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ORIGINAL PAPER

Joint effect of halides and ethanol extract of *Lasianthera africana* on inhibition of corrosion of mild steel in H_2SO_4

N. O. Eddy · S. A. Odoemelam · A. O. Odiongenyi

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Abstract Inhibitive and adsorption properties of ethanol extract of *Lasianthera africana* for inhibition of corrosion of mild steel in H_2SO_4 were studied using gravimetric, thermometric, gasometric, and infrared (IR) methods. The extract was found to be a good inhibitor of corrosion of mild steel in H_2SO_4. Inhibitive properties of the extract were attributed to enhancement in adsorption of the inhibitor on mild-steel surface by saponin, alkaloid, tannin, flavanoid, cardiac glycoside, and anthraquinone (present in the extract). Also, adsorption of the inhibitor was found to be exothermic, spontaneous, and consistent with assumptions of Langmuir and Temkin adsorption isotherms. Synergistic study revealed that, of the investigated halides, only KCl may enhance adsorption of the inhibitor, whereas KBr and KI antagonized its adsorption. Based on the decrease in efficiency of the inhibitor with temperature, with values of activation energy and free energy of adsorption below the threshold values of −40 and 80 kJ mol^{-1}, respectively, a physical adsorption mechanism has been proposed for adsorption of ethanol extract of *Lasianthera africana* on the surface of mild steel.

Keyword Corrosion · Inhibition · *Lasianthera africana* · Synergism

N. O. Eddy (✉)
Department of Chemistry, Ahmadu Bello University, Zaria, Kaduna State, Nigeria
e-mail: nabukeddy@yahoo.com

S. A. Odoemelam · A. O. Odiongenyi
Department of Chemistry, Micheal Okpara University of Agriculture, Umudike, Abia State, Nigeria

1 Introduction

Industrial processes such as prickling and acid cleaning often involve contact between a metal and aggressive solution, requiring the use of an inhibitor (Eddy and Ebenso 2008). Most effective inhibitors are organic compounds containing electronegative functional groups and π electrons in triple or conjugated double bonds [19–22, 35]. These compounds also have heteroatoms (such as N, P, O, and S) and aromatic rings in their structure, which are the major adsorption centers [1–4, 23].

In view of this, several inhibitors have been synthesized and used successfully to inhibit corrosion of metals, including mild steel. However, the major problem associated with most of these inhibitors is that they are not ecofriendly as they contain heavy metals and other toxic compounds (Eddy and Ebenso 2008). "Green" corrosion inhibitors are biodegradable and do not contain toxic substances [9, 26, 30]. Most plants have been successfully used as corrosion inhibitors, but studies on the use of ethanol extract of *Lasianthera africana* as an inhibitor for the corrosion of mild steel is scant. This plant is mostly used as a vegetable in Nigeria. It is commonly found along riversides and is believed to be rich in vitamins and other nutrients. The medicinal value of this plant has also been advocated by some researchers. According to Udosen et al. [29], the ash, crude fat, and crude protein of young *Lasianthera africana* are approximately 12, 7.0, and 21 mg 100 g^{-1} dry matter (DM) respectively, while those of old leaves are approximately 11, 6.0, and 19 mg 100 g $^{-1}$ DM, respectively. The focus of our present study is to investigate inhibitive and adsorption properties of the ethanol extract of *Lasianthera africana* on corrosion of mild steel in H_2SO_4.

2 Experimental

2.1 Materials

Materials used for the study were mild-steel sheet of composition (wt%) Mn (0.6), P (0.36), C (0.15), and Si (0.03) and the rest Fe. The sheet was mechanically press-cut to form coupons, each of dimensions $5 \times 4 \times 0.11$ cm^3. Each coupon was degreased by washing with ethanol, dried in acetone, and preserved in a desiccator. All reagents (H_2SO_4, KI, KCl, and KBr) used for the study were analar grade, and double-distilled water was used for their preparation. Concentrations of H_2SO_4 used for gasometric, thermometric and weight-loss studies were 2.5, 2.5, and 0.1 M, respectively, while the concentration of the halides (KI, KCl, and KBr) was 0.06 M.

2.2 Extraction of plants

Samples of *Lasianthera africana* were obtained from the Modern Botanical Garden, located at Chubb Road, Ikot Ekpene, Southeast Nigeria. Leaves were dried, ground, and soaked in a solution of ethanol for 48 h, after which the samples were cooled and filtered. The filtrates were further subjected to evaporation at 352 K in order to leave the sample free of the ethanol. Stock solutions of the extract so obtained were used in preparing different concentrations of the extract by dissolving 0.1, 0.2, 0.3, 0.4, or 0.5 g of extract in 1 L of 2.5 and 0.1 M H_2SO_4, respectively.

2.3 Chemical analysis

Phytochemical analysis of the ethanol and aqueous extract of the sample was carried out according to the method reported by Udosen et al. [29]. For the identification of saponin, frothing and Na_2CO_3 tests were adopted. For the identification of tannin, bromine water and ferric chloride tests were used. For the identification of cardiac glycosides, Leberman's and Salkowski's tests were adopted. For the identification of alkaloid, Dragendorff, Hagger, and Meyer reagents were used.

IR analysis of ethanol extract of extracts and corrosion products were carried out using spectrophotometer (BUCK model, 500 M infrared). The sample was prepared using Nujol oil and analysis was done by scanning the sample through the wavenumber range 400–4,000 cm^{-1}.

2.4 Gasometric method

Gasometric methods were carried out at 303 and 333 K as described in the literature [10, 12, 20]. From the volume of hydrogen evolved per minute, inhibition efficiency (%*I*), degree of surface coverage (θ), and corrosion rate were calculated using Eqs. 1–3, respectively.

$$\%I = \left(1 - \frac{V^1_{Ht}}{V^o_{Ht}}\right) \times 100 \quad (1)$$

$$\theta = \%I/100 = 1 - \frac{V^1_{Ht}}{V^0_{Ht}} \quad (2)$$

$$\mathrm{CR} = \left(V^0_{Ht} - V^1_{Ht}\right)/\mathrm{t} \quad (3)$$

where V^1_{Ht}, is the volume of H_2 gas evolved at time *t* for inhibited solution and V^0_{Ht} for uninhibited solution. CR is the corrosion rate in cm^3 min^{-1}.

2.5 Thermometric method

Thermometric analysis was carried out as reported elsewhere [13, 14, 24]. From the rise in temperature of the system per minute, the reaction number (RN) was calculated using Eq. 3:

$$\mathrm{RN}\left({}^\circ\mathrm{Cmin}^{-1}\right) = \frac{T_\mathrm{m} - T_\mathrm{i}}{t}, \quad (4)$$

where T_m and T_i are the maximum and initial temperatures, respectively, and *t* is the time (in minutes) taken to reach the maximum temperature. The inhibition efficiency (%*I*) was evaluated from percentage reduction in the reaction number, namely:

$$\%I = \frac{\mathrm{RN_{aq}} - \mathrm{RN_{wi}}}{\mathrm{RN_{aq}}} \times 100, \quad (5)$$

where $\mathrm{RN_{aq}}$ is the reaction number in the absence of inhibitors (blank solution) and $\mathrm{RN_{wi}}$ is the reaction number with 2 M H_2SO_4 containing the studied inhibitors.

2.6 Gravimetric analysis

A previously weighed metal (mild steel) was completely immersed in 250 mL test solution in an open beaker. The beaker was inserted into a water bath maintained at temperature of 30°C. The weight of the sample before immersion was measured using SCALTEC high-precision balance (model SPB31). Every 24 h each sample was removed from the test solution, washed in distilled water containing 50% NaOH and 100 g L^{-1} zinc dust, and dried in acetone before reweighing.

Difference in weight after a period of 168 h was taken as total weight loss. Inhibition efficiency (%*I*) for each inhibitor was calculated using the formula

$$\%I = (1 - W_1/W_2) \times 100, \quad (6)$$

where W_1 and W_2 are the weight losses (g dm^{-3}) for mild steel in the presence and absence of inhibitor in H_2SO_4

solution, respectively. The degree of surface coverage θ is given by Eq. 7

$$\theta = 1 - W_1/W_2. \quad (7)$$

3 Results and discussions

Figure 1 shows the variation of volume of hydrogen gas evolved with time for the corrosion of mild steel in 2.5 M H_2SO_4 containing various concentrations of ethanol extract of *Lasianthera africana* at 303 and 333 K, respectively. From Fig. 1 it was observed that the rate of corrosion of mild steel increased with increasing temperature but decreased with increasing concentration of ethanol extract of *Lasianthera africana*, indicating that ethanol extract of *Lasianthera africana* inhibited corrosion of mild steel in H_2SO_4 and that inhibition efficiency of the extract was affected by temperature and its concentration. Figure 2 shows the variation of weight loss of mild steel with time for the corrosion of mild steel in 0.1 M H_2SO_4 containing various concentrations of ethanol extract of *Lasianthera africana*. From Fig. 2, it was also found that the rate of corrosion of mild steel increased with increasing temperature and with duration of contact. However, the rate decreased with increasing concentration of ethanol extract of *Lasianthera africana*.

Values of inhibition efficiencies of ethanol extract of *Lasianthera africana* obtained from gasometric, thermometric, and gravimetric methods are presented in Table 1. The results revealed that the inhibition efficiency of ethanol

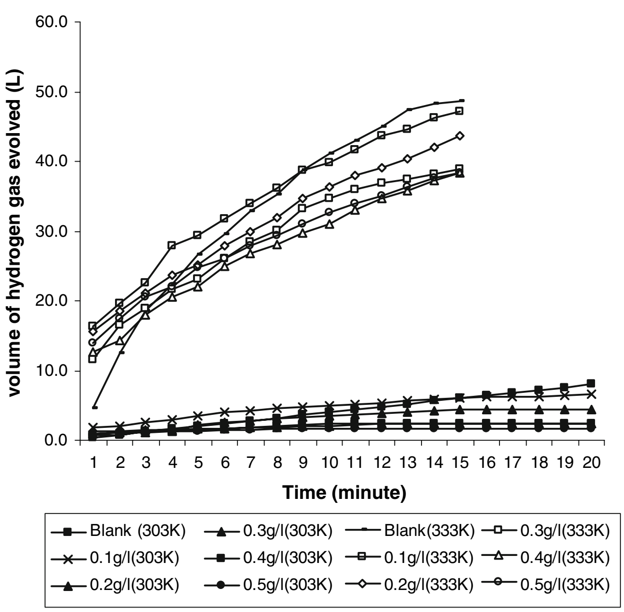


Fig. 1 Variation of the volume of hydrogen gas evolved with time for the corrosion of mild steel in tetraoxosulfate (VI) acid containing various concentrations of ethanol extract of *Lasianthera africana* at 303 and 333 K

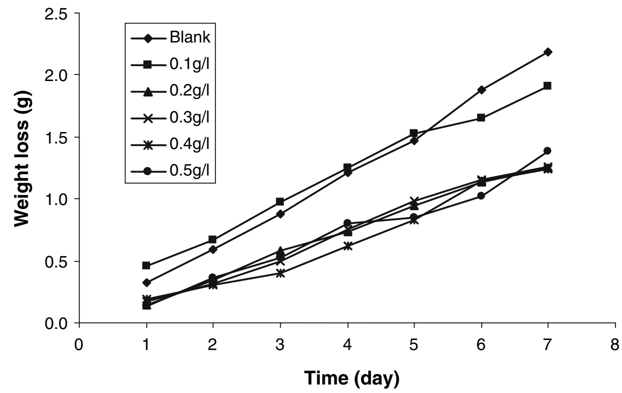


Fig. 2 Variation of weight loss with time for the corrosion of mild steel in tetraoxosulfate (VI) containing various concentrations of ethanol extract of *Lasianthera africana at* 303 K

extract of *Lasianthera africana* increased with increasing concentration of the extract but decreased with increasing temperature, indicating that ethanol extract of *Lasianthera africana* is an adsorption inhibitor for corrosion of mild steel and that the mechanism of adsorption of the inhibitor is physical adsorption (Eddy and Ebenso 2008). Values of inhibition efficiencies obtained from weight-loss measurements were higher than values obtained from gasometric and thermometric measurements. This is attributed to the fact that the weight-loss method measures the average value of the corrosion rate whereas the gasometric and thermometric methods measure instantaneous values of the corrosion rate. However, these data correlated strongly with those obtained from the weight-loss method ($r = 0.9757$ and 0.8424 for thermometric and gasometric data, respectively). In the presence of halides (KI, KBr, and KCl), the inhibition efficiencies of *Lasianthera africana* (Table 1) were altered, indicating that adsorption of inhibitor on the surface of mild steel is affected by presence of halides [31–34].

Data obtained from weight-loss measurements were used to fit curves for different orders of reaction, revealing that corrosion of mild steel in H_2SO_4 containing various concentrations of ethanol extract of *Lasianthera africana* is first order and is consistent with Eq. 8 [7, 16, 27].

$$-\log(\text{weight loss}) = k_1 t/2.303, \quad (8)$$

where k_1 is the rate constant and t is the time (in days). Plots of –log(weight loss) versus time were linear (not shown). Also values of k_1 (Table 2) calculated for various concentrations of *Lasianthera africana* were constant (0.3), indicating that the assumptions of the rate model are valid for the inhibited corrosion reaction of mild steel in H_2SO_4. For a first-order reaction, the half-life ($t_{1/2}$) is dependent on the rate constant (i.e., $t_{1/2} = 0.693/k_1$), implying that the values of $t_{1/2}$ (Table 2) for the various concentrations of the extract were also constant (2 days). These values are

Table 1 Inhibition efficiencies of various concentrations of ethanol extract of *Lasianthera africana* for corrosion of mild steel in H_2SO_4

C (g L^{-1})	WL	T	G		Extract + 0.06 M		
			303 K	333 K	KBr (WL)	KI (WL)	KCl (WL)
0.1	85.58	34.55	38.16	27.66	18.88	6.77	37.13
0.2	87.50	43.22	52.63	30.38	27.76	9.05	40.70
0.3	87.50	46.34	80.26	32.42	42.43	9.92	43.90
0.4	91.99	54.22	84.21	36.51	47.10	20.76	46.23
0.5	94.23	63.84	92.11	39.00	57.29	23.05	46.50

WL, results from weight-loss method; *T*, results from thermometric method; *G*, results from gasometric method

Table 2 Kinetic and thermodynamic parameters for adsorption of ethanol extract of *Lasianthera africana* on mild-steel surface

C (g dm^{-3})	E_a (kJ g^{-1})	Q_{ads} (J mol^{-1})	k_1	$t_{1/2}$ (days)	R^2
0.1	61.67	−10.04	0.3	2	0.9820
0.2	68.06	−19.60	0.3	2	0.9306
0.3	71.74	−44.83	0.3	2	0.9447
0.4	76.25	−46.72	0.3	2	0.8920
0.5	79.53	−60.93	0.3	2	0.9660

greater than the value of 0.14 days obtained for the blank, indicating that the inhibitor extends the half-life of mild-steel corrosion by 2 days.

The logarithmic form of the Arrhenius equation (Eq. 9) was used to calculate the activation energies for the corrosion of mild steel in H_2SO_4 containing various concentrations of ethanol extract of *Lasianthera africana* [11, 15, 28]

$$\log(\mathrm{CR}_2/\mathrm{CR}_1) = E_a/2.303R(1/T_1 - 1/T_2), \quad (9)$$

where CR_2 and CR_1 are the corrosion rates at temperatures T_2 (333 K) and T_1 (303 K), respectively, E_a is the activation energy, and R is the gas constant. Values of E_a calculated from Eq. 9 are recorded in Table 2. These values increase as the concentration of the inhibitor increases, indicating that there is increasing ease of surface coverage accompanied by increasing adsorption as the concentration of the inhibitor increases [6]. Also E_a values are below the threshold value of 80 kJ mol^{-1} required for chemical adsorption, indicating that the mechanism of adsorption of ethanol extract of *Lasianthera africana* is physical adsorption.

The heat of adsorption of ethanol extract of *Lasianthera africana* on the surface of mild steel was calculated using Eq. 10 (Eddy and Ebenso 2008).

$$Q_{ads} = 2.303R[\log(\theta_2/1-\theta_2) - \log(\theta_1/1-\theta_1)] \times (T_1 \times T_2)/(T_2 - T_1), \quad (10)$$

where θ_2 and θ_1 are the degrees of surface coverage at the temperatures T_2 (333 K) and T_1 (303 K), respectively, and R is the gas constant. Values of Q_{ads} calculated from Eq. 10 are recorded in Table 2. These values are negative, indicating that the adsorption of ethanol extract of *Lasianthera africana* on the surface of mild steel is exothermic.

The adsorption characteristics of ethanol extract of *Lasianthera africana* on the surface of mild steel were further investigated by fitting curves for different adsorption models. The isotherms that best described the adsorption behavior of ethanol extract of *Lasianthera africana* on the surface of mild steel were Langmuir and Temkin adsorption isotherms.

Langmuir adsorption isotherm can be expressed according to Eq. 11

$$C/\theta = 1/K + C, \quad (11)$$

where K is the adsorption equilibrium constant and θ is the degree of surface coverage of the inhibitor [5, 8]. Taking the logarithm of Eq. 11 yields Eq. 12

$$\log(C/\theta) = \log C - \log K. \quad (12)$$

Figure 3 shows Langmuir isotherm for the adsorption of ethanol extract of *Lasianthera africana* on the surface of mild steel. Values of adsorption parameters deduced from Langmuir adsorption isotherm are recorded in Table 3. The application of Langmuir adsorption isotherm to the adsorption of this inhibitor indicates that there is no interaction between the adsorbate and the adsorbent [12].

According to Temkin adsorption isotherm, the degree of surface coverage (θ) is related to the concentration of inhibitor (C) in the bulk electrolyte according to Eq. 13 [16]:

$$\exp(-2a\theta) = KC, \quad (13)$$

where K_{ads} is the equilibrium constant of adsorption. Rearranging and taking logarithm of Eqs. 13, one obtains Eq. 14.

$$\theta = \frac{-\ln * \kappa}{2a} - \frac{\ln * \Psi}{2a}. \quad (14)$$

The plots of θ versus log C were linear, implying that the assumptions of Temkin isotherm are valid. Figure 4 shows Temkin isotherm for the adsorption of ethanol extract of *Lasianthera africana* on surface of mild steel. The

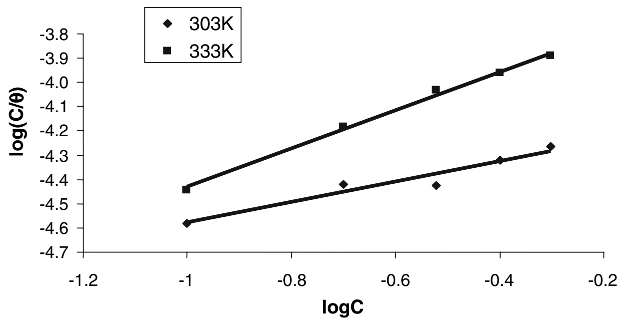


Fig. 3 Langmuir isotherm for the adsorption of ethanol extract of *Lasianthera africana* on the surface of mild steel

Table 3 Langmuir and Temkin isotherm parameters for the adsorption of ethanol extract of *Lasianthera africana* on surface of mild steel

Temperature (K)	Log K	Slope	ΔG_{ads} (kJ mol^{-1})	R^2
Langmuir				
303	0.1571	0.4220	−11.01	0.9981
313	0.3591	0.7891	−13.38	0.9954
Temperature (K)	Log K	a	ΔG_{ads} (kJ mol^{-1})	R^2
Temkin				
303	1.1696	0.615082	−16.88	0.9542
313	0.4247	3.148615	−13.80	0.9162

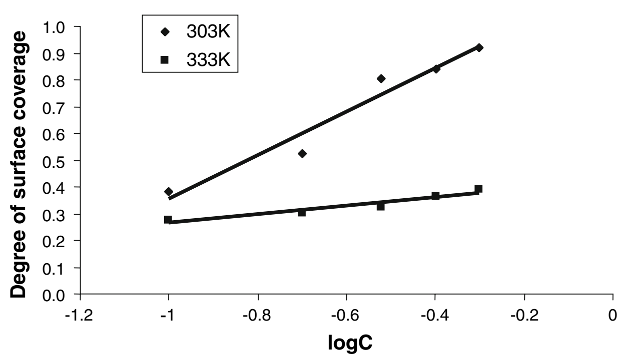


Fig. 4 Temkin isotherm for the adsorption of ethanol extract of *Lasianthera africana* on surface of mild steel

adsorption parameters deduced from Temkin isotherm are also presented in Table 3. From the results obtained, it is seen that values of K decreased with increasing temperature. Generally, larger values of K imply more efficient adsorption and hence better adsorption of ethanol extract of *Lasianthera africana* at lower temperature.

Free energies of adsorption of ethanol extract of *Lasianthera africana* on the surface of mild steel were calculated using Eq. 15:

$$\Delta G_{ads} = -2.303\ RT\log\left(55.5\ K\right), \tag{15}$$

where ΔG_{ads} is the free energy of adsorption, R is the gas constant, T is the temperature, and K is the equilibrium constant deduced from adsorption plots. Calculated values of ΔG_{ads} are recorded in Table 3. These values are negative and less than −40 kJ mol^{-1}, indicating that adsorption of ethanol extract of *Lasianthera africana* on the surface of mild steel is spontaneous and occurred according to the mechanism of physical adsorption.

Table 4 Phytochemical constituents of ethanol and aqueous extract of *Lasianthera africana*

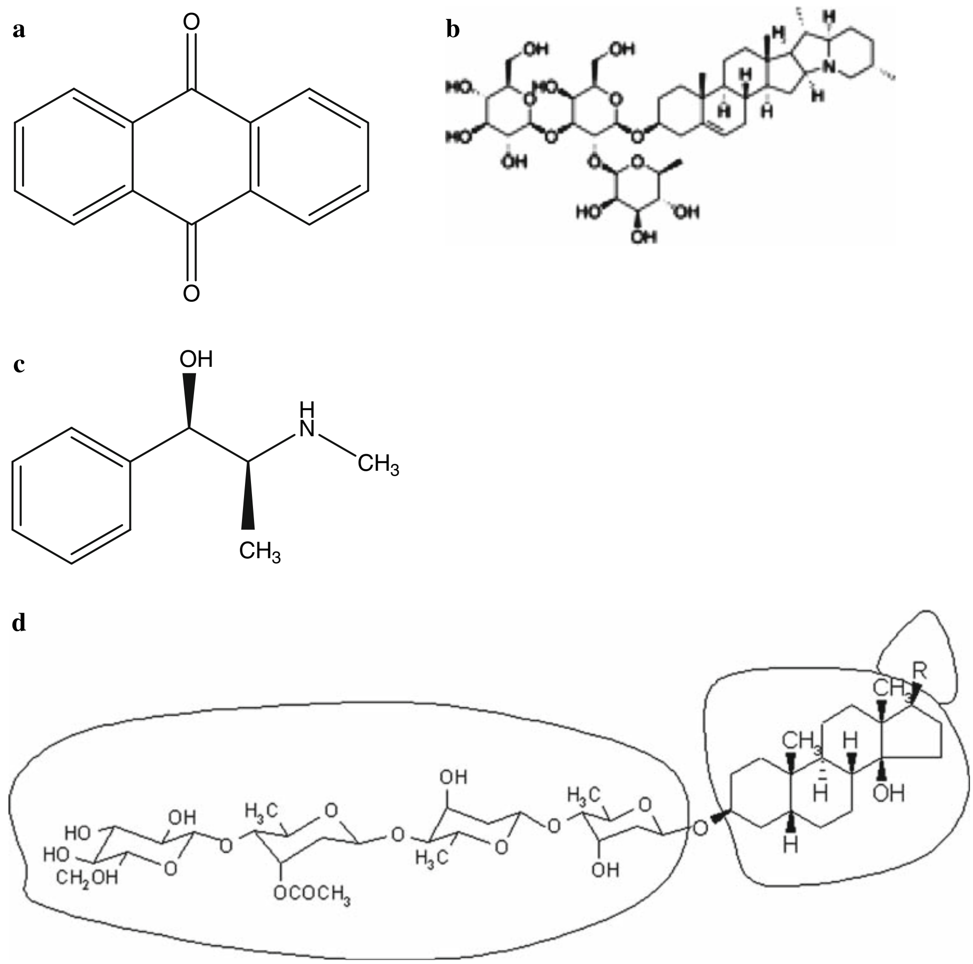
Phytochemicals	Aqueous extract	Ethanol extract
Saponins	−	+++
Terpenes	−	−
Tannins	+	+++
Flavonoid	−	+
Phlobatannins	−	−
Anthraquinones	−	+
Cardiac glycoside	−	++
Alkaloids	−	+

Phytochemical constituents of aqueous and ethanol extract of *Lasianthera africana* are presented in Table 4. Significant constituents of the ethanol extracts are alkaloid, tannin, flavanoid, cardiac glycoside, anthraquinone, and alkaloid. These photochemical have structures (Fig. 5) that may enhanced their electron-donating ability, hence the inhibitive properties of the ethanol extract of *Lasianthera africana* may be attributed to their presence in the ethanol extract of the inhibitor. Careful inspection of Table 4 reveals that aqueous extract of *Lasianthera africana* is not suitable for use as an inhibitor because these constituents are absent.

Figure 6 shows the IR spectrum of the corrosion product (without the inhibitor). Figure 7 shows the IR spectrum of ethanol extract of *Lasianthera africana*, and Fig. 8 shows the IR spectrum of the corrosion product in the present of the inhibitor. From these figures, it was seen that that without the inhibitor there was no adsorption, whereas in the presence of the inhibitor different adsorption bands were found at different wavenumbers. From Figs. 7 and 8, it was found that the –OH stretch at 3,625.74 and 3,418.92 cm^{-1} were shifted to 3,435.63 cm^{-1}, the –NH stretch at 1,633.15 cm^{-1} was shifted to 1,631.62 cm^{-1}, while the C–N stretch was also formed at 1,124.30 cm^{-1}, indicating interaction between ethanol extract of *Lasianthera africana* and the surface of mild steel. Also C–O stretch, =C–H bend, C–H oop, –C=C– bend, and C-Br stretch were missing in the spectrum of the corrosion product, indicating that these functional groups were not used for bonding. Therefore, we proposed that the adsorption of the inhibitor took place via –OH and –NH stretch (Table 5).

Synergism refers to combined total action of a compound greater than the sum of its individual effects, one of

Fig. 5 Chemical structures of (**a**) anthraquinone, (**b**) saponins, (**c**) alkaloid, and (**d**) cardiac glycoside



the most important factors in inhibition processes, serving as the basis for all modern corrosion inhibitor formulation. Synergism of corrosion inhibitors is either due to interaction between components of the inhibitors or due to interaction between the inhibitor and one of the ions present in aqueous solution [17, 25]. Synergistic studies were carried out on the combination of each of the inhibitors with 0.06 M KI, KCl, and KBr, respectively. The synergism parameter (S) for the joint effect of inhibitors was calculated using Eq. 16 [17, 18]:

$$S = \frac{1 - I_A - I_B \pm I_A I_B}{1 - I_{AB}}, \quad (16)$$

where I_A and I_B are inhibition efficiencies of compounds A and B, respectively, and I_{AB} is the inhibition efficiency of a combination of two inhibitors. Values of S calculated from Eq. 16 are recorded in Table 6. These values are less than unity (except for KCl), indicating that adsorption of KI or KBr on surface of mild steel antagonized adsorption of the inhibitors and that the performance of ethanol extract of *Lasianthera africana* cannot be enhanced by KI and KBr. On the other hand, adsorption of KCl may enhance adsorption of the inhibitor ($S > 1$). In order to further ascertain the effect of halides on adsorption of the inhibitor, Langmuir adsorption isotherms were plotted for the inhibitor and inhibitor–halide combination. Adsorption parameters deduced from the isotherm (plots not shown) are recorded in Table 7. The results indicated that KCl slightly improve the value of ΔG_{ads}, indicating enhancement of adsorption. Such enhancement was not significant with KBr. Also KI significantly affected the adsorption behavior of the inhibitor such that the Langmuir isotherm was no longer applicable, as indicated by the low value of R^2.

4 Conclusion

From the study, the following conclusions can be made:

i. Ethanol extract of *Lasianthera africana* is a good inhibitor of corrosion of mild steel in H_2SO_4. The inhibitor extends the half-life of mild steel corrosion in H_2SO_4.

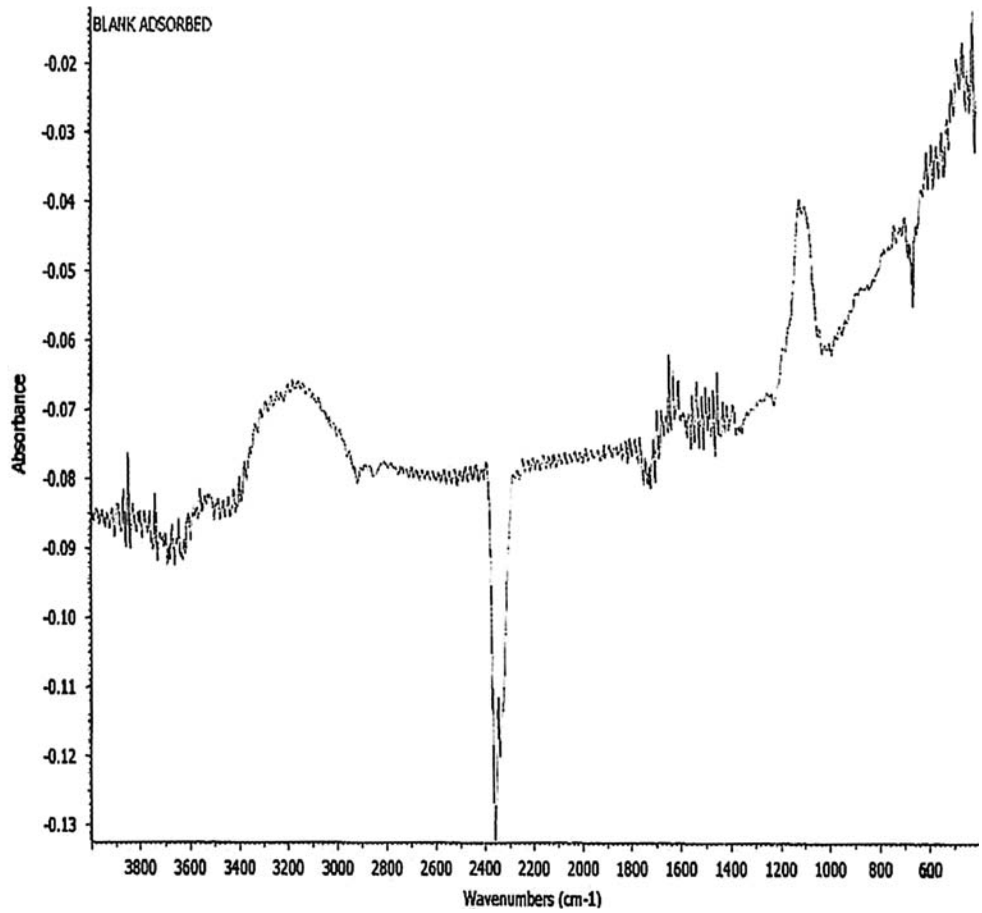


Fig. 6 IR spectrum of the corrosion product (without inhibitor)

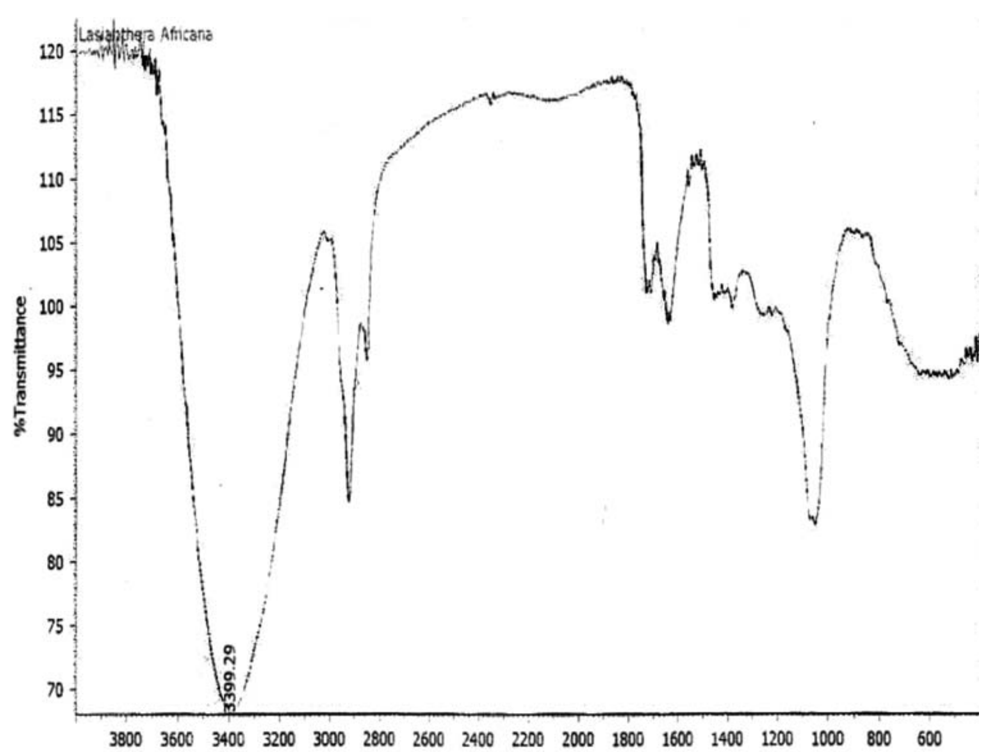


Fig. 7 IR spectrum of ethanol extract of *Lasianthera africana*

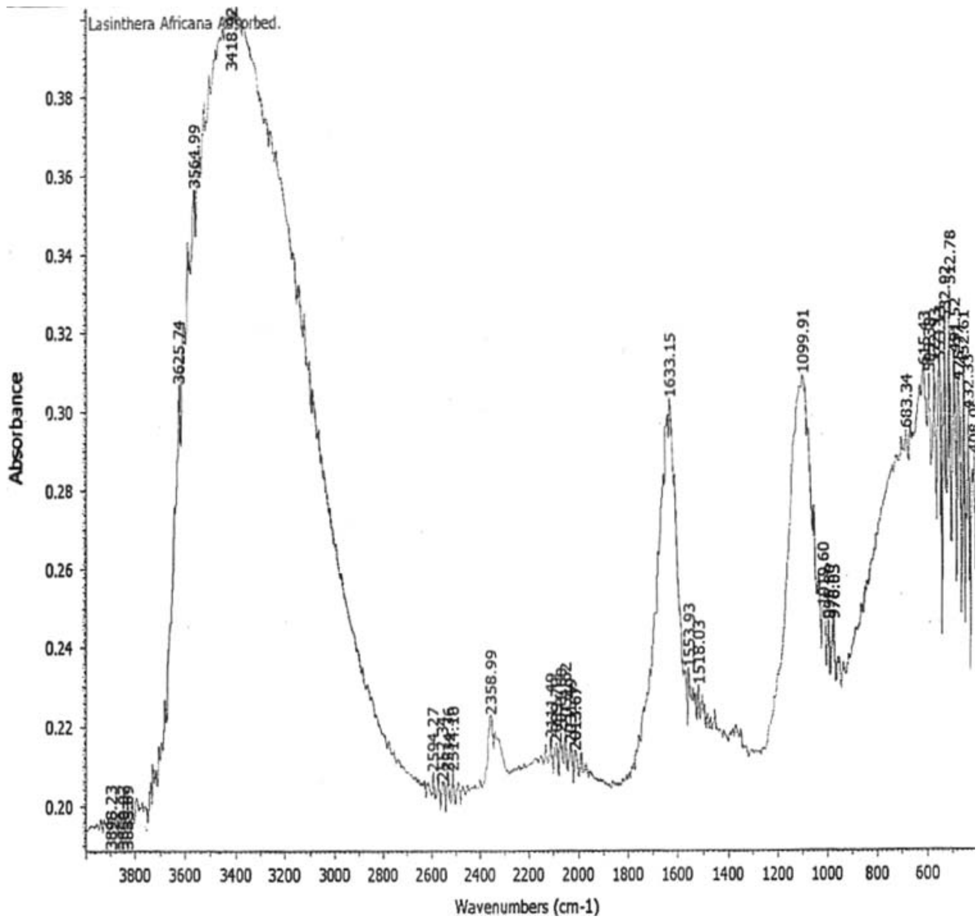


Fig. 8 IR spectrum of the corrosion product (in the presence of ethanol extract of *Lasianthera africana*

Table 5 Frequencies and peaks of IR adsorption by ethanol extract of *Lasianthera africana*

Pure extract			Extract adsorbed		
Frequency (cm^{-1})	Height (cm)	Assignment	Frequency (cm^{-1})	Height (cm)	Assignment
3,625.74	49.382	–OH stretch,	3435.63	10.670	–OH stretch
3,418.92	39.572	–OH stretch	1631.62	36.512	N–H bend, NO_2 asym. stretch
1,633.15	49.826	N–H bend, NO_2 asym. stretch	1,124.30	64.050	C–N stretch
1,553.93	58.324	N–H bend, NO_2 asym. stretch			
1,518.03	58.899	N–H bend, NO_2 asym. stretch			
1,099.91	49.156	C–O stretch			
1,019.60	56.280	C–O stretch			
995.80	56.718	=C–H bend, alkenes			
976.03	56.719	=C–H bend, alkenes			
683.34	50.744	C–H "oop", aromatic			
615.43	49.939	–C≡C–: C–H bend, alkynes			
593.38	49.125	C–Br stretch, alkyl halides			

ii. The extract inhibits corrosion of mild steel by being adsorbed onto the surface of the metal.
iii. The adsorption behavior of the inhibitor is consistent with Langmuir and Temkin adsorption models.
iv. Alkaloid, anthraquinone, saponin, cardiac glycoside, tannin, and flavanoid are the major constituents of ethanol extract of *Lasianthera africana* that enhance its inhibitive and adsorption properties.

Table 6 Synergistic parameters for joint effect of halides and ethanol extract of *Lasianthera africana*

C (g L^{-1})	Extract + 0.06 M		
	KBr	KI	KCl
0.1	−9.11	−680.79	149.09
0.2	−7.23	−259.74	93.82
0.3	−5.59	−124.27	91.81
0.4	−6.08	−98.20	104.28
0.5	−5.76	−42.80	53.38

Table 7 Langmuir parameters for joint adsorption of halides and ethanol extract of *Lasianthera africana* on the surface of mild steel at 303 K

	Log K	Slope	ΔG_{ads} (kJ mol^{-1})	R^2
Langmuir				
LA	0.0162	0.9437	−10.19	0.9910
LA + KBr	0.0365	0.3017	−10.31	0.9271
LA + KCl	0.2824	0.2824	−11.73	0.9996
LA + Kl	0.2223	0.2223	–	0.2980

v. The mechanism of physical adsorption is applicable to the adsorption of ethanol extract of *Lasianthera africana* on surface of mild steel.

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