

CORROSION INHIBITION OF COPPER IN HYDROCHLORIC AND TETRAOXOSULPHATE (VI) SOLUTIONS USING ALOE VERA BARBADENSIS GEL.

ABSTRACT

The inhibition of corrosion of copper in hydrochloric acid and tetraoxosulphate (VI) acid solutions by Aloe Vera gel at 303K- 353K has been investigated using the weight loss technique. Values of inhibition efficiency are in good agreement and are dependent upon the concentrations of the inhibitor used as well as the corrosion media. The corrosion rate increased with increase in temperature while the inhibition efficiency as well as surface area decreased with increase in temperature. The difference in inhibitive behaviour of the inhibitor in hydrochloric acid and tetraoxosulphate (VI) acid corrosion media has been explained in terms of the hydration of the ions of the corresponding acids vis-a-vis mode of adsorption on the copper surface. A first-order type of mechanism has been deduced from the kinetic treatment of the weight loss results and the process of inhibition is attributed to physical adsorption. The adsorption of the Aloe Vera gel on copper was found to obey the Langmuir adsorption Isotherm.

Keywords: Aloe Vera; Inhibition; Copper; Weight loss; Corrosion.

1.0 INTRODUCTION

Metals and its alloys are exposed to the action of acids in industrial processes which causes severe problems such as decrease in mass and corrosion of surfaces resulting in economic losses (Adebayo, 2004). Copper is one of the most essential metals owing to its large industrial applications. About 65% of copper that is produced is used for electrical applications. The important uses of copper include, use in power generation and transmission of electricity. It is used in transformers, motors, bush bars, generators, etc., to provide electricity through out the country, safely and efficiently (Li et al., 1998). It is also used in wiring, mobile phones and in the circuitry (Ahmed and Mohamed, 1989). It is generally a relatively noble metal; however, it is susceptible to corrosion by acids and strong alkaline solutions, especially in the presence of oxygen or oxidants (Jayaraman et al., 2007).

In order to reduce the corrosion of copper metal, several techniques have been applied. The use of inhibitors during acid pickling procedure is one of the most practical methods for protection against corrosion in acid media (Obot et al., 2011).

By definition, a corrosion inhibitor is a chemical substance that, when added in small concentration to an environment, effectively decreases the corrosion rate (Hackerman and Snaveley, 1984). The use of inhibitors is one of the best options of protecting metals against

corrosion. Corrosion inhibition is a surface process which involves the adsorption of the organic compounds on the metal surface (Abiola and Oforka, 2002).

The applicability of organic compounds as corrosion inhibitors for metals in acidic media has been recognized for a long time (Umoren, et al 2009). The existing data from their research revealed that most organic inhibitors act by adsorption on the metal surface. The adsorption of inhibitors occurs through heteroatoms such as nitrogen, phosphorus, sulphur, triple bonds or aromatic rings. These compounds which are adsorbed on the metallic surface block active corrosion sites (Obot and Egbedi, 2009).

Naturally occurring substances as inhibitors of acid cleaning processes has continued to receive attention as replacement for synthetic organic inhibitors which are sometimes toxic to the environment (Umoren et al., 2008 and Okafor.et-al., 2007). The greatly expanded interest on naturally occurring substances is attributed to the fact that they are cheap, readily available, renewable, ecologically friendly, and possess no threat to the environment. Literature has shown that that plant material such as Paradisi juice extract (Abiola et-al., 2004), aqueous extract of fenugreek leaves (Ehteram, 2007), and extract of Kalmegh (*Andrographis paniculata*) leaves (Ambrish et al., 2008), extract of *Musa acuminata* peel (Eddy et al., 2008), the root of ginseng, (Obot and Obi-Egbedi 2009) has been used to inhibit the corrosion of various metals, in a similar study Nnabuk et al., (2009), studied the ethanol extract of *Terminalia catappa* as a green inhibitor for the corrosion of mild steel in H_2SO_4 ; the inhibitive and adsorption properties of ethanol extract of *Terminalia catappa* for the corrosion of mild steel in H_2SO_4 were investigated using weight loss, hydrogen evolution, and infra red methods of monitoring corrosion. Ethanol extract of *T. catappa* was seen to be a good adsorption inhibitor for the corrosion of mild steel in H_2SO_4 .

Aloe barbadensis, one of 450 species of Aloe Vera is a succulent perennial herb which grows up to 160 cm tall, without stem or with a short stem up to 30 cm long, freely suckering and forming dense groups. Aloe Vera is an important medicinal plant which belongs to the family of Liliacea (Rajendran, 2007).

The gel, which constitutes the bulk of the leaf substance, serves as the water storage organ for the plant. This gel, which may be removed as a semi-solid "fillet" before processing, contains more than 200 different substances. Chief among these are polysaccharides, glycoproteins, vitamins, mineral and enzymes (Ekpandu, 2004).

Table 1: Phytochemical components of Aloe Vera gel.

Class	Compounds

Anthraquinones/anthrones	Aloe-emodin, aloetic-acid, anthranol, aloin A and B (or collectively known as barbaloin), isobarbaloin, emodin, ester of cinnamic acid
Carbohydrates	Pure mannan, acetylated mannan, acetylated glucomannan, glucogalactomannan, galactan, galactogalacturan, arabinogalactan, galatoglucoarabinomannan, pectic substance, xylan, cellulose
Chromones	8- <i>C</i> -glucosyl-(2'- <i>O</i> -cinnamoyl)-7- <i>O</i> -methylaloediol A, 8- <i>C</i> -glucosyl-(<i>S</i>)-aloesol, 8- <i>C</i> -glucosyl-7- <i>O</i> -methyl-(<i>S</i>)-aloesol, 8- <i>C</i> -glucosyl-7- <i>O</i> -methyl-aloediol, 8- <i>C</i> -glucosyl-noreugenin, isoaloeresin D, isorabaichromone, neoaloesin A
Enzymes	Alkaline phosphatase, amylase, carboxypeptidase, catalase, cyclooxygenase, cyclooxygenase, lipase, oxidase, phosphoenolpyruvate carboxylase, superoxide dismutase
Inorganic Compounds	Calcium, chlorine, chromium, copper, iron, magnesium, manganese, potassium, phosphorous, sodium, zinc
Miscellaneous including organic compounds and lipids	Arachidonic acid, γ -linolenic acid, steroids (campesterol, cholesterol, β -sitosterol), triglicendes, triterpenoid, gibberillin, lignins, potassium sorbate, salicylic acid, uric acid
Non-essential and essential amino acids	Alanine, arginine, aspartic acid, glutamic acid, glycine, histidine, hydroxyproline, isoleucine, leucine, lysine, methionine, phenylalanine, proline, threonine, tyrosine, valine

Proteins	Lectines, lectin-like substance
Saccharides	Mannose, glucose, L-rhamnose, aldopentose
Vitamins	B1, B2, B6, C, β -carotene, choline, folic acid, α -tocopherol

Source: Talmadge *et al.*, 2004.

Aloe Vera gel as commonly called is organic in nature and can be used in the production of green corrosion inhibitors. It is one of the natural inhibitors which have an inhibitive action on the corrosion of metals. Aloe Vera gel is colorless mucilaginous gel obtained from the parenchymatous cell in fresh leaves of Aloe Vera. It contains various active compounds such as salicylates, magnesium lactate, acemannan, lupeol, campesterol, sterol, linolenic, aloctin and anthraquinones (Al-Turkustani et al 2010).

This present work was designed as contribution to the growing interest on environmentally benign corrosion inhibitors to assess the corrosion inhibitive properties of Aloe Vera gel in hydrochloric and sulphuric acid solutions on Copper at 303K- 353K and to carry out Kinetic studies of the reaction.

2.0 Experimental

2.1 Material preparation

Copper sheets used for this work were bought from Steel village Market, Port Harcourt. The chemical composition of the copper metal are Cu 95.0% and Zn 5.0%. The copper sheets were cut into rectangular specimens of dimension 4cm by 2cm by 0.1cm containing a small hole of about 2mm diameter near the upper edge to allow passage of thread for suspension of the copper coupons into the test solution. The coupon were degreased in absolute ethanol, rinsed with double distilled water and dried in acetone. The treated coupons were then stored over calcium chloride in moisture free desiccators before use for corrosion studies to prevent contamination.

2.2 Preparation of Aloe Vera gel.

The procedure for the preparation of the Aloe Vera gel is similar to that reported by Abiola and James, 2009. Stock solutions of the Aloe Vera gel was prepared by cutting open the Aloe leaves with a clean cutting material to expose the gel content of the leaf. 350ml of the gel was squeezed out from 697.9g of the Aloe leaves. The gel was then allowed to filter through a clean white handkerchief in order to get the pure liquid. From the stock solution; the Aloe gel test solutions were prepared at concentrations of 10, 5, 4, 3, 2 and 1 %v/v for solutions of HCl and H₂SO₄ respectively. Thus the highest concentration of 10%v/v was prepared by diluting 10ml of Aloe gel extract with 90ml of HCl or H₂SO₄. Other concentrations (5%, 4%, 3%, 2% and 1%. vol/vol) were prepared by diluting 5ml, 4ml, 3ml, 2ml and 1ml of Aloe gel extract with 95ml, 96ml, 97ml, 98ml and 99ml of HCl or H₂SO₄ respectively).

2.3 Weight Loss Method.

98 The copper metal, sheet of 0.1cm thickness and 98.79% purity used for weight loss measurement
 99 were of dimension 4cm by 2cm .The total geometric surface area of the coupons exposed to the
 100 corrodent was 16.00cm²(since both faces of the coupons were immersed in the corrodent).The
 101 average weight of the copper coupons was 4.48 - 4.58g.

102 Fifteen 250ml beakers, which separately contained 100ml of 10%,5%,4%,3%,2% 1% v/v and
 103 2M of HCl and H₂SO₄ which served as the control respectively were maintained at 303K,313K,
 104 323K, 333K and 353K constituting two set of experiments for each acid. Previously weighed
 105 copper coupons were each suspended in each beaker through a 0.1cm hole in diameter.

106 The copper coupons in HCl and H₂SO₄ acid solutions at 303K, 313K, 323K, 333K and 353K
 107 were retrieved at 24hours interval progressively for 168 hours (7days) respectively.

108 The copper coupons retrieved were immediately washed thoroughly in water, rinsed in acetone
 109 and dried in an oven to constant weight before final weighing.

110 The coupons retrieved were immersed in a solution of 20% sodium hydroxide to terminate the
 111 corrosion reaction. The coupons was scrubbed with brittle brush several times inside water to
 112 remove corrosion products, dried in ethanol and acetone and then weighed.

113 The weight loss was calculated in grams as the difference between the initial weight prior to
 114 immersion, and weight after removal of the corrosion product.

115 Triplicate experiments were performed in each case and the mean values reported to the nearest
 116 0.0001g on an AB54 AR analytical weighing balance.

117 The inhibition efficiency (%I) was computed using equation 2.1:

118 i. Inhibition efficiency at 303K, 313K, 323K, 333K and 353K.

$$119 \quad \% \text{Inhibition Efficiency} = \frac{\Delta W_B - \Delta W_1}{\Delta W_B} \times 100. \quad \dots\dots\dots (2.1)$$

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121 Where ΔW_B and ΔW_1 are the weight loss of copper metal coupons without and with inhibitor
 122 respectively.

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126 The corrosion rate (W) was computed using the equation 2.2:

127 ii. Corrosion rate at 303K, 313K, 323K, 333K and 353K.

128 a) Corrosion rate (g/cm²/day) = $\frac{\Delta W}{\dots\dots\dots}$(2.2)

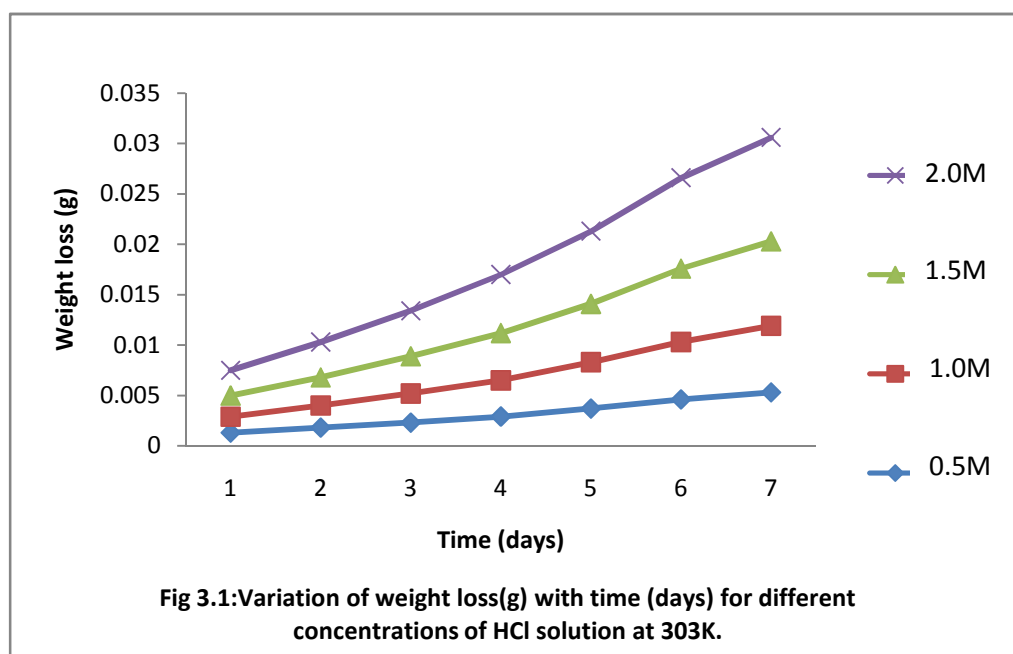
Area (cm²) x day

Where ΔW is the change in weight or weight loss (g), A is the area of the specimen (cm²) and t is the exposure time in days.

3.0 Results and Discussion.

3.1 Effect of Corrodent Concentration on Copper Corrosion.

Fig.3.1 and Fig. 3.2 shows that copper corrodes in different concentrations of HCl and H₂SO₄ respectively, since there was a decrease in original weight of copper. The corrosion is attributed to the presence of water, air and H⁺, which accelerates the corrosion process. The corrosion of copper in HCl and H₂SO₄ increases with the concentration of the acid and time. Similar results were obtained at 313K, 323K, 33K and 353K. This observation is attributed to the fact that the rate of chemical reaction increases with increasing concentration (James, et al., 2007).



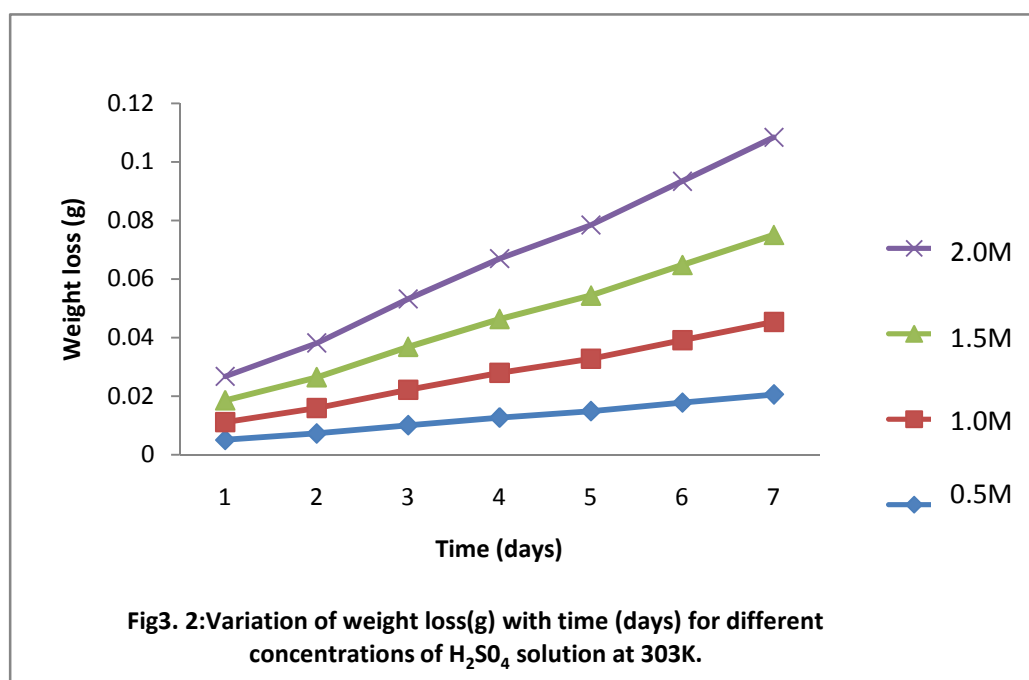


Table 2: Values of corrosion rate ($gdm^{-2}day^{-1}$) and inhibition efficiency (% I) for copper in 2M HCl.

Inhibitor Concentration (%)	Inhibition efficiency (% I)					Corrosion Rate ($gdm^{-2}day^{-1}$) $\times 10^{-4}$				
	303K	313K	323K	333K	353K	303K	313K	323K	333K	353K
1.0	37.77	24.71	18.75	14.07	11.40	1.75	4.18	5.68	7.25	8.25
2.0	40.10	28.08	20.53	16.29	13.42	1.68	4.00	5.56	7.06	8.06
3.0	44.44	35.95	33.92	26.66	21.47	1.56	3.56	4.62	6.25	7.31
4.0	53.33	43.82	37.50	32.59	28.85	1.31	3.12	4.37	5.68	6.62
5.0	64.44	55.05	49.10	43.70	37.58	1.00	2.5	3.56	4.75	5.81
10.0	71.11	65.16	60.71	53.33	48.99	0.812	1.93	2.75	3.93	4.75

151 Table 3: Values of corrosion rate ($\text{gdm}^{-2}\text{day}^{-1}$) and inhibition efficiency (% I) for copper in 2M
152 H_2SO_4 .

Inhibitor Concentration (%)	Inhibition efficiency (% I)					Corrosion Rate ($\text{gdm}^{-2}\text{day}^{-1}$) $\times 10^{-4}$				
	303K	313K	323K	333K	353K	303K	313K	323K	333K	353K
1.0	30.66	21.77	16.29	10.95	7.59	3.25	6.06	7.06	8.12	9.12
2.0	34.66	25.01	18.51	13.69	10.75	3.06	5.81	6.87	7.87	8.81
3.0	37.33	33.87	28.14	21.23	17.72	2.93	5.12	6.06	7.18	8.12
4.0	45.33	40.32	35.55	27.37	25.94	2.56	4.26	5.43	6.62	7.31
5.0	56.00	50.8	45.92	39.04	34.81	2.06	3.81	4.56	5.56	6.43
10.0	64.10	58.87	54.07	47.94	46.20	1.68	3.18	3.87	4.75	5.31

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154 3.2 Effect of Temperature.

155 The electrochemical nature of corrosion causes the corrosion rate to increase exponentially with
156 rise in temperature, according to Arrhenius-type dependence. Thus the influence of temperature
157 on the corrosion behavior of Cu in 2M HCl and 2M H_2SO_4 in the absence and presence of Aloe
158 gel of varying concentrations were investigated by weight loss method at 300K, 313K, 323K,
159 333K and 353K. In examining the effect of temperature on the corrosion inhibition process, the
160 apparent activation energies (E_a) were calculated from the Arrhenius equation (Obot et-al.,
161 2011).

$$162 \quad E_a = \frac{2.303RT_1T_2(\log k_2/k_1)}{T_2 - T_1} = \frac{\text{Jmol}^{-1}}{1000} = \text{KJmol}^{-1} \dots\dots\dots (2.3)$$

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164 Where R = is the gas constant in Joule (J)

165 T = Absolute temperature in Kelvin

166 R = $8.314 \text{ Jmol}^{-1} \text{ K}^{-1}$

167 K_1 and K_2 are the rate constants at T_1 (300K) and T_2 (313K) respectively or T_1 (313K) and T_2
 168 (323K) and so on for the rest of the temperatures.

169 Table 4: Activation energies of Aloe Vera gel exudate on the surface of copper for HCl and
 170 H_2SO_4 .

Activation Energy $KJmol^{-1}$		Activation Energy $KJmol^{-1}$		Average Activation Energy $KJmol^{-1}$			
HCl		H_2SO_4					
303K-313K	323K-313K	303K-313K	323K-313K	303K-313K		323K-313K	
60.71	30.43	57.72	11.83				
66.25	33.88	56.51	13.01				
63.04	24.67	46.44	17.45	65.88	51.21	31.60	15.08
69.18	30.79	48.61	16.35				
67.75	37.02	49.29	14.13				
68.35	32.85	48.72	17.72				

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 172 The Activation energies were calculated from equation 2.3 and recorded in Table 4. The values of
 173 E_a ranged from 65.88 to 31.60 KJ/mol and 51.21 to 15.08 KJ/mol for HCl and H_2SO_4
 174 respectively. These values are less than the threshold value of 80 KJ/mol required for chemical
 175 adsorption (Eddy, 2009), hence the adsorption of Aloe Vera gel on the surface of copper
 176 supports the mechanism of physical adsorption.

177 3.3 The Inhibitive Effect of Aloe Vera Gel.

178 The values of percentage inhibition efficiency and corrosion rate obtained from weight loss
 179 method at different concentrations of the Aloe gel at 300K-353K are summarized in Tables 2 and
 180 3 above. Inspection of the data in the tables above reveals that the addition of Aloe gel decreases
 181 as the corrosion rate of copper in HCl and H_2SO_4 acidic solutions. From Figures 3.3 and 3.4
 182 below it is also observed that the inhibition efficiency increases as the concentration of the Aloe
 183 gel is increased.

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3.4 The Active Component Responsible for the Inhibitory Action of Aloe Vera Gel.

The observed inhibition of the Aloe gel could be attributed to the adsorption of the components on the copper coupon surface. The formed layer, of the adsorbed molecules, isolates the metal surface from the aggressive medium leading to decreasing of the corrosion rate. The chemical components of the exudate of Aloe Vera was studied and reported in literature (Al-Turkustani et al 2010). As earlier stated in the introductory remarks, Aloe Vera gel is a complex mixture of as salicylates, magnesium lactate, acemanan, lupeol, campesterol, sterol, linolenic, aloctin and anthraquinones. Other components include saponins, glycosides and vitamins, which implies that they contain heteroatoms such as oxygen and nitrogen in its molecule. The corrosion inhibition of copper may be attributed to adsorption of Aloe gel exudate onto the metal surface. Owing to complex chemical composition of the exudate, it is quite difficult to assign the inhibitive effect to a particular constituent. Further investigation using surface analytical techniques will enable the characterization of the active materials in adsorbed layer and assist in identifying the most active ingredients. It is worthy to note that in acidic media, nitrogen atoms accept protons from the acid solution and form cations which electrostatically are attracted to the pre-adsorbed Cl^{-1} or SO_4^{-2} anions on the copper coupon surface (Khaled, 2008).

The following components with structures contain electron rich oxygen and nitrogen that could serve as good active ingredients which are responsible for corrosion inhibition in Aloe Vera gel.

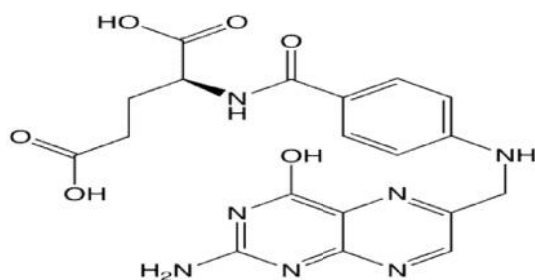


Fig 3a: Folic Acid

(A Vitamin)

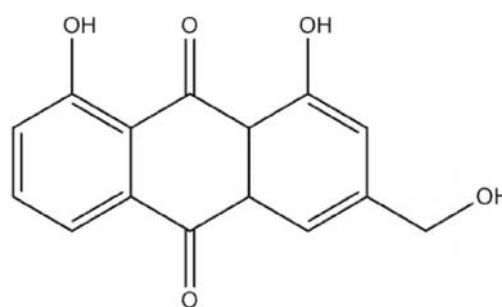
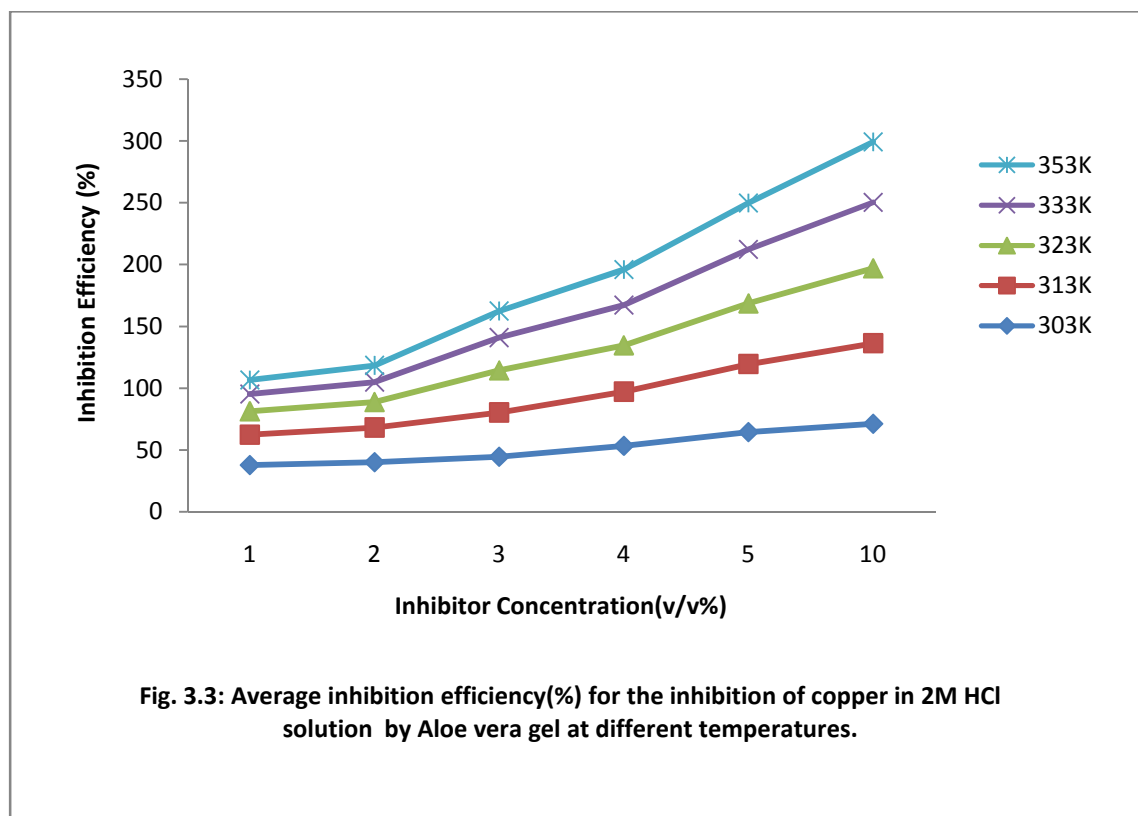
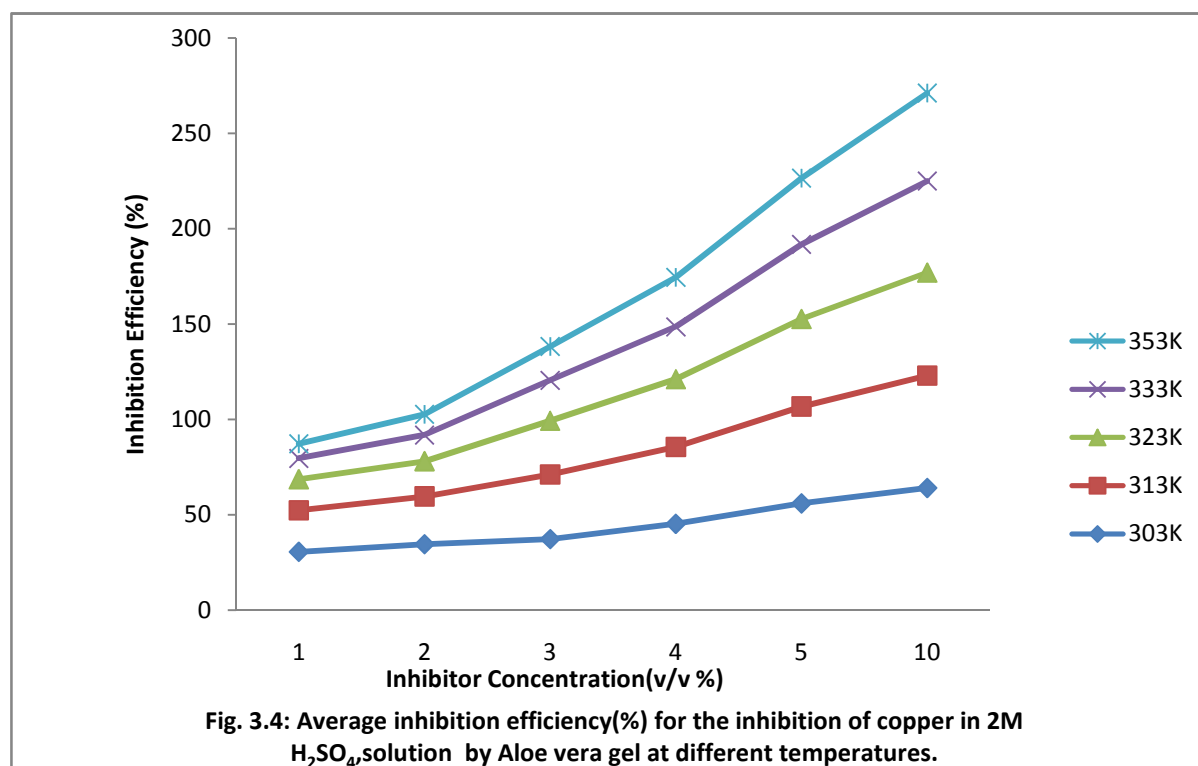


Fig 3b: Aloe Emodin ($\text{C}_{15}\text{H}_{10}\text{O}_5$)

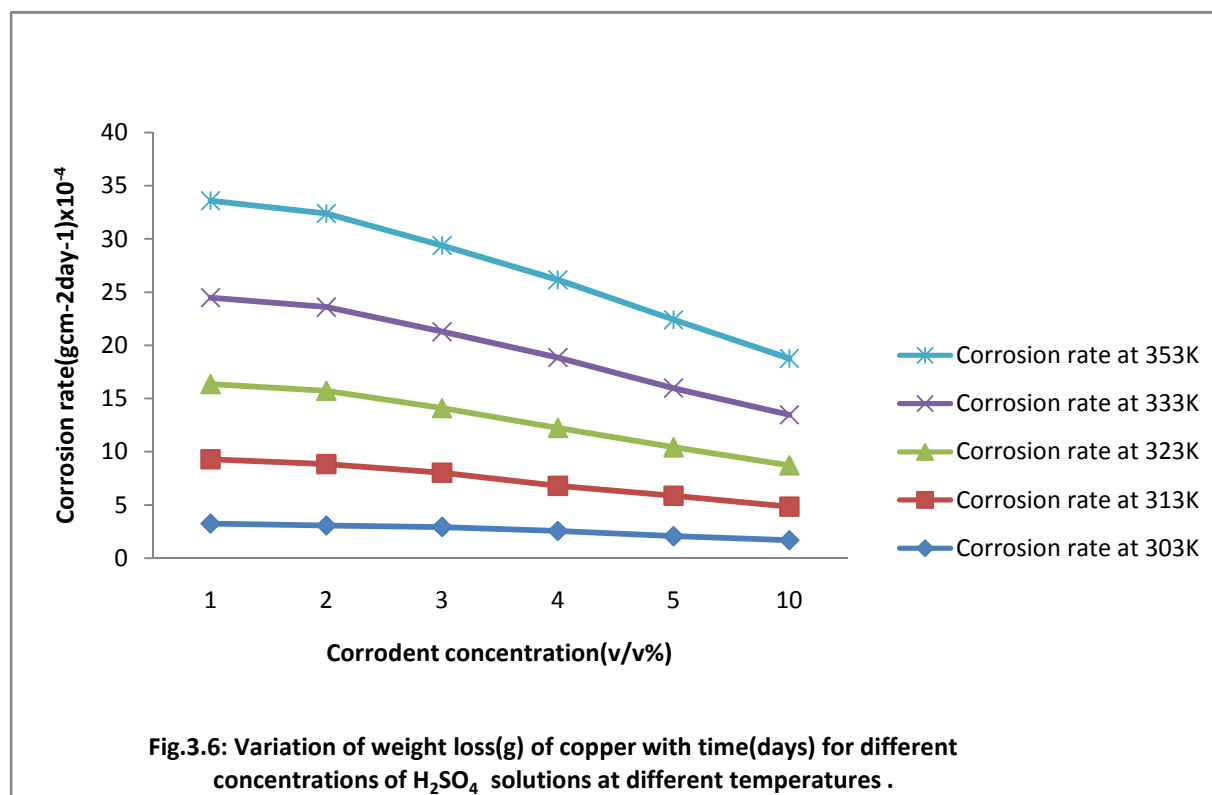
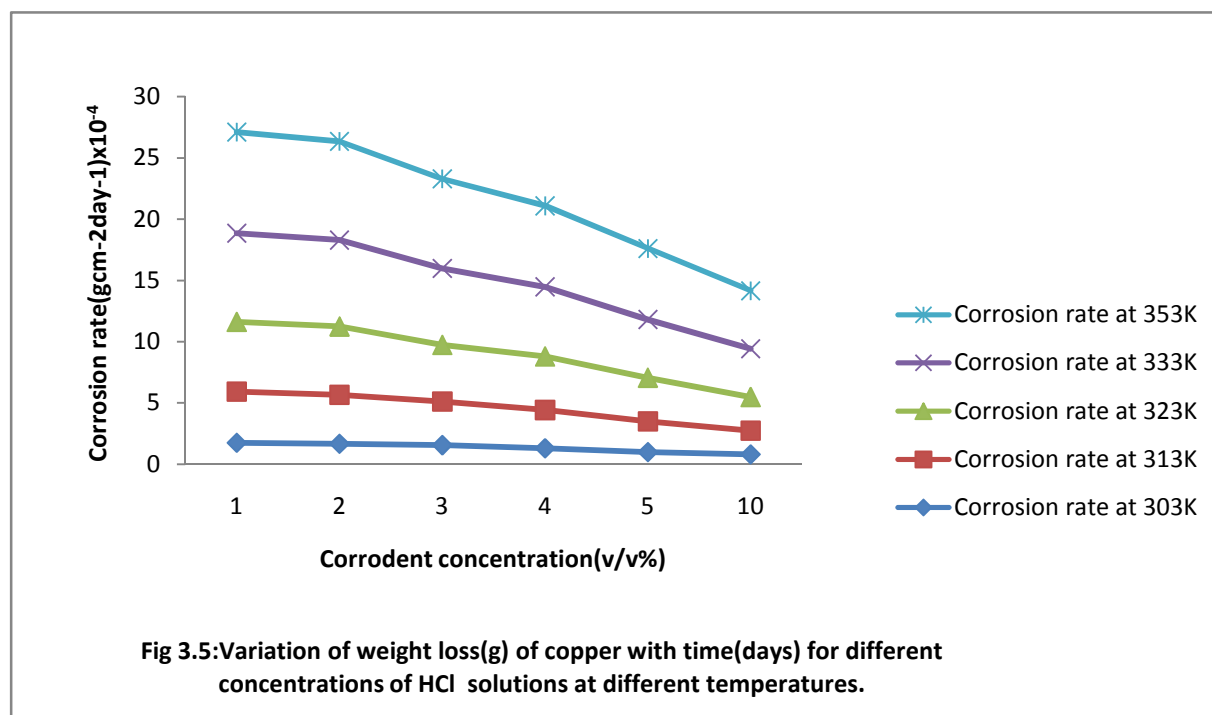
(An Anthraquinone)



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3.5 Adsorption Consideration

The effectiveness of organic compounds as corrosion inhibitors can be ascribed to the adsorption of molecules of the inhibitors through their polar functions on the metal surface. Surface coverage values were evaluated from the weight loss measurements assuming direct relationship between inhibition efficiency and surface coverage as follows: % I = $\theta \times 100$. The surface coverage values were fitted to different adsorption isotherm models and best results fitted the Langmuir isotherm.

Langmuir isotherm is given by the expression:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{e.c.a}} + C_{inh} \dots \dots \dots (2.4)$$

Where θ is the surface coverage, C_{inh} is the concentration, $K_{e.c.a}$ is the equilibrium constant of the adsorption process. The plot of C/θ against C is shown in Figures 3.7 and 3.8

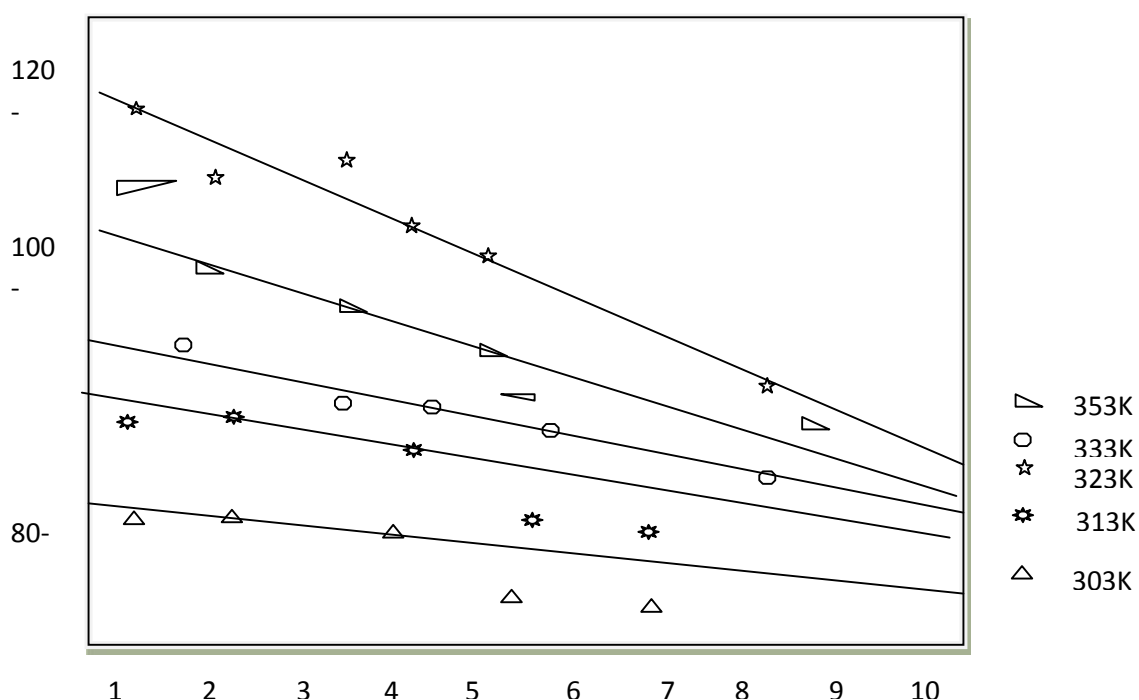


Fig.3.7: Langmuir adsorption isotherm (plotted as C/θ) versus C for the inhibition of copper in 2MHCl solution by Aloe Vera gel.

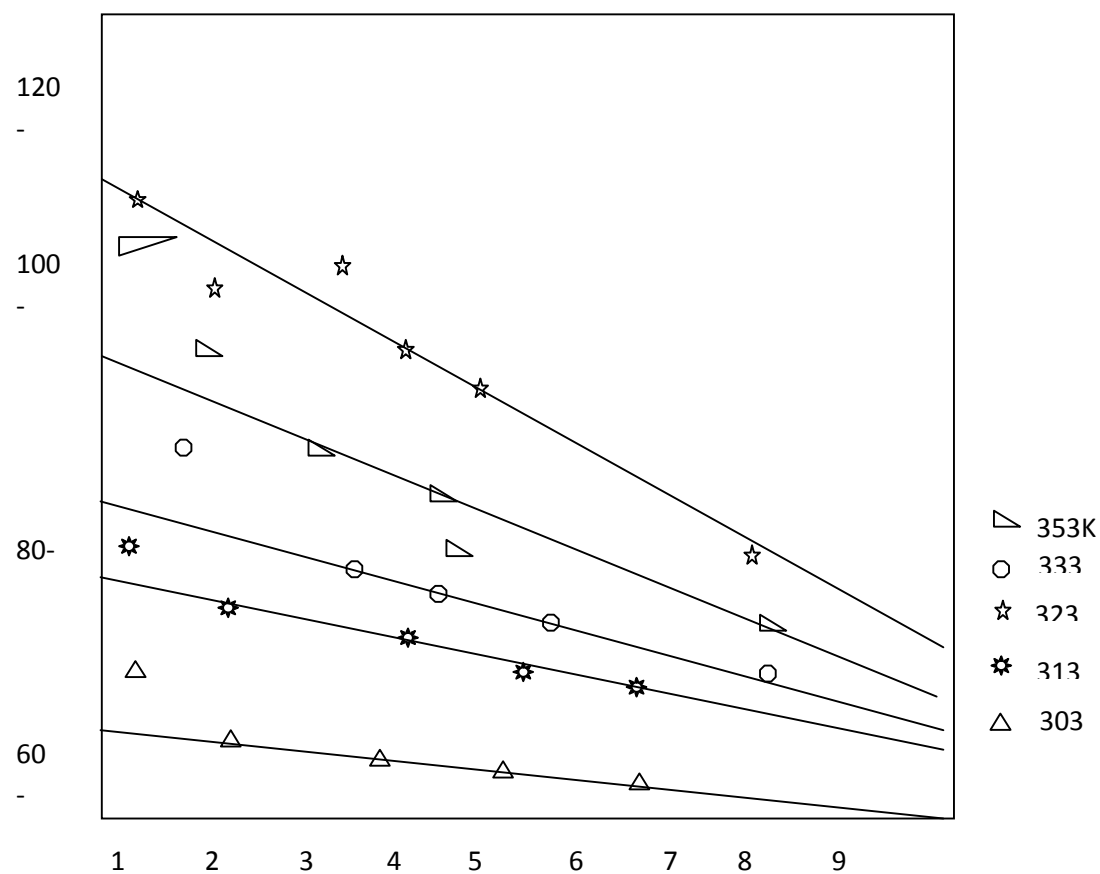


Fig.3.8: Langmuir adsorption isotherm (plotted as C/θ) versus C for the inhibition of copper in $2\text{MH}_2\text{SO}_4$ solution by Aloe Vera gel.

The plot at the various temperatures studied which gives a straight line fit the adsorption of Aloe Vera gel on the copper metal to Langmuir adsorption isotherm.

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4.0 Conclusion

From the results obtained from this study, the following conclusions have been made:

- (i) The rates of corrosion of copper in HCl and H₂SO₄ acid solutions without inhibitor increases with increase in acid concentration and temperature range studied.
- (ii) The methods used show that Aloe Vera gel inhibited the corrosion of copper to a reasonable extent in both HCl and H₂SO₄ acid solutions.
- (iii) The additive used in this investigation (Aloe Vera gel) inhibited the acid corrosion of copper in HCl and H₂SO₄ to different degrees.
- (iv) The percentage inhibition efficiency (%) of the inhibitors is in the order of HCl > H₂SO₄, that is there was better corrosion inhibition in HCl than H₂SO₄.
- (v) Kinetic treatment of the results of the corrosion of copper in HCl and H₂SO₄ in both inhibited and uninhibited reactions confirm a first order type of mechanism.
- (vi) The values of the activation energy obtained in the presence and absence of inhibitors are below 80KJmol⁻¹ (see Table 4). The inhibition is probably due to the adsorption of the inhibitor which was physically adsorbed on the surface of the copper metal.
- (vii) The adsorption fits well to the Langmuir adsorption Isotherm.
- (viii) The results obtained suggest that Aloe Vera gel is a corrosion inhibitor for copper metals in HCl and H₂SO₄ and they can be used to replace toxic and nonbiodegradable inhibitors.

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330 **Corrections made:**

331 Tables are now Table 1: Phytochemical components of Aloe vera gel.

332 Table 2: Values of corrosion rate ($\text{gdm}^{-2}\text{day}^{-1}$) and inhibition efficiency (% I) for copper in 2M
333 HCl.

334 Table 3: Values of corrosion rate ($\text{gdm}^{-2}\text{day}^{-1}$) and inhibition efficiency (% I) for copper in 2M
335 H_2SO_4 and

336 Table 4: Activation energies of Aloe Vera gel exudate on the surface of copper for HCl and
337 H_2SO_4 .

338 serially.

339 While figures are fig.3.1, fig. 3.2, fig.3.3, fig.3.4, fig.3.5, fig.3.6, fig. 3.7 and fig.3.8 serially.

340 Equations are equation (2.1), (2.2), (2.3) and (2.4) serially.

341 Structures are Fig. 3a: Folic acid and Fig. 3b: Aloe Emodin