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Inhibitory action of *Albizia zygia* gum on mild steel corrosion in acid medium

Paul Ocheje Ameh

Physical Chemistry Unit, Department of Chemistry, Nigeria Police Academy, Wudil, P. M B. 3474, Kano State Nigeria.

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The corrosion and inhibition behaviours of mild steel in sulphuric acid in the presence of *Albizia zygia* gum (AZ) have been studied using the weight loss, gasometric, Fourier transform infrared (FTIR) and thermometric techniques. The temperature studies reflect that the percentage of inhibition efficiency is decreased with rise in temperature and that indicates the mechanism of physical adsorption. The calculated values of activation energy (E_a) also support the physisorption process. The thermodynamic parameters such as heat of adsorption (Q_{ads}) and free energy of adsorption (ΔG_{ads}) are suggested that the adsorption of inhibitor on the mild steel surface is exothermic and followed by spontaneous process. The adsorption of the inhibitor on mild steel surface involves the formation of multimolecular layer through C-H bending vibrations due to alkenes at 703.08 and 933.58.01 cm^{-1}, C-O stretches due to carboxylic acid, ester, ether and alcohol at 1084.03 and 1150.58 cm^{-1}, C-O stretch due to ketone /aldehyde/carboxylic acid at 1826.65 cm^{-1}, C-H aliphatic stretch at 2820.99 cm^{-1} and C-H stretch at 3026.41.79 cm^{-1}, which support physical adsorption. The observed results indicate that *A. zygia* gum could serve as an effective inhibitor on mild steel in sulphuric acid.

Key words: *Albizia zygia* gum, thermodynamics, mild steel, sulphuric acid, adsorption.

INTRODUCTION

Corrosion has been one of the biggest problem of the human being economically especially from the beginning of last century. Researches have been geared towards protecting metals from corrosion by the use of inhibitors (Ashassi-Sorkhabi and Nabavi-Amri, 2002; Migahed and Nassar, 2008; Ameh et al., 2012; Fouda and Ellithy, 2009). Corrosion inhibitors are widely used in industry to control metal dissolution and reduce the corrosion rate in contact with aggressive acid solution. Most acid inhibitors are organic compounds containing nitrogen, sulphur and/or oxygen in their molecule (Ogoko et al., 2009; Odiongenyi et al., 2009). The inhibition action is due to the formation of protection film on to the metal surface blocking the metal from the corrosive agents present in solution (Ameh et al., 2013).

Plant products are organic in nature and some of the constituents including tannins, organic and amino acids, alkaloids, and pigments are known to exhibit inhibiting action. Moreover, they can be extracted by simple procedures with low cost. Literature on the use of gums as corrosion inhibitors is scanty (Ameh and Eddy, 2013a; Abdallah, 2004, Umoren, 2008; Ameh, 2012). *Albizia zygia* gum is of particular interest because of their safe use, high solubility in water and high molecular size. The gas chromatography – mass spectroscopy (GCMS) spectra of the gum indicate the presence of hetero atoms

E-mail: nocaseoche@yahoo.com.

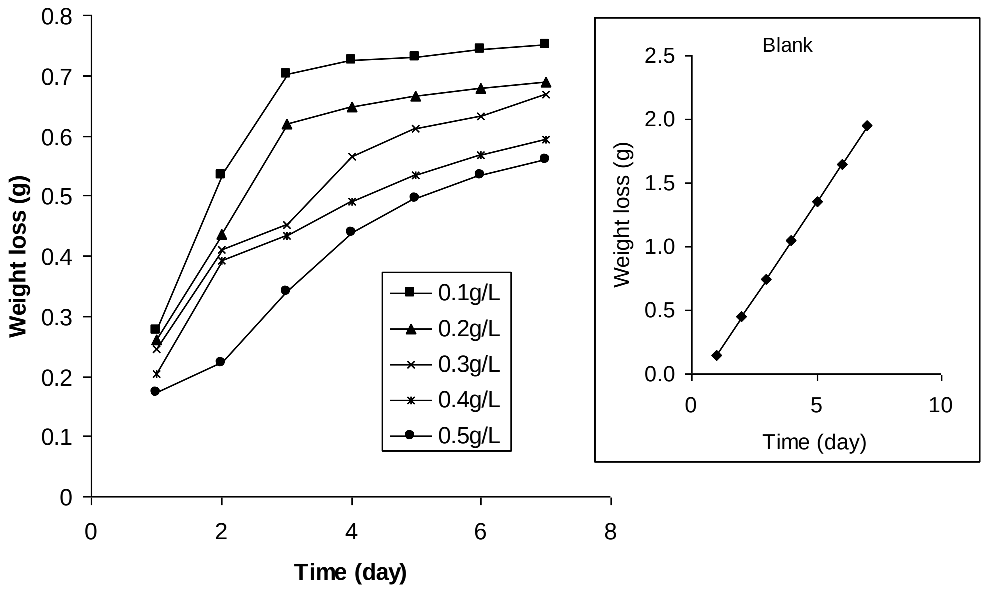

Figure 1. Variation of weight loss with time for the corrosion of mild steel in solutions of H_2SO_4 containing various concentrations of *A. zygia* gum (insert is the plot for the blank) at 303K.

like nitrogen, sulfur and oxygen in their structure (Ameh and Eddy, 2013b).

Albizia gum derived from trees of the genus *Albizia* is formed as round elongated bars of variable size and colour ranging from yellow to dark brown (Mital and Adotey, 1973).

In the present report, the inhibitive action of *A. zygia* gum on corrosion of mild steel in H_2SO_4 solutions is been reported. The inhibition has been evaluated by weight loss, thermometric, Fourier transform infrared (FTIR) and gasometric measurements. The experimental data obtained from different adsorption isotherms at different temperatures was tested so as to determine the thermodynamic functions for the adsorption process.

METHODOLOGY

Corrosion study

Mild steel [composition Fe (98.86), Mn (0.6), P (0.36), C (0.15) and Si (0.03)] coupons of dimension 5.0 cm × 4.0 cm × 0.11 cm were cut and wet-polished with silicon carbide abrasive paper, rinsed with distilled water, dried in acetone and warm air, and weighed before immersion in the test solutions. Tests were conducted under total immersion conditions in 250 ml of the aerated and unstirred test solutions. The mild steel coupons were retrieved from the test solutions at 24 h progressively for 168 h at 303 and 333 K respectively. The retrieved coupons were appropriately cleaned, dried, and reweighed. The weight loss was taken to be the difference between the weight of the coupons at a given time and its initial weight. All tests were run in triplicate and the data showed good reproducibility. From the average weight loss (mean of three replicate analysis) results, the inhibition efficiency (%I) of the inhibitor, the degree of surface coverage (θ) and the corrosion rate of mild steel (CR) were calculated using Equations (1), (2) and (3) respectively (Ladan et al., 2012);

$$\%I = \left(1 - \frac{W_1}{W_2}\right) \times 100 \quad (1)$$

$$\theta = \left(1 - \frac{W_1}{W_2}\right) \quad (2)$$

$$CR = \Delta W/At \quad (3)$$

Where W_1 and W_2 are the weight losses (g) for mild steel in the presence and absence of the inhibitor, θ is the degree of surface coverage of the inhibitor, $\Delta W = W_2 - W_1$, A is the area of the mild steel coupon (in cm^2), t is the period of immersion (in hours) and ΔW is the weight loss of mild steel after time, t.

Thermometric and hydrogen evolution experiments were carried out as reported previously (Eddy et al., 2011).

FTIR analysis

The FTIR analysis of gum was carried out and that of the corrosion products (in the absence and presence of gum were carried out using Scimadzu FTIR-8400S Fourier transform infra-red spectrophotometer. The sample was prepared in KBr and the analysis was carried out by scanning the sample through a wave number range of 400 to 4000 cm^{-1}.

RESULTS AND DISCUSSION

Corrosion inhibition study

Figures 1 and 2 show plots for the variation of weight loss of mild steel with time for the corrosion of mild steel in 0.1

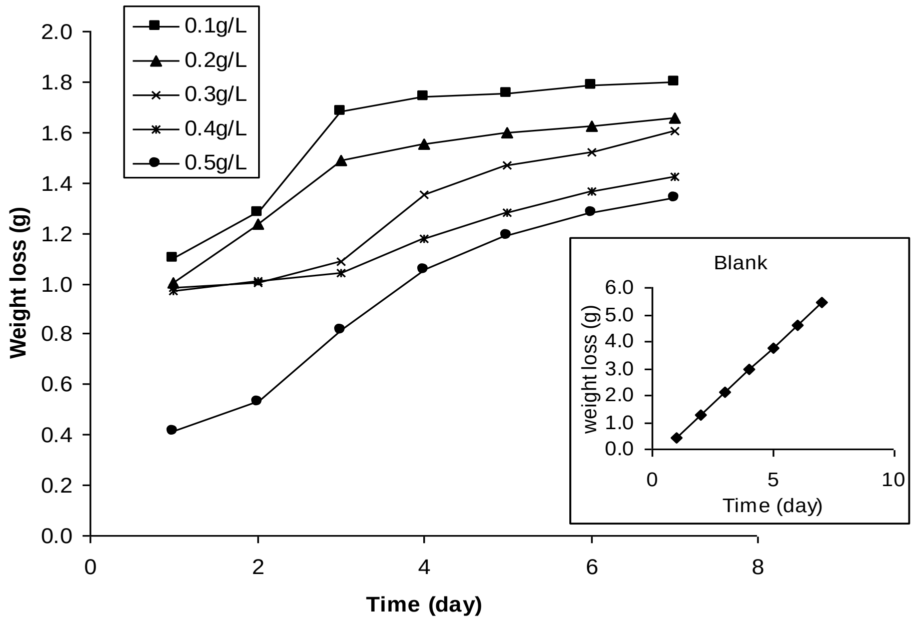

Figure 2. Variation of weight loss with time for the corrosion of mild steel in solutions of H_2SO_4 containing various concentrations of *A. zygia* gum (insert is the plot for the blank) at 333K.

Table 1. Corrosion rates (CR) of mild steel and inhibition efficiencies (%I) of *Albizia zygia* gum in various media.

C (g/L)	CR × 10^4 at 303K	CR × 10^4 at 333K	% I_W (303 K)	%I_W (333 K)	% I_T (303 K)	%I_T (333 K)	% I_G (303 K)
Blank	5.8	16.3	-	-	-	-	-
0.1	2.2	5.4	66.99	61.49	69.64	64.02	71.21
0.2	2.1	4.9	69.67	64.62	75.00	70.26	76.45
0.3	2.0	4.8	70.59	65.69	78.81	74.25	79.32
0.4	1.8	4.2	73.93	69.59	79.44	77.51	81.94
0.5	1.7	4.0	75.43	71.33	81.75	79.54	83.01

M H_2SO_4 containing various concentrations of *A. zygia* gum at 303 and 333K respectively. From the plots, it is evident that the weight loss of mild steel was also found to decrease with increase in the concentration of *A. zygia* gum. The weight loss of mild steel in the blank solution was also found to be higher than those obtained for solutions of H_2SO_4 containing various concentrations of *A. zygia* gum. These indicate that *A. zygia* gum is an adsorption inhibitor for the corrosion of mild steel in solutions of H_2SO_4. A close comparison between Figures 1 and 2 also revealed that weight loss of mild steel also increase with increasing temperature indicating that the rate of corrosion of mild steel also increase with increase in temperature.

In Table 1, values of the corrosion rates of mild steel and inhibition efficiency of *Albizia zygia* gum in various media are presented. It can be seen that the maximum of 83.01% inhibition efficiency is achieved at 0.5 g/L of inhibitor concentration. This is mainly due to the active chemical constituent of viz, π bonds, hetero atoms (O and N). The almost greater than 83% of surface coverage (θ) is due to the co-ordination between the metal and the hetero atom present in the inhibitor. The value of inhibition efficiency decreasing with rise in temperature suggests that physical adsorption mechanism (Ameh and Eddy, 2013b). These results indicate that the adsorption of main active components present in the inhibitor shield the metal surface at room temperature (Petchiammal et al., 2012). However it may be deshielded from the surface with rise in temperature.

Kinetic study

The kinetic of corrosion of mild steel in the absence and presence of various concentrations of *Albizia zygia* gum

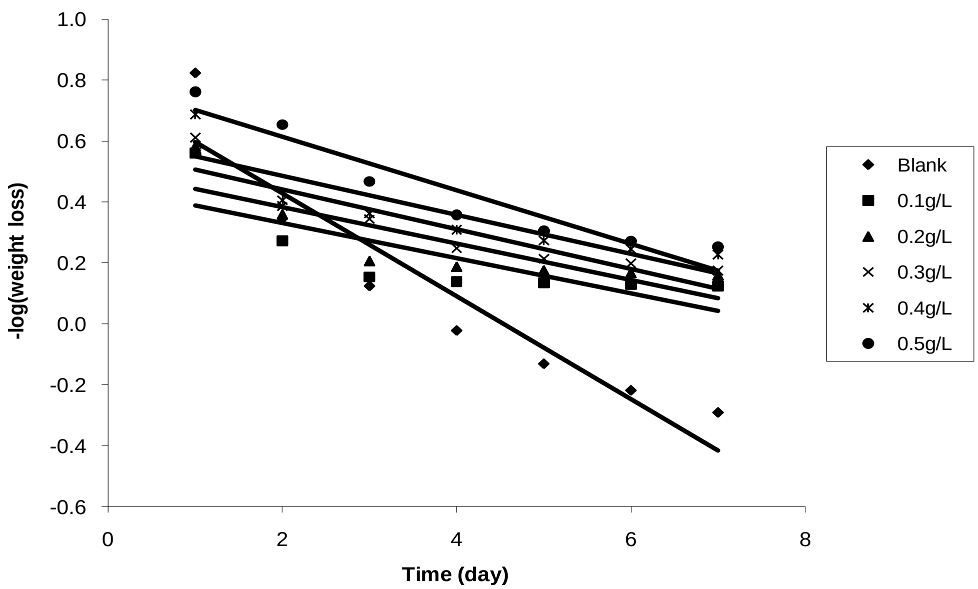

Figure 3. Variation of –log(weight loss) versus time for the corrosion of mild steel in solutions of H_2SO_4 containing various concentrations of *A. zygia* gum at 303K.

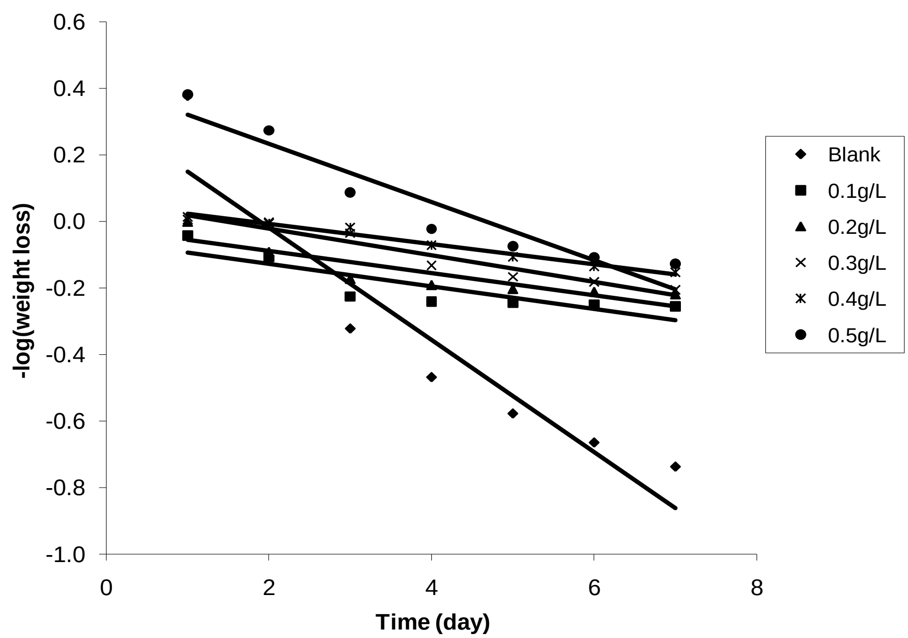

Figure 4. Variation of –log(weight loss) versus time for the corrosion of mild steel in solutions of H_2SO_4 containing various concentrations of *A. zygia* gum at 333K.

was investigated by plotting values of –log (weight loss) versus time as supported by the equation given below (El-Etre, 2006):

$$-\log (\text{weight loss}) = k_1t/2.303 \quad (4)$$

Where k_1 is the first - order rate constant and t is the time in day. Figures 3 and 4 shows kinetic plots for the corrosion of mild steel in solutions of H_2SO_4 containing various concentrations of *A. zygia* gum at 303 and 333 K. The observed linearity from the plots reveals first order kinetics for the inhibition process. Values of kinetic constants deduced from the plots are recorded in Table 2. Since the half-life ($t_{1/2}$) = $0.693/k_1$, calculated values of

Table 2. Kinetic parameters for the for the corrosion of mild steel in 0.1 M HCl containing various concentrations of *A. zygia* gum at 303 and 333K.

C (g/L)	k_1		$t_{1/2}$ (day)		R^2	
	303K	333K	303K	333K	303K	333K
Blank	0.3887	0.3887	2	2	0.8806	0.8806
0.1	0.1329	0.0778	5	9	0.7605	0.7382
0.2	0.1382	0.0762	5	9	0.7848	0.7831
0.3	0.1499	0.0924	5	8	0.8324	0.9383
0.4	0.1474	0.0700	5	10	0.7622	0.9758
0.5	0.2017	0.0942	3	7	0.9021	0.9021

Table 3. Heat of adsorption and activation energy for the inhibition of the corrosion of mild steel in solutions of HCl by *A. zygia* gum.

Concentration of *A. zygia* gum (g/L)	E_a (kJ/mol)	Q_{ads} (kJ/mol)
0.0	18.93	-
0.1	25.14	5.03
0.2	23.72	4.81
0.3	24.51	4.74
0.4	23.72	4.50
0.5	23.96	4.40

$t_{1/2}$ are also presented in the Table. From the results, it can be seen that the half-life values for inhibited systems are higher than those for the blank solutions.

Effect of temperature

The effect temperature on the corrosion of mild steel in the absence and presence of an inhibitor was studied using the Arrhenius equation, which can be written as follows:

$$\log\frac{CR_2}{CR_1} = \frac{E_a}{2.303R}\left(\frac{1}{T_1}-\frac{1}{T_2}\right) \quad (5)$$

Where E_a is the minimum energy required before corrosion can proceed (that is, activation energy) CR_1 and CR_2 are the corrosion rates of mild steel at the temperatures T_1 (303K) and T_2 (333K) respectively. Calculated values of E_a, which ranged from 23.72 to 25.14 kJ/mol (Table 3), which are lower than 40.0 kJ/mol indicating that the inhibitor is physically adsorbed on the metal surface. Physical adsorption requires that the average E_a be 40.0 kJ/mol and the inhibition efficiency should increase at lower temperatures (Onen et al., 2013)

The activation energies are within the range of values expected for the mechanism of physical adsorption. Therefore the adsorption of *A. zygia* gum on mild steel surface is consistent with the mechanism of physical adsorption.

Thermodynamics/adsorption study

Heat of adsorption (Q_{ads}) is the amount of heat needed for the inhibitor to be adsorbed on the surface of the metal. In addition since the corrosion inhibition was carried out at constant pressure, the heat adsorbed should approximate the enthalpy change. In this study, the heat of adsorption of *A. zygia* gum was calculated using the following equation:

$$Q_{ads} = 2.303R\left[\log\left(\frac{\theta_2}{1-\theta_2}\right)-\log\left(\frac{\theta_1}{1-\theta_1}\right)\right]x\left(\frac{T_{1X}T_2}{T_2-T_1}\right)kJmol^{-1} \quad (6)$$

Where θ_1 and θ_2 are the degrees of surface coverage of the inhibitor at temperatures, T_1 (303K) and T_2 (333K) respectively and R is the gas constant. Calculated values of Q_{ads} are also recorded in Table 3. These values are positive indicating that the adsorption of *A. zygia* gum on mild steel surface is endothermic.

The adsorption characteristics of *A. zygia* gum was also investigated by fitting data calculated for the degree of

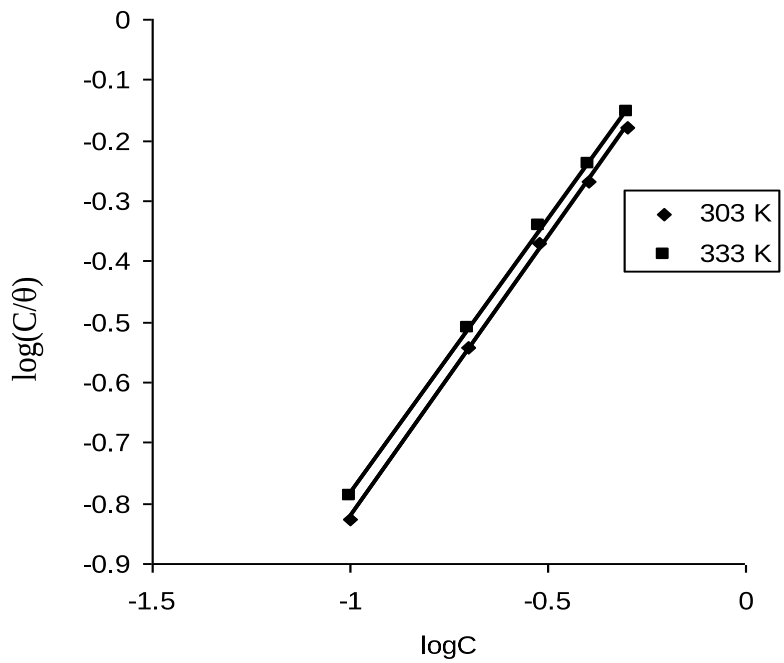

Figure 5. Langmuir isotherm for the adsorption of *A. zygia* gum on mild steel surface**.**

Table 4. Langmuir parameters for the adsorption of *A. zygia* gum on mild steel surface.

T (K)	Slope	$\log b_{ads}$	ΔG^0_{ads} (kJ/mol)	R^2
303	0.9278	0.105	-11.76	0.9996
333	0.9095	0.1247	-11.89	0.9993

surface coverage of the inhibitor into various adsorption isotherms including Langmuir, Freudlich, Temkin, El awardy, Frumkin and Flory - Huggins adsorption isotherms. The tests indicated that Langmuir isotherm best described the adsorption characteristics of the inhibitor. The assumptions establishing the Langmuir adsorption model can be written as follows (Emregul and Hayvali, 2006):

$C/\theta = C + 1/b_{ads}$

9 $\log(C/\theta) = \log C - \log b_{ads}$ (9)

Where C is the concentration of the inhibitor in the bulk electrolyte, b is the adsorption equilibrium constant and θ is the degree of surface coverage of the inhibitor. Figure 5 presents the Langmuir isotherms for the adsorption of *A. zygia* gum on mild steel surface. Values of adsorption parameters deduced from the plots are presented in Table 4.

The equilibrium constant of adsorption calculated from the intercept of the Langmuir plot is related to the free energy of adsorption according to the following equation:

$\Delta G^0_{ads} = -2.303RT\log(55.5*b_{ads})$ (10)

Where ΔG^0_{ads} is the standard free energy of adsorption of ethanol extract of *A. zygia* gum on mild steel surface, R is the universal gas constant and K_{ads} is the equilibrium constant of adsorption. Calculated values of free energy of adsorption are also presented in Table 4. The free energies are negatively less than the threshold value of -40 kJ/mol required for the mechanism of chemical adsorption. Therefore, the adsorption of *A. zygia* gum on mild steel surface is spontaneous and supports the mechanism of charged transfer from the charged inhibitor to the charged metal surface.

Mechanism of inhibition

A. zygia gum has been found to be a good corrosion inhibitor. The inhibition potential of this gum can be explained in terms of interaction between the metal and the gum. Most efficient corrosion inhibitors are long-carbon chain or aromatic compounds that have

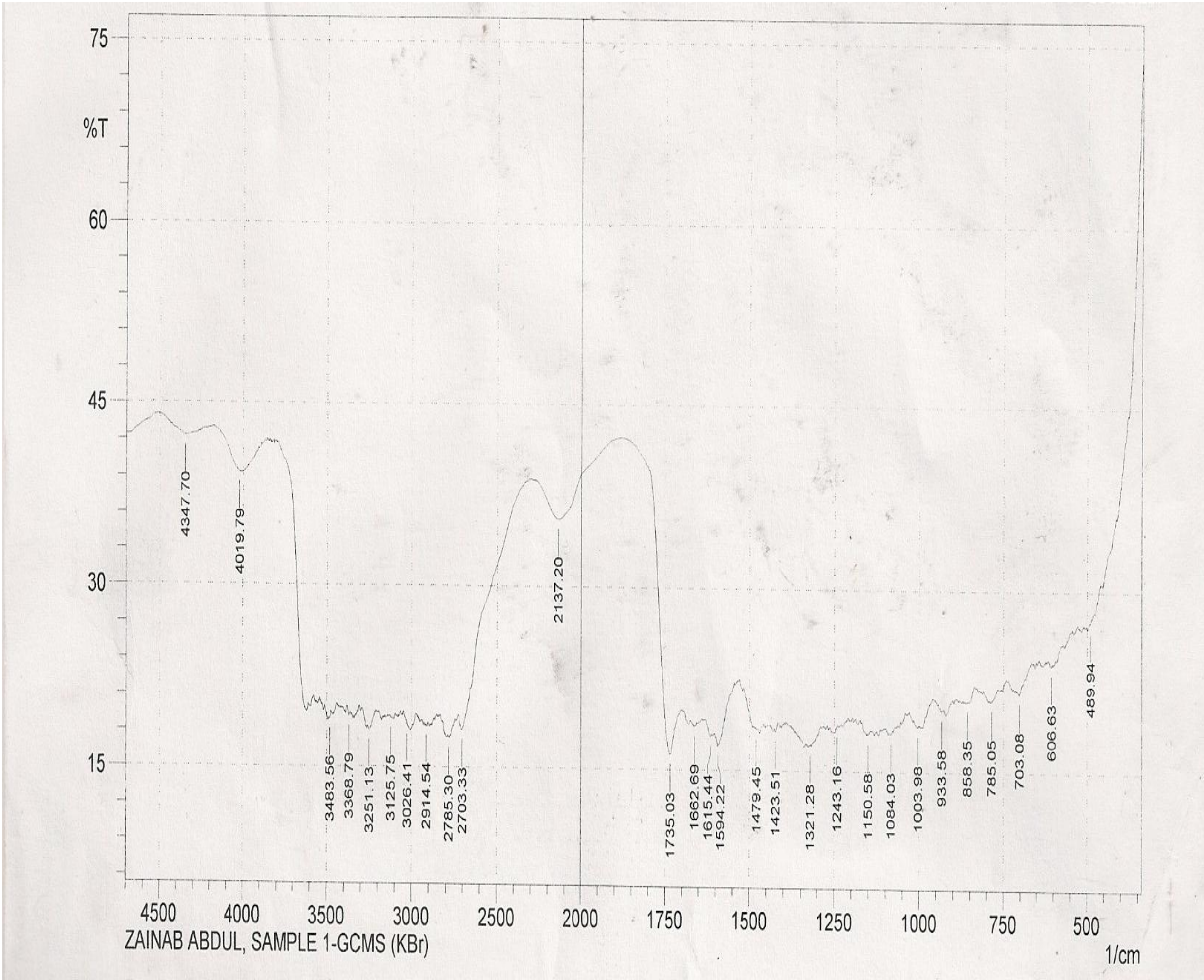

Figure 6. FTIR spectrum of *A. zygia* gum.

heteroatoms (N, S, P, and O) in their system. The present of π-electron rich functional groups have also been found to enhance inhibition efficiency of a corrosion inhibitor.

Figures 6 and 7 shows the FTIR spectrum of A. zygia gum and the corrosion product respectively. Wave numbers and intensities of adsorption deduced from the FTIR spectrum of *A. zygia* gum and the corrosion product as well as the assignment of vibration type and functional groups are presented in Tables 5 and 6. From the obtained results, it can be seen that the FTIR spectrum of the gum consist of several C-H bends which occurred in the wave number range of 703.08 to 1003.98 cm^{-1}, C-O stretch (1084.03 - 1243.16 cm^{-1}), C-N stretch (1321.28 cm^{-1}), N-H bending vibrations (1594.22 and 1615.44 cm^{-1}), C-H scissoring and bending vibrations (1423.51 and 1479.45 cm^{-1}). Other vibrations included C=C stretch at 1662.69, C≡C stretch at 2137.20, O-H stretch at 2785.30, 2785.30, 3125.75 and 3251.13 cm^{-1}. C-H bend at 1735.03 cm^{-1}, C-H stretches at 2914.54, 3026.41 and 3483.56 cm^{-1} as well as N-H stretch was also found at 3368.79 cm^{-1}.

The corrosion product of mild steel with that of the purified gum sample found that CH bend at 703.08, 785.05, 858.35 and 933.58 cm^{-1} were shifted to 701.15, 751.30, 804.34 and 883.43 cm^{-1}, the C-O stretch at 1084.03 and 1150.58 cm^{-1} were shifted to 1026.16 and 1149.61 cm^{-1} respectively, the C-H stretch at 1321.28 cm^{-1} was shifted to 1328.03 cm^{-1}, the C-H scissoring and bending vibrations at 1423.51 cm^{-1} was shifted to 1424.48 cm^{-1}, the C=C Stretch at 1594.22 cm^{-1} was shifted to 1600.97 cm-1, C≡C stretch at 2137.20 cm^{-1} was shifted to 2163.24 cm^{-1}, the OH stretch at 2785.30 cm^{-1} was shifted to 2717.79 cm^{-1},the C-H stretch at 2914.54 cm^{-1} was shifted to 2956.97 cm^{-1} and the OH stretch at 3151.75 was shifted to 3128.64 cm^{-1}. The shifts in the frequencies of vibration indicate that there is interaction between the inhibitor and the metal surface. In addition, the C-H scissoring at 1479.45 cm^{-1}, the N-H bend at 1615.44 cm^{-1}, the C=C stretch at 1662.69 cm^{-1},

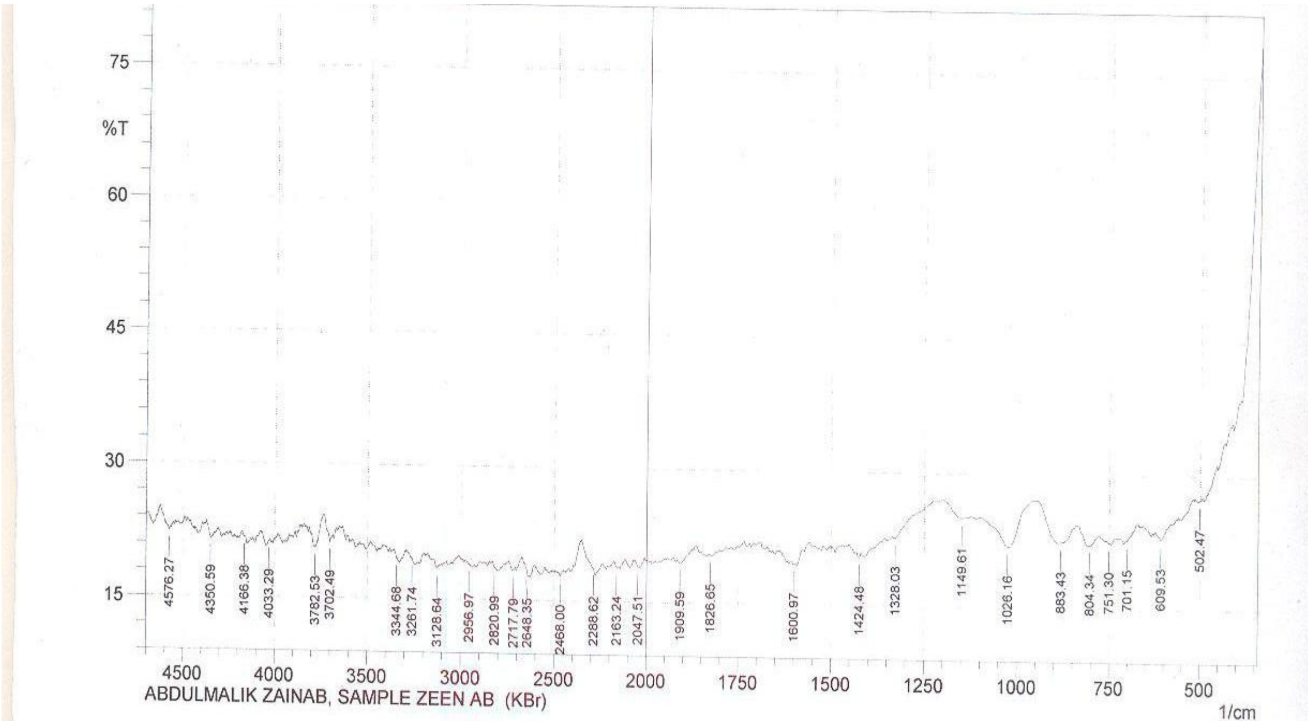

Figure 7. FTIR spectrum of the corrosion product of mild steel when *A. zygia* was used as an inhibitor.

Table 5. Peaks and intensity of adsorption of FTIR by *A. zygia* gum.

Peak (cm^{-1})	Intensity	Assignment (functional group)
703.08	21.507	C-H bend, phenyl ring substitution band
785.05	20.887	C-H bend, phenyl ring substitution band
858.35	20.822	C-H bend, phenyl ring substitution band
933.58	20.021	C-H bend, alkenes
1003.98	18.675	C-H bend, alkenes
1084.03	18.115	C-O stretch, carboxylic acid, ether, ester, alcohol
1150.58	18.043	C-O stretch, carboxylic acid, ether, ester, alcohol
1243.16	18.610	C-O stretch, carboxylic acid, ether, ester, alcohol
1321.28	17.092	C-N stretch, amine
1423.51	18.245	C-H scissoring and bending, alkanes
1479.45	18.467	C-H scissoring and bending, alkanes
1594.22	16.990	N-H bend, amines
1615.44	17.772	N-H bend, amines
1662.69	18.586	C=C stretch, alkenes
1735.03	16.277	C-H bend, phenyl ring substitution
2137.20	35.556	C≡C stretch, alkynes
2703.33	18.065	O-H stretch, carboxylic acid
2785.30	17.436	O-H stretch, carboxylic acid
2914.54	18.425	C-H stretch
3026.41	18.501	C-H stretch, alkenes
3125.75	19.035	O-H stretch
3251.13	18.023	O-H strech, alcohol, phenol
3368.79	19.172	N-H stretch, amine
3483.56	18.942	C-H stretch

Table 6. Peaks and intensity of adsorption of FTIR by the corrosion product of mild steel when *Albizia zygia* gum is used as an inhibitor

Peak (cm^{-1})	Intensity	Assignment (functional group)
609.53	22.90	C-H bend alkynes
701.15	22.73	C-H bend
751.30	22.73	C-H bend alkenes
804.34	22.19	C-H bend
883.43	22.42	C-H bend
1026.16	21.89	C-O stretch
1149.61	25.00	C-O stretch
1328.03	22.72	C-H stretch
1424.48	20.43	C-H scissoring and bending
1600.97	19.55	C≡C stretch, aromatic ring
1826.65	20.41	C=O stretch
1909.59	19.44	C-H stretch, phenyl ring subst.
2047.51	18.95	C≡C stretch
2163.24	18.85	C≡C stretch
2288.62	18.74	C- N stretch
2468.00	17.72	
2648.00	18.42	OH stretch
2717.79	18.42	OH stretch
2820.99	18.35	C-H aliphatic stretch
2956.97	18.94	C-H aliphatic stretch
3128.64	18.32	OH stretch

the OH stretch at 2703.22 cm^{-1} the C-H stretch at 3026.41 cm^{-1}, the OH stretch at 3251.13 cm^{-1}, the N-H stretch at 3368.79 cm^{-1} and the C-H stretch at 3483.56 cm^{-1} were absence in the spectrum of the corrosion product of mild steel suggesting that these functional groups were used in the adsorbing the inhibitor to the metal surface. On the other hand the 609.53 cm^{-1},C=O stretch at 1826.65 cm^{-1}, the C-H stretch at 1909.58 cm^{-1}, C≡C stretch at 2047.51 cm^{-1}, the C-N stretch at 2288.62 cm^{-1}, the OH stretch at 2648.00 and the CH aliphatic stress at 2820.99 cm^{-1} were new to the spectrum of the corrosion product indicating that new bonds were formed.

Conclusions

A. zygia gum is a good adsorption inhibitor for the corrosion of mild steel in acidic media. The adsorption of the inhibitor on mild steel surface involves the formation of multi molecular layer through C-H bending vibrations due to alkenes at 703.08 and 933.58.01 cm^{-1}, C-O stretches due to carboxylic acid, ester, ether and alcohol at 1084.03 and 1150.58 cm^{-1}, C-O stretch due to ketone /aldehyde/carboxylic acid at 1826.65 cm^{-1}, C-H aliphatic stretch at 2820.99 cm^{-1} and C-H stretch at 3026.41.79 cm^{-1}. The adsorption of the inhibitor was also found to be spontaneous, exothermic and supported the mechanism of physical adsorption

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