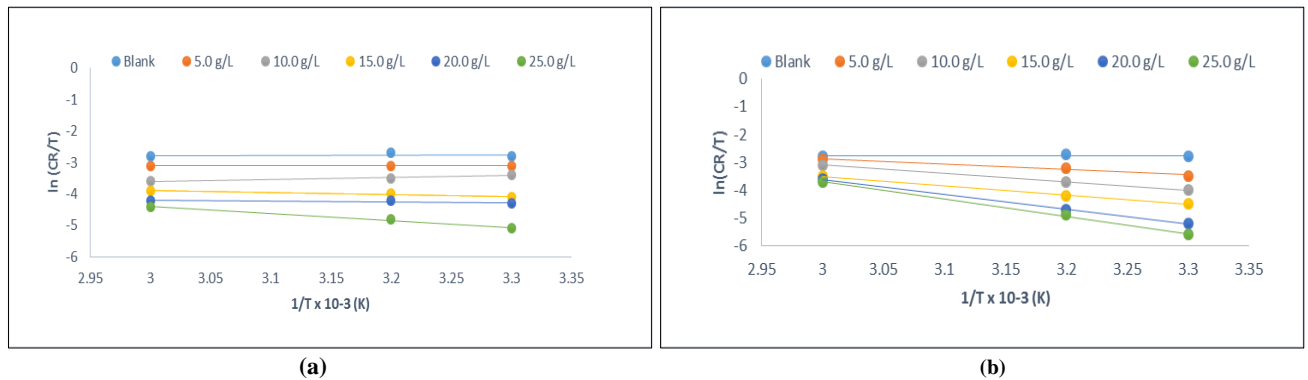


Fig 4: Plots of the Transition state equation for the determination of enthalpy and entropy of adsorption of (a) AEMCP and (b) NAEMCP on federated mild steel in 0.5 M H₂SO₄.

Temperature effect on federated mild steel corrosion with and without extract concentration was investigated using the Arrhenius equation 2. The Arrhenius equation gives the relationship between the corrosion rate and the minimum energy needed before adsorption is activated (activation energy). Generally, activation values for the blank were lower than those obtained for the inhibited system, which indicated that adsorption is strongly activated [9]. It was also

realized that there was increasing Ea with increase extract concentration which also indicated that there is increasing strength of adsorption as the concentration of the respective inhibitors increases [9, 16]. Finally the low activation energies obtained in this work suggest the influence of diffusion of the inhibitor’s molecule unto the surface of the metal. Taking a closer look on the result, it is obvious that it is in agreement with phytical adsorption mechanism (<20 kJ/mol)

Table 5: Thermodynamic data showing the activation energy, enthalpy and entropy of adsorption of the inhibitor/metal interaction.

Conc. (g/L)	AEMCP			NAEMCP		
	Ea kJmol-1	ΔH kJmol-1	ΔS kJmol-1	Ea kJmol-1	ΔH kJmol-1	ΔS kJmol-1
Control	8.43	35.20	-59.36	31.43	35.20	-59.36
5.0 g/L + 0.5 M H ₂ SO ₄	10.36	32.14	-66.31	21.07	31.43	-55.09
10.0 g/L 0.5 M H ₂ SO ₄	11.21	22.14	-46.30	15.15	26.23	-50.45
15.0 g/L 0.5 M H ₂ SO ₄	12.23	16.07	-43.21	14.27	16.22	-40.45
20.0 g/L 0.5 M H ₂ SO ₄	13.06	14.26	-38.25	13.35	15.01	-39.16
25.0 g/L 0.5 M H ₂ SO ₄	31.443	11.43	-35.09	11.43	19.15	-34.16

AEMCP – Alkaloid extracts of *Matricaria chamomilla* Plant. NAEMCP – Non alkaloid extracts of *Matricaria chamomilla* Plant

E_a – Energy of activation. ΔH_{ads} – Enthalpy of adsorption. ΔS_{ads} – Entropy of adsorption

3.5 Adsorption studies of the inhibitors

The Frumkin model has a parameter that account for the interaction between metal and inhibitor of all molecules (Dada *et al.*, 2012; Sudhish and Ebenso, 2011). This molecular interaction parameter was determined from the slope of the plot log (Θ/(1-Θ))/C vs. C (g/L) (Figs. 5a – b) according to Equation 3:

$$\ln (\Theta / (1-\Theta)) / C = \ln K_{ads} + 2a\theta \dots\dots\dots 3$$

where, θ is surface coverage, ‘a’ is interaction parameter,

K_{ads} is the equilibrium constant and C is the extract concentration. The molecular interactive parameter can be positive or negative. Positive values suggest attraction forces between adsorbed molecules while negative values suggest repulsive forces between the adsorbed molecules. Results from Table 6 indicate a strong attractive force between the metal/inhibitor interfaces. Decreasing values of the equilibrium constant (k) with increasing temperature suggest a physical adsorption due to the gradual desorption of the inhibitor molecules which is in agreement with earlier results [1, 9, 17]. The correlation coefficient values suggested that the

reaction obeyed the Frumkin isotherm with $R^2 = 0.99$. The Gibbs free energy values follows a physical adsorption

process as values are below -20 kJ/mol, the negative values indicate a more stable and spontaneous reaction process [18].

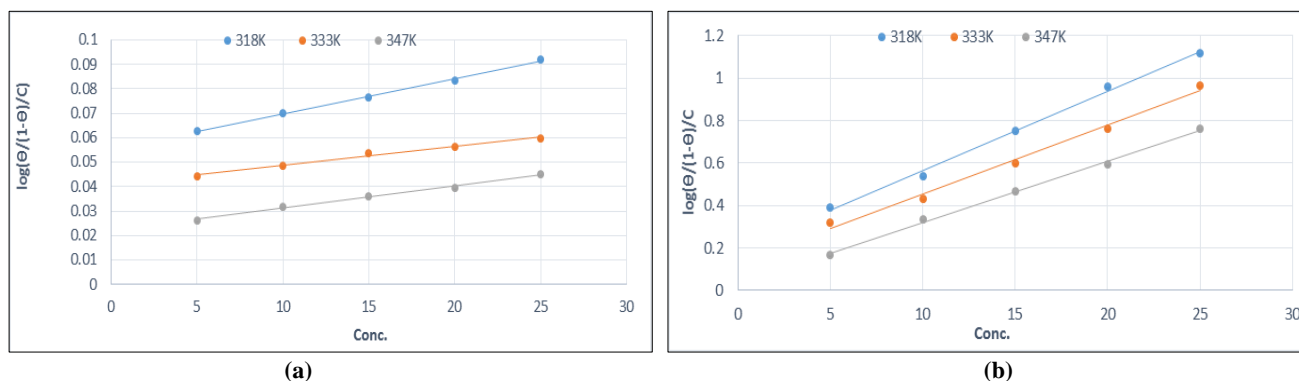


Fig 5: Frumkin adsorption isotherm plots for the adsorption of (a) AEMCP and (b) NAEMCP on federated mild steel in 0.5 M H₂SO₄.

Table 6: Frumkin adsorption isotherm data for the adsorption of (a) AEMCP and (b) NAEMCP on federated mild steel in 0.5 M H₂SO₄.

Temp. (K)	AEMCP					NAEMCP				
	k (g/L)	R2	Slope	a	ΔG _{ads} (kJ/mol)	k (g/L)	R2	Slope	a	ΔG _{ads} (kJ/mol)
318	0.0555	0.9978	0.0014	71.429	-2.9744	0.1915	0.9965	0.0374	26.738	-6.2488
333	0.0408	0.9906	0.0008	125.011	-2.2628	0.1305	0.9909	0.0324	30.864	-5.4818
347	0.0223	0.9935	0.0009	111.111	-0.6151	0.0325	0.9973	0.0289	34.602	-1.7017

ΔG_{ads} – Gibbs free energy of adsorption. a – molecular interaction parameter. R2 – Correlation coefficient. k – Equilibrium constant

The El-Awady *et al.* [19] adsorption isotherm (equation 4) is an isotherm that provide information on site of adsorption with respect to number of an inhibitor occupied. The assumptions establishing the El-Awady isotherm can be written according to Equation 4.

$$\log(\theta/(1-\theta)) = \log B + y \log C \dots\dots\dots 4$$

where, C is the extract concentration in the bulk solution, θ is

the extent of surface coverage of the extract, B is binding constant of adsorption process; and y is the occupancy parameter. From Equation 4, a plot of $\log \theta/(1-\theta)$ against $\log C$ should be linear with slope and intercept equal to y and $\log K$ respectively. The curve shows straight lines which is an indication that the isotherm is well fitted by the data (Figs. 6).

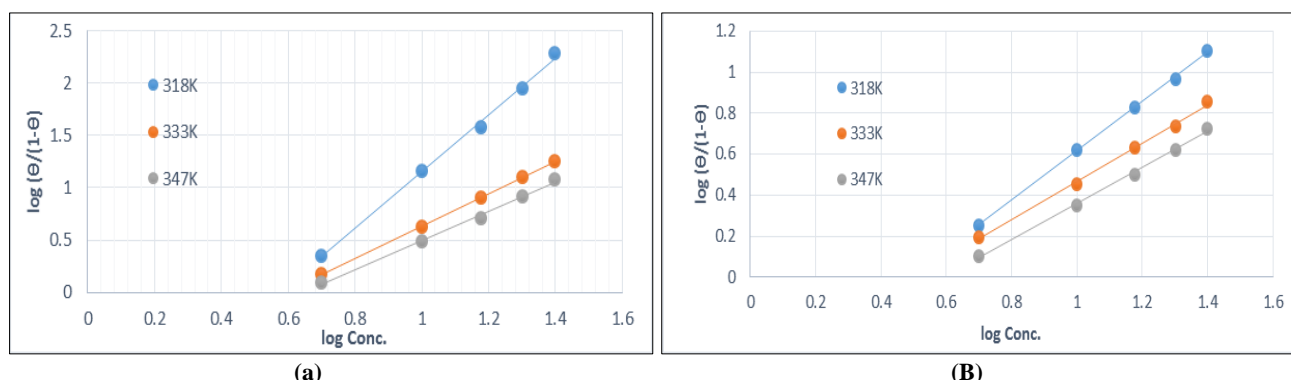


Fig 6: El – Awady *et al* isotherm for the adsorption of (a) AEMCP and (b) NAEMCP on federated mild steel in 0.5 M H₂SO₄.

Values of y (size parameter) less than one infer a weak multilayer or monolayer of adsorption, while y value greater than unity means a stronger multilayer adsorption [19]. The values of y obtained from the El-awady *et al.* (1992) isotherm are listed in Table 7.

It is evident from Tables7 that the values of y are nearly equal to unity, showing a weak monolayer adsorption which points towards physisorption mechanism and also agrees with the thermometric analysis data on percentage inhibition efficiency.

Table 7: El – Awady *et al* adsorption isotherm data for the adsorption of (a) AEMCP and (b) NAEMCP on federated mild steel in 0.5 M H₂SO₄

Temp. (K)	AEMCP					NAEMCP				
	Binding constant (B) (g/L)	R2	Slope	y	ΔG _{ads} (kJ/mol)	Binding constant (B) (g/L)	R2	Slope	y	Δ Gads (kJ/mol)
318	1.5553	0.9976	2.7131	0.3686	-11.786	0.5853	0.9993	1.2002	0.8332	-9.203
333	0.9069	0.9999	1.5448	0.6473	-10.849	0.4659	0.9971	0.9320	1.0730	-9.005
347	0.8952	0.9960	1.3967	0.7160	-11.268	0.5266	0.9977	0.8838	1.1325	-9.737

Δ Gads – Gibbs free energy of adsorption. y – Size parameter. R² – Correlation coefficient.

4. Conclusion

The following findings were arrived at after a successful completion of the study:

1. Federated mild steel can be effectively managed through the use of hetero compounds of environmentally friendly herbal plant like *Matricaria chamomilla*. This was verified from the inhibition efficiency results of 99.46 % and 95.13 % respectively.
2. Even at higher temperatures, both inhibitors showed a significant inhibition of corrosion of federated mild steel with inhibition efficiency of 89.25 and 80.37 % for AEMCP and NAEMCP, respectively at 347K.
3. The alkaloid part of the plant shows a better inhibitor compared to the non-alkaloid part. This may be related to the strong molecular attraction of hetero-atoms to the metal surface compared to the non-alkaloids.
4. Thermodynamic data of the inhibitor revealed a stable inhibitor, spontaneity in the forward direction and endothermic reaction process giving rise to formation of strong bonds between inhibitor molecules and metal.
5. The electrochemical data revealed the strong inhibitor capacity noticed from the increasing values of the charge transfer resistance and decreasing values of the corrosion current density.
6. The inhibitors are mixed types but with inhibition impact at the anodic reaction giving rise to oxidation (corrosion) reduction.
7. The inhibitors obeyed the Temkin and El-Awady *et al* adsorption isotherms as correlation coefficient was approximately unity (0.99).

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