

GCMS Studies On *Anogessus Leocarpus* (Al) Gum and Their Corrosion Inhibition Potential for Mild Steel in 0.1 M HCl

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GCMS study of *Anogessus leocarpus* gum (AL gum) indicated the presence of sucrose (10.03 %), phthalic acid (2.53 %), n-hexadecanoic acid (11.73 %), oleic acid (30.49 %), pentacenequinone (4.41 %) and 2,3- diphenylnaphthoquinone (21.43 %). The gum was found to be a good adsorption inhibitor for the corrosion of mild steel in HCl solution. The adsorption of the inhibitor on mild steel surface is exothermic, spontaneous, Langmuir type and supports the mechanism of charged transfer from the inhibitor's molecule to the charged metal surface. FTIR study of the AL gum and the corrosion product of mild steel (in the presence of AL gum as an inhibitor) indicated the presence of interaction between the inhibitor and the metal surface (some shifts in frequencies of IR adsorption), some functional groups were used for the adsorption of the inhibitor onto the metal surface (missing functional groups) and others for the formation of Fe-inhibitor complex (new functional groups formed). The inhibition of mild steel corrosion is proposed to occur through synergistic combination of the constituents of the inhibitor, which facilitates the formation of multi-molecular layer of adsorption and the formation of inhibitor-metal complex, which protected the metal from further corrosion attack.

Keywords: *Anogessus leocarpus* gum, GCMS, FTIR, corrosion inhibition, adsorption

1. INTRODUCTION

Several researches have indicated that some polymers can be used as corrosion inhibitors because, through their functional groups, they form complexes with metal ions and on the metal surfaces. These complexes occupy a large surface area, thereby blanketing the surface and protecting the metals from corrosive agents present in the solution. The corrosion inhibition by various cationic polymers such as polyethyleneimine derivative, polyacrylamide derivative, polydicyanodiamide

derivative, and anionic polymers such as polymaleic acid derivative, polyacrylic acid derivative, and polyacrylic acid, have been investigated by Sekine *et al.* [1]. They found that polymers having the COOH group are effective polymer-based systems that can function as corrosion inhibitors. Muller [2] studied the corrosion inhibition of polymethacrylic acid and styrene maleic acid co-polymer on zinc pigments in aqueous alkaline medium.

Abdel Rehim *et al.*[3] found that the amino polycarboxylic acids, such as diethylenetriaminepentaacetic acid, polyacrylic acid, and polymethacrylic acid, are good inhibitors for metal corrosion.

Sedahmed *et al.* [4] investigated the use of a formulation containing polyethylene oxide, polyacrylamide, and CMC as corrosion inhibitors for iron in acidic and neutral media using electrochemical methods and found that this formulation is an excellent inhibitor. Khairou and Sayed [5] studied the inhibiting action of polyacrylamide, PVA, sodium polyacrylate, poly (ethylene glycol), pectin, and carboxymethyl cellulose (CMC) on the corrosion of Cd in 0.5 M HCl solution. Meena *et al.* [6] investigated the synergistic effect between CMC and Zn^{2+} on the corrosion inhibition of carbon steel in NaCl solutions and found that the combination produces strong inhibition potential.

Studies on the use of some macromolecules (i.e natural polymers) have also been carried out. For example, Umoren *et al.* [7] studied the potential of *Gum arabic* as corrosion inhibitor for aluminium in alkaline medium. The inhibition of aluminium corrosion by *Gum arabic* was attributed to the presence of arabinogalactan, oligosaccharides, polysaccharides and glucoproteins since these compounds contain oxygen and nitrogen atoms which are the centers of adsorption. The inhibitive effect of exudate gum from *Dacryodes edulis* on the corrosion of aluminium in HCl solutions was studied using weight loss and thermometric methods at 30-60 °C by Umoren *et al* [8] and the results revealed that the exudate gum acted as an inhibitor for the corrosion of aluminium in HCl solution. The inhibition efficiency increases with an increase in the concentration of the exudate gum but decreases with increase in temperature.

The effect of naturally occurring exudate gum from *Raphia hookeri* on the corrosion of mild steel in H_2SO_4 between 30 and 60 °C have also been investigated by Umoren *et al* [9] using weight loss and hydrogen evolution techniques. Results obtained reveal that the exudate gum is a good inhibitor for the corrosion of mild steel in acidic media. The inhibition efficiency increased with an increase in exudate gum content and decreases with increase in temperature. The adsorption of exudate gum from *Raphia hookeri* on the mild steel follows Langmuir adsorption isotherm. *Guar gum* has been shown to be an effective corrosion inhibitor for some metals in aggressive acid environment by Abdallah [10]. Results obtained show that natural substances act as effective corrosion inhibitor in the different test media. Inhibition efficiency was found to increase with increase in the concentration of the tested material.

It should be pointed out that the potentials of polymers as corrosion inhibitors is dependent on its chemical composition and most of the studies carried out on polymers are done without reference to their chemical properties.

Therefore, the present study is aimed at elucidating the chemical structure of *Anogessus leocarpus* gum (AL) and to investigate its corrosion inhibition potential for mild steel in solutions of HCl.

2. MATERIALS AND METHODS

2.1. Corrosion study

Materials used for the study were mild steel sheet of composition (wt %, as determined by quantimetric method) Mn (0.6), P (0.36), C(0.15) and Si (0.03) and the rest Fe. The sheets were mechanically pressed cut into different coupons, each of dimension, 5x4x0.11cm. Each coupon was degreased by washing with ethanol, cleaned with acetone and allowed to dry in the air before preservation in a desiccator. All reagents used for the study were analar grade and double distilled water was used for their preparation. Concentrations of HCl used for gasometric, thermometric and weight loss studies were 2.5, 2.5 and 0.1 M respectively. The *Anogessus leocarpus* gum exudates used for the studies was purified and concentrations of 0.1 – 0.5g/l were used.

2.2. Gravimetric method

In the gravimetric experiment, a previously weighed metal (mild steel) coupon was completely immersed in 250 ml of the test solution in an open beaker. The beaker was covered with aluminium foil and inserted into a water bath maintained at 303 K. After every 24 hours, the corrosion product was removed by washing each coupon (withdrawn from the test solution) in a solution containing 50 % NaOH and 100 g l⁻¹ of zinc dust. The washed coupon was rinsed in acetone and dried in the air before re-weighing. The experiment was repeated at 333 K. In each case, the difference in weight for a period of 168 hours was taken as the total weight loss. 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 [11];

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

$$\Theta = 1 - W_1/W_2 \quad 2$$

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

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 cm²), t is the period of immersion (in hours) and ΔW is the weight loss of mild steel after time, t .

2.3 Gasometric method

Gasometric methods were carried out at 303 K as described in literature [12]. From the volume of hydrogen evolved per minute, inhibition efficiencies were calculated using equation 4.

$$\%I = \left(1 - \frac{V_{Hr}^1}{V_{Hr}^o} \right) \times 100 \quad 4$$

where V_{Hr}^1 and V_{Hr}^o are the volumes of H_2 gas evolved at time 't' for inhibited and uninhibited solutions respectively.

2.4 Thermometric method

This was also carried out as reported elsewhere [13]. From the rise in temperature of the system per minute, the reaction number (RN) was calculated using equation 5 [13]

$$RN \left(^\circ C \text{ min}^{-1} \right) = \frac{T_m - T_i}{t} \quad 5$$

where T_m and T_i are the maximum and initial temperatures respectively and 't' is the time (min) taken to reach the maximum temperature. The inhibition efficiency (%I) of the inhibitor was evaluated from percentage reduction in the reaction number namely.

2.5. FTIR analysis

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

2.6. GC-MS analysis

GC-MS analysis was carried out on a GC clarus 500 Perkin Elmer system comprising a AOC-20i autosampler and gas chromatograph interfaced to a mass spectrometer (GC-MS) instrument employing the following conditions: column

Elite-1 fused silica capillary column (30 x 0.25 mm ID x 1 μ M df, composed of 100% Dimethyl poly diloxane), operating in electron impact mode at 70eV; helium (99.999%) was used as carrier gas at a constant flow of 1 ml /min and an injection volume of 0.5 μ l was employed (split ratio of 10:1) injector temperature 250 $^\circ$ C; ion-source temperature 280 $^\circ$ C.

The oven temperature was programmed from 110 $^\circ$ C (isothermal for 2 min), with an increase of 10 $^\circ$ C/min, to 200 $^\circ$ C, then 5 $^\circ$ C/min to 280 $^\circ$ C, ending with a 9min isothermal at 280 $^\circ$ C. Mass spectra were taken at 70 eV; a scan interval of 0.5 seconds and fragments from 40 to 450 Da. Total GC running time is 36min.

2.7. Interpretation of GC-MS spectrum

Interpretation on mass spectrum GC-MS was conducted using the database of National Institute Standard and Technology (NIST) having more than 62,000 patterns. The spectrum of the unknown component was compared with the spectrum of the known components stored in the NIST library. The name, molecular weight and structure of the components of the test materials were ascertained. Concentrations of the identified compounds were determined through area and height normalization.

3. RESULTS AND DISCUSSIONS

3.1 GCMS

The GCMS spectrum of AL is presented in Fig. 1.

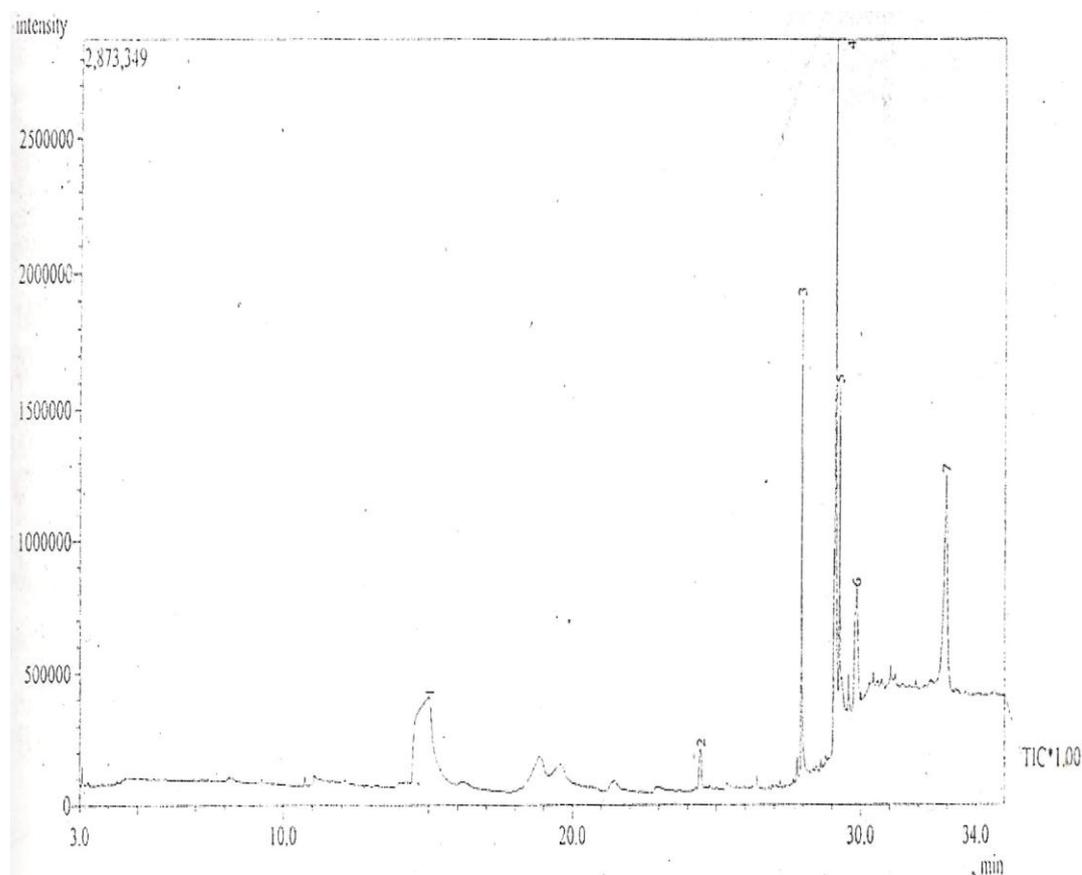


Figure 1.GC-MS spectrum of AL gum

Chemical structures of most probable compounds deduced from the spectrum are presented in Fig. 2. Seven peaks were isolated from the GC analysis while Table 1 presents data associated with each of the lines in the spectrum. Line 1 indicated the presence of sucrose (10.03 %) while lines 2 to 5

indicated the presence of carboxylic acids. These included phthalic acid (2.53 %), n-hexadecanoic acid (11.73 %) and oleic acid (30.49 %).

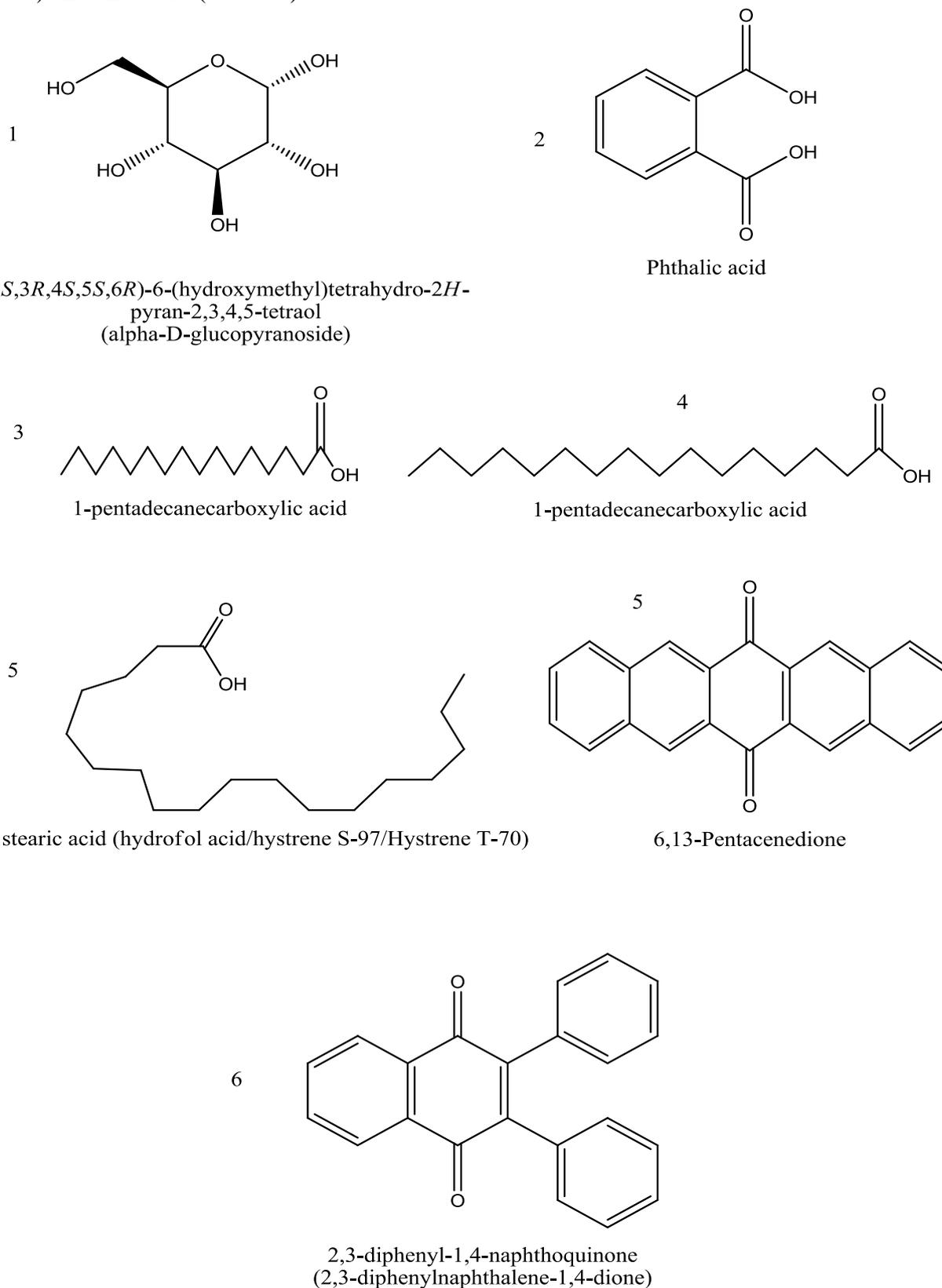


Figure 2. Chemical structures of compounds identified in GC-MS spectrum of AL gum (Numbering correspond to the GC line number)

In line 6, 4.41 % of pentacenequinone was identified. Pentacenequinone is a highly conjugated compound and functions as an organic semiconductor.

The compound generates exactions upon absorption of ultra-violet (UV) or visible light; this makes it very sensitive to oxidation. For this reason, this compound, which is a purple powder, slowly degrades upon exposure to air and light.

Table 1. Summary of GC-MS results for peaks in AL gum spectrum.

Peak NO	Peak area	C (%)	Compound	MF	MW	RT (sec)	S.I	FRAGMENTATION PEAKS
1.	0.3	10.03	Sucrose	C ₁₂ H ₂₂ O ₁₁	342	15.050	81	68 (55%), 77 (50%), 133 (100%), 151 (70%)
2	0.5	2.53	Phthalic acid	C ₂₀ H ₂₆ O ₄	330	7.5	78	43(10%), 51 (20%), 59(100%), 75(30%), 105(40%), 121(5%), 149 (2 %), 165 (2 %)
3	2.3	11.73	n-Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	256	31.7	86	27 (20%) 41(80%), 43 (100%), 60(90%), 73(100%), 85(20%), 98(15%), 115 (15%), 129 (30 %), 143(5%), 157 (10%), 171(10%), 185(10%), 213(20%), 256(50%)
4	2.8	30.49	1-pentadecane carboxylic acid	C ₁₈ H ₃₄ O ₂	282	33.0	84	27 (25%), 41(100%), 55 (80%), 69(40%), 83(30%), 97(20%), 123(2%), 137 (2%), 264 (5 %).
5	2.3	6.886	Stearic acid	C ₁₈ H ₃₆ O ₂	284	33.1	69	27 (18%) 41(80%), 57 (70%), 73(70%), 85(30%), 98(35%), 115 (10 %), 129 (40 %), 143(5%), 171(5%), 185(20%), 199(5%), 241(20%), 284(80%).
6	2.2	4.41	6,13-Pentacenedione	C ₁₄ H ₁₂ N ₂ O ₃	254	33.5	64	39 (1%) 64(1%), 78 (1%), 91(1%), 108(1%), 119(1%), 128(1%), 146 (1%), 155(30%), 169(2%), 185 (2%), 198(10%), 211(1%), 226(2%), 239(5%), 254(100%).
7	5.3	21.43	2,3-diphenyl-1,4-naphthoquinone	C ₂₀ H ₂₈ O ₂	300	34.9	66	41(20%) , 43(40%), 69(2%), 77(2%), 91(5%), 105(2%), 117(10%), 131(10%), 141 (20 %), 159(10%), 169 (5%), 183(5%), 197(40%), 239(20%), 285(100%)

Finally, in line 7, 21.43 % of 2,3- diphenylnaphthoquinone was the most probable compound. This compound is a potential chemotherapeutic agent against *Trypanosoma cruzi*.

3.2 Corrosion inhibition study

Fig. 3 shows the variation of weight loss with time for the corrosion of mild steel in 0.1 M HCl containing various concentrations of AL gum as an inhibitor at 303 K. From the figure, it is evident that weight loss of mild steel increases with increase in the period of contact but decreases with

increase in the concentration of AL gum indicating that AL gum retarded the rate of corrosion of mild steel in solutions of HCl and that AL gum is an adsorption inhibitor for the corrosion of mild steel. At higher temperature (333 K; plots not shown), weight loss of mild steel was found to increase with increasing temperature indicating that the mechanism of inhibition of mild steel corrosion by AL gum is by physisorption [14].

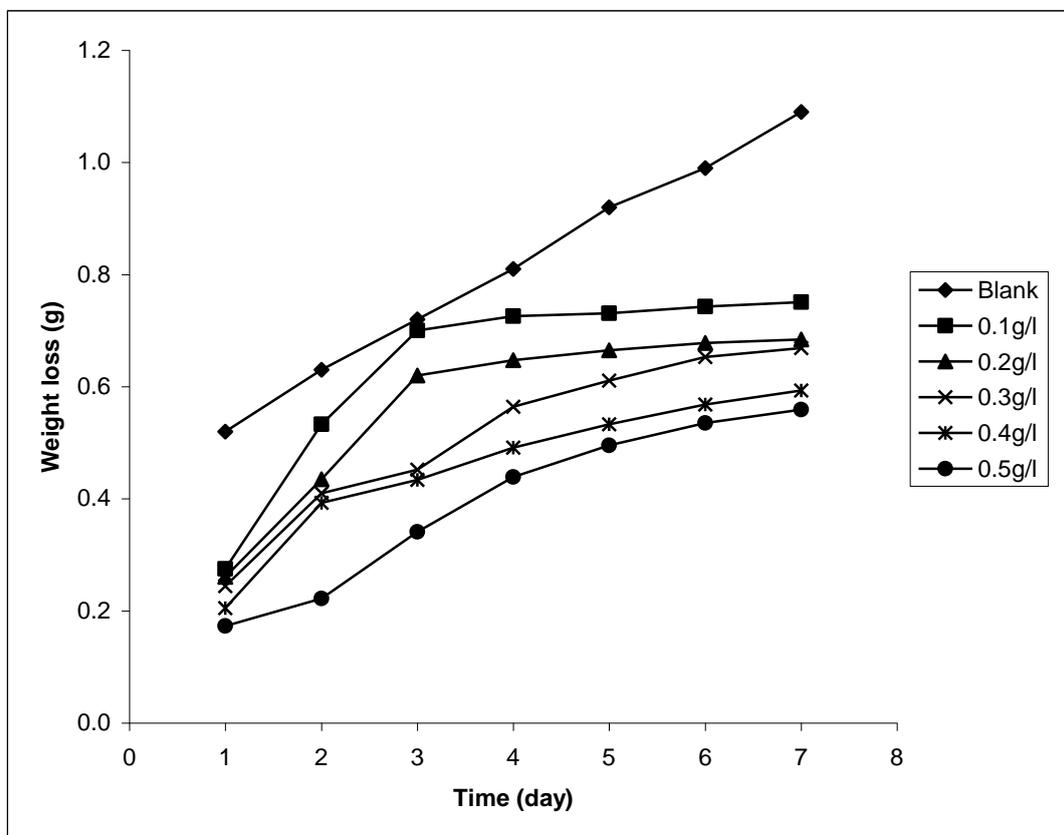


Figure 3. Variation of weight loss with time for the corrosion of mild steel in 0.1 M of HCl containing various concentrations of AL at 303 K

For a physical adsorption mechanism, the extent of adsorption tend to decrease with increasing temperature but decreases with increasing concentration for a chemical adsorption mechanism [15].

In Table 2, values of inhibition efficiency of AL gum calculated from weight loss, hydrogen evolution and gasometric measurements are presented. From the results obtained, it can be seen that the inhibition efficiency of AL gum increases with increasing concentration but decreases with increasing temperature. Also, inhibition efficiencies of various concentrations of AL gum obtained from gasometric and thermometric measurements were higher than those obtained from gravimetric measurement implying that the instantaneous inhibition efficiency of AL gum is better than the average inhibition efficiency of AL gum. This is consistent with the fact that weight loss method measures average inhibition efficiency (i.e inhibition efficiency over a long time) while gasometric and

thermometric methods measure instantaneous inhibition efficiency (i.e inhibition efficiency over a short time).

Table 2. inhibition efficiency of various concentrations of AL gum at 303 and 333 K for the corrosion of mild steel in 0.1 M HCl

Concentration of AL (g/l)	Inhibition efficiency (%)				
	Gravimetric		Gasometric		Thermometric
	303K	333 K	303 K	333 K	303 K
0.1	47.06	41.72	54.32	46.37	52.38
0.2	51.45	45.51	60.00	48.44	56.00
0.3	55.46	51.23	65.49	56.33	58.80
0.4	58.97	56.30	70.32	59.00	62.00
0.5	64.53	60.20	76.40	64.50	66.00

3.3 Effect of temperature

In order to study the effect of temperature on the corrosion of mild steel in H₂SO₄ containing various concentrations of AL gum, the Arrhenius equation was used [16]

$$\log \frac{CR_2}{CR_1} = \frac{E_a}{2.303R} \left(\frac{1}{T_1} - \frac{1}{T_2} \right) \tag{6}$$

where CR₁ and CR₂ are the corrosion rates of mild steel at the temperatures, T₁(303 K) and T₂(333 K) respectively, E_a is the activation energy and R is the gas constant. Calculated values of activation energy are presented in Table 3.

Table 3. Activation energy and heat of adsorption of AL gum on mild steel surface

Concentration of AL (g/l)	E _a (J/mol)	Q _{ads} (J/mol)
Blank	38.97	-
0.1	45.16	-61.69
0.2	53.83	-59.46
0.3	54.88	-66.58
0.4	56.07	-76.23
0.5	55.78	-66.09

The activation energies ranged from 45.16 to 56.07 J/mol and are within the range of values expected for the mechanism of physical adsorption. Also, the activation energies are greater than the

value of 38.97 kJ/mol obtained for the blank indicating that AL gum retarded the corrosion of mild steel in solutions of HCl.

3.4 Thermodynamic/adsorption study

The heat of adsorption (Q_{ads}) of AL gum on mild steel surface was calculated using the following equation [17]

$$Q_{ads} = 2.303R [\log(\theta_2/(1-\theta_2)) - \log(\theta_1/(1-\theta_1))] \times (T_1 \times T_2)/(T_2 - T_1) \tag{7}$$

Values of Q_{ads} calculated from equation 7 are also recorded in Table 3.

The adsorption characteristic of the inhibitors was investigated by fitting data obtained from weight loss measurement into different adsorption isotherms including Langmuir, Freundlich, Temkin, Flory Huggins, Frumkin and El awardy adsorption isotherm. The tests revealed that the adsorption behaviour of the inhibitors is best described by the Langmuir adsorption model, which can be expressed as follows,

$$\log(C/\theta) = \log C - \log b \tag{8}$$

where b is the adsorption equilibrium constant and θ is the degree of surface coverage of the inhibitor [18].

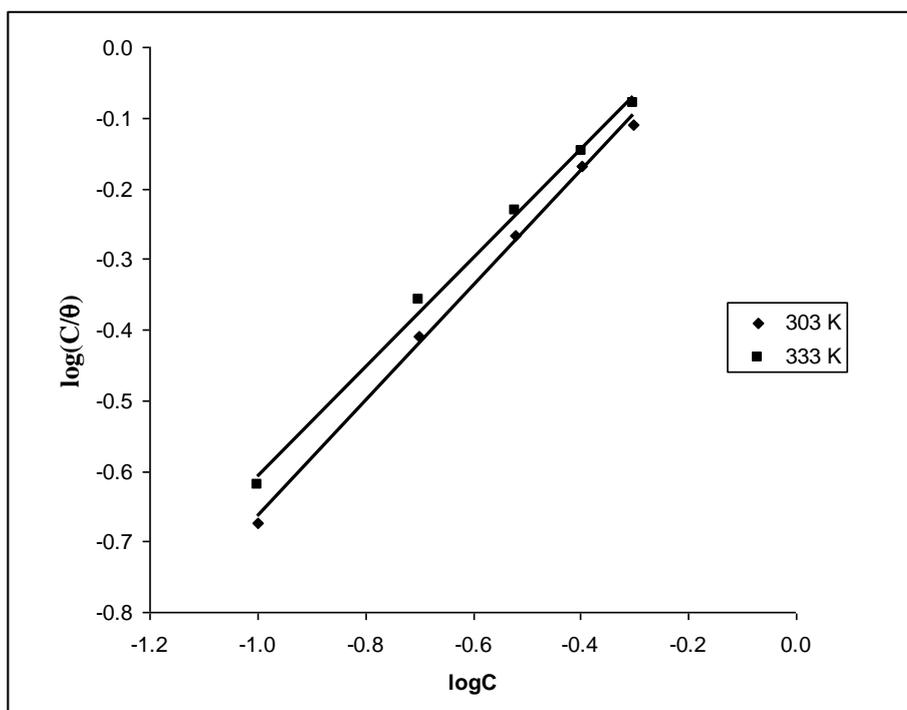


Figure 4. Langmuir isotherm plots for adsorption of AL gum on mild steel surface at 303 and 333 K

Using equation 8, plots of $\log(C/\theta)$ versus $\log C$ were linear for all the inhibitors. Fig. 4 presents the Langmuir isotherm for the adsorption of AL gum on mild steel surface. Adsorption parameters deduced from the plots are presented in Table 4.

Table 4. Langmuir parameters for the adsorption of AL gum on mild steel surface

T (K)	Slope	Logb	ΔG_{ads}^0 (J/mol)	R^2
303	0.813	0.1491	-36.25	0.9975
333	0.7688	0.1613	-36.49	0.996

From the results obtained, it is evident that R^2 values and slopes of the plots are close to unity confirming the application of the Langmuir model for the adsorption of AL gum. Hence, there is no interaction between the adsorbed species.

The equilibrium constant of adsorption obtained from the Langmuir adsorption isotherm is related to the standard free energy of adsorption according to the following equation [19]

$$\Delta G_{\text{ads}} = -2.303RT \log (55.5b) \quad 9$$

where R is the gas constant in kJ/mol, T is the temperature in Kelvin, b is the equilibrium constant of adsorption and 55.5 is the molar concentration of H_2SO_4 in water. Calculated values of ΔG_{ads} are recorded in Table 4. The results indicated that the free energies are negatively less than the threshold value of -40 kJ/mol. Therefore the adsorption of AL gum on mild steel surface is spontaneous and supports the mechanism of physical adsorption.

3.5 Mechanism of inhibition

The basic requirements for a given compound to be a good corrosion inhibitor are as follows

- i. possession of aromatic or long carbon chain that has hetero atom
- ii. presence of hetero atom(s) in the compound
- iii. Presence of suitable functional groups (i.e π -electron rich functional systems)
- iv. Presence of conjugated system

From the chemical structures of the compounds presence in AL gum (Fig. 2), it is evident that all the compounds presence in AL gum meet the first three conditions. The fourth condition is significantly met by 6,13-pentacenedione, which is a highly conjugated system. Consequently, inhibition of the corrosion of mild steel by AL gum can be considered through synergistic interaction of its chemical constituents. FTIR is a powerful tool for identifying the functional groups associated with the adsorption of an inhibitor [20].

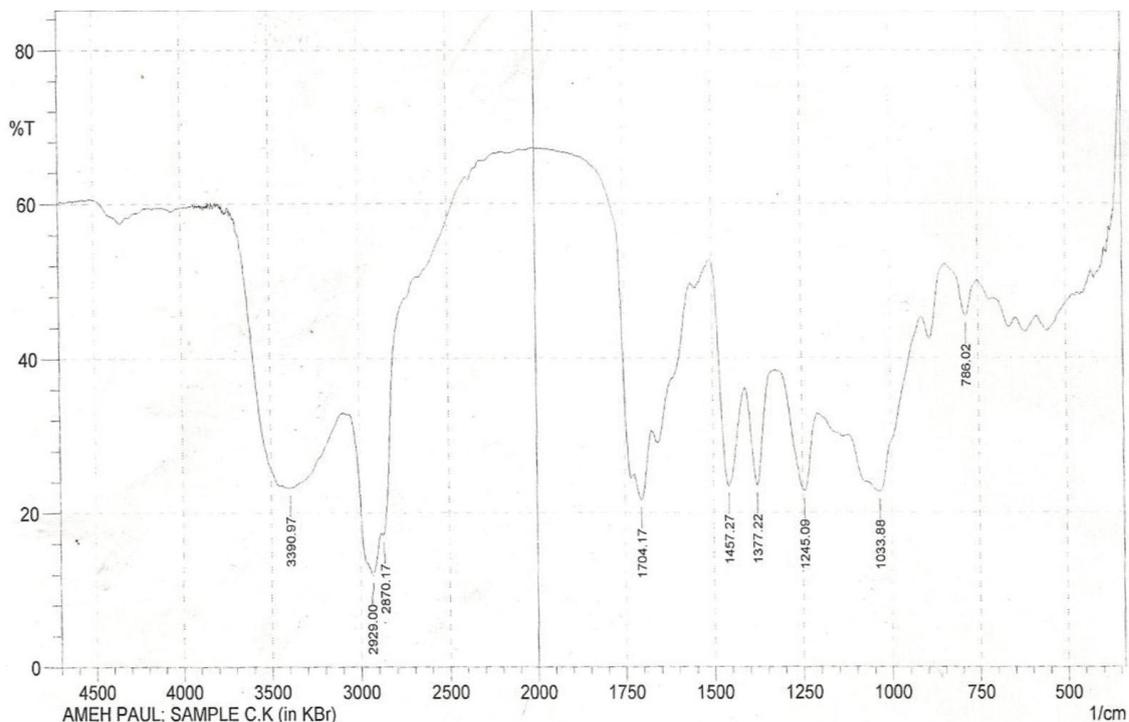


Figure 5. FTIR of AL gum

By comparing the FTIR of AL gum (Fig. 5) with that of the corrosion product of mild steel when AL gum is used as an inhibitor (Fig. 6), functional groups responsible for adsorption can be deduced.

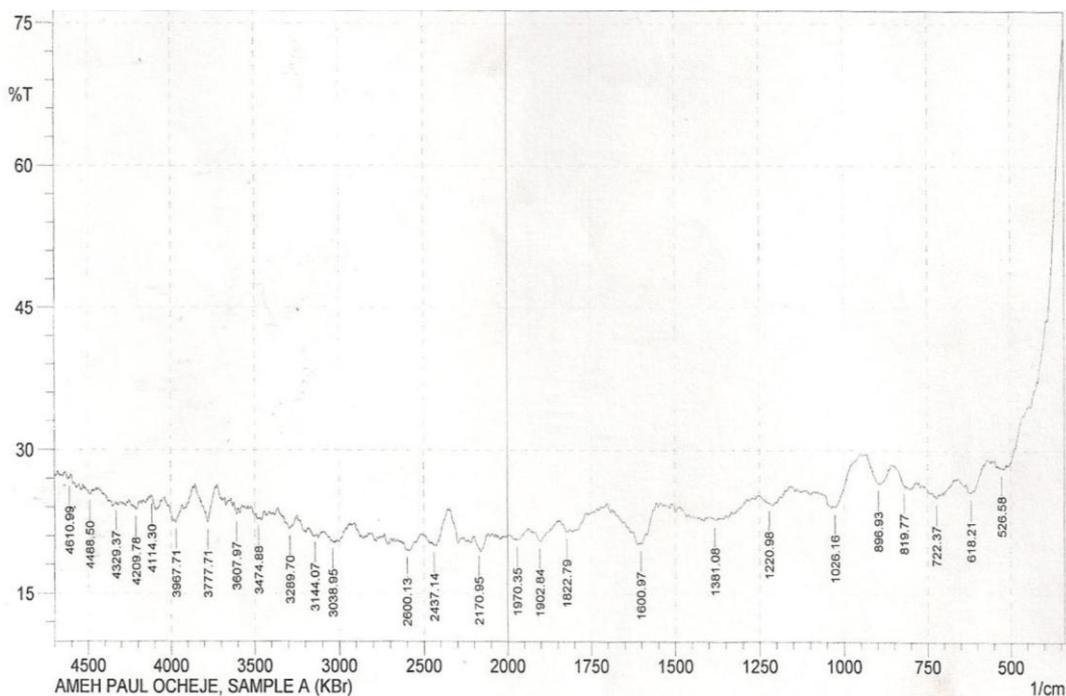


Figure 6. FTIR spectrum of the corrosion product of mild steel when AL gum was used as an inhibitor

Table 5. Peaks, wavelength, % concentration and assignment of functional group for FTIR adsorption by AL

Wave number (cm ⁻¹)	Intensity	Area	Assignments
606.63	23.32	1.828	Finger print
703.08	21.01	5.855	C-H bend
976.01	17.73	2.893	C-H bend, C-O
1055.10	17.66	2.177	C-O stretch
1152.51	17.63	37.590	C-O stretch
1270.17	17.97	2.155	C-O stretch
1402.30	17.23	10.248	C-H scissoring and bending
1620.26	18.98	89.409	Alkene C=C
1733.10	23.12	68.061	C=O stretch
2130.45	50.39	57.269	C-C triple bond
2920.32	18.33	26.596	C-H stretch
3083.31	18.31	5.673	OH stretch
3178.79	17.62	5.079	OH stretch
3281.02	18.42	2.123	-OH stretch
3373.61	18.40	4.244	-OH stretch
3578.07	20.03	11.956	-OH stretch

Table 6. FTIR spectrum of the corrosion product of mild steel in the presence of AL gum as an inhibitor

Peak (cm ⁻¹)	Intensity	Area (cm ²)	Assignment (functional group)
606.63	25.692	2.270	C-Br stretch
732.97	24.180	1.187	C-H oop due to aromatic compound
813.02	23.717	7.761	=C-H bend due to alkene
875.71	24.832	16.772	C-H oop due to aromatic compound
1024.24	24.905	29.009	C-O stretch
1108.14	25.823	5.663	C-O stretch
1444.73	23.468	3.032	C=C aromatic stretch
1620.26	21.586	18.437	C=C stretch due to alkene
1818.93	22.811	9.232	C=O stretch
1929.84	21.748	5.105	C-H stretch due to Phenyl ring substitution
2277.04	19.906	3.376	C≡C stretch
2645.46	18.790	7.671	OH stretch
2826.77	18.975	4.166	H-C=O:C-H stretch
3023.52	18.970	4.164	OH stretch
3277.17	21.100	2.603	OH stretch
3503.81	21.118	3.897	OH stretch
3610.86	21.871	9.422	OH stretch

From the results of analysis of the FTIR spectra of AL gum (Table 5) and that of the corrosion product (Table 6), it can be seen that the C-Br vibration at 606.63 cm⁻¹ and the C=C stretch at 1620.26

cm^{-1} were not shifted in the spectrum of the corrosion product but the C-H bend at 703.08 was shifted to 732.97 cm^{-1} , the C-O stretches at 1055.10 and 1152.51 were shifted to 1024.24 and 1108.14 cm^{-1} respectively, the C \equiv C stretch at 2130.45 was shifted to 2277.04 cm^{-1} , the OH stretches at 3083.31, 3281.02 and 3578.07 were shifted to 3023.52, 3277.17 and 3503.86 cm^{-1} . The shifts in frequencies indicate that there is interaction between AL gum and the surface of the metal [21]. However, the C-H bends at 703.08 and 976.01, the C-H scissoring/bending at 1402.30, the C-H stretch at 2920.32, the OH stretches at 3178.79 and 3373.02 cm^{-1} were missing in the spectrum of the corrosion products indicating that adsorption of AL gum on mild steel surface might have preceded through these functional groups [22]. Also, some new bonds were found in the spectrum of the corrosion product. This included the =CH bend at 813.02 cm^{-1} , C-H oop at 875.71 cm^{-1} , the C=C aromatic stretch at 1444.26 cm^{-1} , the C=O stretch at 1818.93 cm^{-1} , the C-H stretch at 1929.84 cm^{-1} and OH stretches at 2645.46, 2826.77 and at 3610.86 cm^{-1} . This also indicates that some new bonds were also formed through these functional groups [23].

CONCLUSION

From the results of our study, the following conclusions are made,

- (i) The major chemical constituents of AL gum are sucrose, n-hexadecanoic acid, 1-pentadecane carboxylic acid and 2,3-diphenyl-1,4-naphthoquinone
- (ii) AL gum is a good corrosion inhibitor for mild steel in acidic medium. The mechanism of inhibition of the corrosion of mild steel by AL gum is joint adsorption of the inhibitor on mild steel surface and via the formation of inhibitor-Fe complex
- (iii) The adsorption of AL gum on mild steel surface is exothermic, spontaneous and favours the mechanism of physical adsorption
- (iv) The use of AL gum as an inhibitor for the corrosion of mild steel in acidic medium is hereby advocated

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