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**Corrosion Inhibitive Potential Of Hibiscus Sabdariffa Calyx Extract For Low Carbon Steel** In 0.5M H<sub>2</sub>SO<sub>4</sub> Acid Solution

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#### Abstract

The inhibitive potential of Hibiscus Sabdariffa calyx extract on low carbon steel corrosion in 0.5M H<sub>2</sub>SO<sub>4</sub> have been investigated by weight loss method which is considered more informative than other laboratory methods. The studies were carried out using extracts obtained from 5-25g dried calyx powder. The test coupons were immersed in the corroding media at the time intervals of 24-168hours. The results obtained showed that the concentration of the inhibitor in the corrodent impacted differently on the test coupons. The corrosion rate was found to decrease while inhibition efficiency increase as the concentration of the extract was increased. The maximum inhibition efficiencies of 95.01% and 94.41% were obtained at 96hours exposure time from the extract of 20g and 25g dried calyx powder respectively. The results has clearly shown that the extract has the inhibiting capacity for reducing the corrosion of low carbon steel in the acidic medium.

### **Keywords**: Hibiscus Sabdariffa, inhibitive potential, low carbon steel and $H_2SO_4$ .

### Introduction

Corrosion is responsible for the huge loss of materials occuring everywhere, every moment in the world involving billions of dollars annually. Studies of the cost of corrosion in Australia, Great Britain, Japan, and other countries have also been carried out. In each country studied, the cost of corrosion is approximately 3-4% of the Gross National Product (Kruger in Uhlig, 2000). The economic factor is a very important motivation for much of the current research in corrosion. Corrosion is one of the most serious destructive agents, and is also, one of the major technological problems of modern society. Though a lot of scientific understanding of the many phases of corrosion has been developed, it is a very complex phenomenon as there are number of complex variables responsible for it (Singh, 2011).

Corrosion is a natural process, because in nature, most metallic elements (except noble metals) occur in the most stable state having low energy, normally as oxides. Energy is used to convert these oxides to metallic state to be useful to mankind against this natural tendency. Thus, the metals are in higher energy state, and try to revert to an oxidized state. The main reason of the reactions (corrosion) between metals and their environments is the tendency to decrease the energy of the system as a result of the corrosion reactions.

Corrosion has gained so much importance that an engineer, while selecting an alloy for an application, also takes into account the corrosion resistance of the alloy in the specific environment. Corrosion and its prevention is a problem of great importance to the engineering industry. The practical solution of the problems of corrosion, lies in controlling it by reducing the rate at which the corrosion reactions proceed. There are various methods by which corrosion can be controlled, among which is the used of inhibitors. Inhibitor is a chemical substance that when added in small concentration to an environment, effectively decreases the corrosion rate (Revie and Uhlig, 2008). There are several mechanisms that may account for the effectiveness of inhibitors. Some react with and virtually eliminate a chemically active species in the solution (such as dissolved oxygen). Other inhibitor molecules attach themselves to the corroding surface and interfere with either the oxidation or the reduction reaction, or form a very thin protective coating (Callister, 2006).

The inorganic and synthetic chemical inhibitors have been used to a great extent to suppress the corrosion of

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metals in many environments. There are certain limitations that are associated with these type of inhibitors, they are toxic and may cause environmental damage. The pure synthetic chemicals are costly and non bio-degradable as such their disposal creates pollution problems (Fontana, 1986 and Suleiman et al, 2011). Inhibitors of plant extracts are environmentally friendly, bio-degradable, non toxic, readily available and of potentially low cost. This probably, could have accounted for the recent research interest by investigators to established their potential as a viable alternatives to other existing inhibitors for corrosion control.

Available researches has shown that extensive work have been carried out on different varieties of plant extracts as corrosion inhibitor for metals/ alloys in various agressive environment. It is realized that the environmental conditions for which some of the inhibitors of plant origin have been used is limited. Suleiman et al (2011), investigated the potential of using acacia sengalensis extract as corrosion inhibitor for carbon steel in 0.5M H<sub>2</sub>SO<sub>4</sub> medium, and concluded that the weight loss of mild steel in the blank solutions were higher than those obtained for the solutions containing various concentrations of acacia sengalensis. Idenyi et al (2010), studied the corrosion characteristics of aluminium-manganese alloys in salinated vegetable extract (Bitter leaf) environment, and found that, the corrosion of aluminium alloys is influenced by the alloys impurity, environment type, concentration and duration of exposure time in the given media. Asuke et al (2009), investigated the potential of palm exudate (palm wine )as corrosion inhibitor for Al-Si alloy in caustic soda solution, and discovered that palm wine exhibited moderate inhibition potential for Al-Si alloy in 0.5M NaOH solution. Asuke et al (2009), studied the used of propargyl as corrosion inhibitor for Al-5%Si-15%SiC composite in 0.5M NaOH solution, and concluded that propargyl is a moderate inhibitor for the composite, with a maximum inhibition efficiency of 59.23% at 30°C and 1.5% v/v concentration. Monticlli et al (2004) in Asuke et al (2009) studied corrosion and corrosion inhibition of alumina particulate/aluminium alloys metal matrix composite (MMC) in neutral chloride solution and found that, among tungsten and molybdenum-containing inorganic salts tested as corrosion inhibitors, only ammonium tetrathiotungstate exhibited good inhibiting properties, towards the AA2014-based MMC. Oguzie (2008) studied the corrosion inhibitive effect and adsorption bahaviour of Hibiscus Sabdariffa extract on mild steel in 2MHCl and 2MH<sub>2</sub>SO<sub>4</sub> solutions using gasometric technique and found that Hibiscus Sabdariffa calyx

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extract decreased the corrosion reaction in both acid media and the inhibition efficiency increased with extract concentration. Subhashini in Buchweishaija (2009) investigated the inhibition effect of the seeds extracts of Alfa alfa (Aa), A denanthera pavonina phaseolus lunatus (Pl), psophocarpus (Ap), tetragonolobus (Pt) and sesbania grandiflora (Sg). The seeds extracts were tested as corrosion inhibitors of mild steel in 1MHCl and 0.5M H<sub>2</sub>SO<sub>4</sub> with various immersion time and concentratons, using mass loss, polarization and electrochemical impedance spectroscopy. The analysed surfaces of the coupons tested showed decrease in the corrosion rate with increase in concentration of the extract and immersion time. Yawas (2008) studied the corrosion inhibition characteristics of Parkia Clappertoniana Kay and Mangifera Indica on carbon steel in 0.5M H<sub>2</sub>SO<sub>4</sub> using weight loss and mechanistic techniques. The studies were carried out at different temperatures and various concentration of the inhibitors. The results obtained revealed that the corrosion effiencies of extracts increases with increased in the concentration of the inhibitors. The maximum inhibition efficiency of each extract was obtained at 5%. Ogoko, et al., (2009) reported that the initial step in any corrosion inhibition process is the adsorption of the inhibitor on the surface of the metal, and thus, suppress the metal dissolution and reduction reactions. Although, the stability of the inhibitor film formed over the metal surface depends on some physiochemical properties of the molecules related to its functional groups such as aromaticity, type of the corrosive medium and nature of the interaction between the inhibitors with the d-orbital vacant of iron.

The focus of the present study is to investigate the inhibitive potential of Hibiscus Sabdariffa calyx extract as a cheap and environmentally friendly corrosion inhibitor for low carbon steel in 0.5M H<sub>2</sub>SO<sub>4</sub> medium by weight loss method. Several methods have been used to study the relative resistance of different metals/alloys in certain corroding media and conditions. The weight loss method though long and tedious process is by far more informative than many of the laboratory tests (Khanna, 2008 and Rajput, 2010)). Low carbon steels are the most widely used engineering materials for structural applications due to its availability, ease of fabrication, low cost and good tensile strength. However, low carbon steels have inadequate corrosion resistance when it comes in contact with acid solutions during acid cleaning, transportation of acid, de-scaling, storage of acids and other chemical processes. Inhibitors has been found to be effective in retarding the corrosion of low carbon

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steels after dissolution of the fouling oxides (Pierre, 2000).

Hibiscus Sabdariffa (Rosselle) also known as guinea sorrel or Indian sorrel is a medicinal herb cultivated for its seeds, calyx and leaf, and is grown in the tropics, subtropics and other parts of the world (Dalziel, 1973) but is utilized beyond these areas of cultivation globally. Rosselle is used in preparation of local, non alcoholic beverage, tea, jam, industrial wine, and marmalade (Mounigan, et al., 2007). In Northern Nigeria, the production of a non alcoholic drink called Zobo that is prepared from the red calvees is popular. The leaves and the calyces are eaten as vegetable after cooking.

The phytochemical analysis of Hibiscus Sabdariffa calyces extensively carried out by Obidoa, et al (2011) using the Harborne method revealed the presents of flavonoids, glycosides, tannins, acidic compounds, steriods, proteins, carbohydrates and reducing sugars. Carbohyhrates, flavonoids and tannins were found to be present in higher concentration. Recent study by Ekpe et al., (1994) has shown that the presents of flavonoids and tannins in many of the plant extract accounts for their ability in retarding the corrosion rate of some metals/alloys in various agressive environments.

#### **Materials, Equipment and Methods**

The materials and equipment used for the study were: Hibiscus Sabdarrifa calyx, distilled water, 0.5M H<sub>2</sub>SO<sub>4</sub> solution, emery papers of various grades, acetone, measuring cylinders, beakers, mild steel, desiscator, air gun dryer and analytical weighing balance.

### **Preparation of the inhibitor**

The Hibiscus Sabdariffa calyces were purchased from Ogbaru main market, Onitsha. The calyces were carefully sorted to remove any impurities, then dried under the Sun, after which they were grounded into fine powder. The fine powdered calvces were then measured into varying concentrations ranging from 5g, 10g, 15g, 20g and 25g. Each of this concentration was dissolved in a 250ml of distilled water in a 500ml beaker and allowed to stay over night after which they were filtered to remove residues present. Each concentration was then mixed with already prepared 0.5M H<sub>2</sub>SO<sub>4</sub> solution and were labelled for identification.

### **Experimental procedures**

The commercial grade of the low carbon steel used in the study was collected from the central store of the National Metallurgical Training Institute,

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Onitsha. The chemical composition of the as-received low carbon steel is shown in Tables 1. A total of 42 test coupons with average surface area of 8.61cm<sup>2</sup> with dimension 2.1x4.1x6cm were used for the study. There were divided into seven (7) groups for the six (6) media. Each group was immersed in sufficient volume of the appropriate medium to cover the coupons for a period ranging from 24 – 168hrs, for a maximum of seven (7) days. A set of coupons from the media were withdrawn, washed in distilled water, clean with acetone, dried and the final weight recorded. This was done at the intervals of 24hrs, 48hrs, 72hrs, 96hrs, 120hrs, 144hrs and 168hrs respectively. From the results obtained, the corrosion rate and inhibition efficiency were calculated.

#### Table 1: Chemical composition of the as-received low carbon steel

Elements	% Composition
С	0.156
Si	0.220
Mn	0.393
Р	0.025
S	0.004
Cr	0.085
Ni	0.011
Мо	0.002
Al	0.031
Cu	0.053
Со	0.003
Ti	0.001
Nb	0.002
V	0.002
W	0.032
Pb	0.001
В	0.002
Sn	0.003
Zn	0.002
As	0.005
Bi	0.001
Ca	0.001
Се	0.003
Zr	0.001
La	0.002
Fe	Balanced

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### **Corrosion rate (mpy) and inhibitor efficiency (%)**

The corrosion rate was determined from the standard expression for measurement of corrosion rate in mils per year (mpy), (Fontana, 1987).

$$Mpy = \frac{534W}{DAT} \qquad \dots \qquad (1)$$

Where; W = weight loss (g), D = density of the material  $(g/cm^3)$ , T = time of exposure (hours), A = total surface area ( $cm^2$ ).

### Inhibitor efficiency

The inhibition efficiency (IE) was evaluated using the expression Revie, et al (2008).

 $IE = (rate_{no Inhib.} - rate_{with inhib.})$ 

- X 100% ------ (2)

## rate<sub>no inhib.</sub>

Where; rate<sub>no Inhib.</sub> and rate<sub>with inhib</sub> are corrosion rate without and with inhibitors respectively.

### **Results and discussion**

The results of the study are presented in Tables 2-7. The visual observation of all the coupons in the studied media, revealed changes in the colour of the coupons from bright shiny surfaces to dull ones. Pits were observed on the surfaces of all the coupons indicating corrosion attack by the acidic medium.

Table 1show the variation of corrosion rate with time of exposure of the steel coupons immersed in  $0.5 \text{ M H}_2\text{SO}_4$  solution. It is clearly evident that the coupons immersed in the blank acidic solution exhibited the highest corrosion rates compared to those immersed in various concentration of the inhibitor. The was a progressive increase in the corrosion rate as the time of exposure was increased from 24hours to 96hours, a pronouced decrease with time at 120hours, and a subsequent increase in the corrosion rate with time at 144hours and 168hours respectively. The loss of chemical reactivity (passivity) observed at 120hours time of exposure of the coupon could be attributed to the formation of surface film (protective barrier) on the metal surface which was relatively stable at 120hours, and relatively nonprotective formed oxide film with increased in the exposure time from 144-168hours could be that the oxides may have been growing under tension leading to the discontinous, porous film possessing low protective properties. This is in agreement with several authors (Fontanna, 2007, Kutz, 2002, Ihom, et al., 2008, Ogundare et al., 2011).

Coupon	Time (Hours)	Initial Weight	Final Weight	Weight Loss	Corrosion Rate
	(1100115)	(g)	(g)	(g)	(Mpy)
A <sub>1</sub>	24	38.2	34.6	3.6	1.193
$A_2$	48	38.1	30.8	7.3	1.209
A <sub>3</sub>	72	38.2	27.2	11	1.215
A <sub>4</sub>	96	38.1	21.9	16.2	1.342
A <sub>5</sub>	120	38.1	30.0	8.1	0.537
A <sub>6</sub>	144	38.2	18.6	19.6	1.060
A <sub>7</sub>	168	38.1	15.5	22.6	1.070

Table 2: Variation of corrosion rate with time of exposure in 0.5M H<sub>2</sub>SO<sub>4</sub>

Tables 3-7 show the variation of corrosion rates with time of exposure in various concentration of the extract. The results indicated that the concentration of the inhibitor in the corroding medium impacted differently on the steel coupons. The pronouced decrease in the corrosion rates of the test coupons as the inhibitor concentration in the acidic medium was increased implies that the more the concentration of the inhibitor in the acidic medium the more readily the inhibitor molecules get adsorbs on the metal surface, and the smaller the residual anodic areas becomes, thus, favouring increased anodic polarization and ultimately passivation. The degree of inhibition was found to depend on the concentration of the extract in the acidic environments. The variation in the corrosion rate with time of exposure of the steel coupons in the corrodents containing various concentration of the extract could be attributed to the different organic compounds in the inhibitor which has been proven to posses high inhibiting capacity, such as flavonoids, glycosides, tannins, acidic compounds, steriods, proteins, carbohydrates, reducing sugars and pigments (Obidoa, et al., 2011 and Oguzie, 2008). The net adsorption of the organic constituents on the metal surfaces creates a protective barrier that isolates the metal surface from the corrodents. The adsorption mechanisn of Hibiscus Sabdariffa extract on metal surface as described by Oguzie, et al., (2008) is a mixed type, where the molecular as well as the protonated organic species in the extract contribute to the inhibiting action.

The inhibition efficiency of inhibitors of plant origin has been reported (Asuke, et al., 2009, ) to be dependent on the degree surface coverage of the organic constituents on the metal surface. The inhibition efficiency of the extract in the 0.5 M H<sub>2</sub>SO<sub>4</sub> solution was found to increase as the concentration of the extract were increased from 5-25g. The maximum inhibition efficiencies of 95.01% and 94.41% were obtained at 96hours exposure time, from the extract of 20g and 25g of the calyx powder respectively. The variation in the inhibition efficiency with time of exposure could probably be as a result of the variation oxidation and reduction rate with time thereby impacting residual stresses on the absorbed molecules, on the metal surfaces, thus affecting their stability.

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Coupon	Time	Initial	Final	Weight	Corrosion	Inhibition
	(Hours)	Weight	Weight	Loss	Rate	Efficiency
		(g)	(g)	(g)	(Mpy)	(%)
<b>B</b> <sub>1</sub>	24	38.1	37.1	1.0	0.331	72.25
<b>B</b> <sub>2</sub>	48	38.1	35.3	2.8	0.464	61.62
<b>B</b> <sub>3</sub>	72	38.2	34.5	3.7	0.409	66.34
<b>B</b> <sub>4</sub>	96	38.1	33.4	4.7	0.389	71.01
<b>B</b> <sub>5</sub>	120	38.2	36.2	2.0	0.133	75.23
<b>B</b> <sub>6</sub>	144	38.2	30.3	7.9	0.436	58.87
<b>B</b> <sub>7</sub>	168	38.1	29.1	9.0	0.426	60.19

Table 3: Variation of corrosion rate with time of exposure of 5g Hibiscus Sabdariffa calyx powder extract in 0.5MH2SO4

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Coupon	Time	Initial	Final	Weight	Corrosion	Inhibition
1	(Hours)	Weight	Weight	Loss	Rate	Efficiency
		(g)	(g)	(g)	(Mpy)	(%)
<b>C</b> <sub>1</sub>	24	38.2	37.5	0.7	0.232	80.55
$C_2$	48	38.1	36.8	1.3	0.215	82.22
C <sub>3</sub>	72	38.1	37.3	0.8	0.088	92.76
$C_4$	96	38.2	34.6	3.6	0.298	77.79
C <sub>5</sub>	120	38.2	36.5	1.7	0.113	78.96
C <sub>6</sub>	144	38.1	32.8	5.3	0.293	72.36
C <sub>7</sub>	168	38.2	31.8	6.4	0.303	71.68

Table 4: Variation of corrosion rate with time of exposure of 10g Hibiscus Sabdariffa calyx powder extra	act in 0.5M
H2SO4	

Table 5: Variation of corrosion rate with time of exposure of 15g Hibiscus Sabdariffa calyx powder extract in 0.5M  $H_2SO_4$ 

			$H_{2}SO_{4}$			
Coupon	Time	Initial	Final	Weight	Corrosion	Inhibition
	(Hours)	Weight	Weight	Loss	Rate	Efficiency
		(g)	(g)	(g)	(Mpy)	(%)
<b>D</b> <sub>1</sub>	24	38.1	37.7	0.4	0.133	88.85
$D_2$	48	38.2	37.0	1.2	0.199	83.54
D <sub>3</sub>	72	38.1	37.4	0.7	0.077	93.66
$D_4$	96	38.1	35.1	3.0	0.248	81.52
$D_5$	120	38.1	34.6	3.5	0.232	56.80
$D_6$	144	38.2	33.4	4.8	0.265	75.00
$D_7$	168	38.1	32.4	5.8	0.275	74.30

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Coupon	Time	Initial	Final	Weight	Corrosion	Inhibition
Coupon	(Hours)	Weight	Weight	Loss	Rate	Efficiency
	(110tarb)	(g)	(g)	(g)	(Mpy)	(%)
E <sub>1</sub>	24	38.2	38.0	0.2	0.066	94.47
$E_2$	48	38.1	37.7	0.4	0.066	94.54
E <sub>3</sub>	72	38.2	37.4	0.8	0.088	92.76
$E_4$	96	38.1	37.3	0.8	0.067	95.01
$E_5$	120	38.1	36.9	1.2	0.080	85.10
E <sub>6</sub>	144	38.2	35.1	3.1	0.171	83.87
E <sub>7</sub>	168	38.1	33.8	4.4	0.208	80.56

Table 6: Variation of corrosion rate with time of exposure of 20g Hibiscus Sabdariffa calyx powder extract in 0.5M H2SO4

Table 7: Variation of corrosion rate with time of exposure of 25g Hibiscus Sabdariffa calyx powder extract in 0.5M H<sub>2</sub>SO<sub>4</sub>

Coupon	Time	Initial	Final	Weight	Corrosion	Inhibition
	(Hours)	Weight	Weight	Loss	Rate	Efficiency
		(g)	(g)	(g)	(Mpy)	(%)
$F_1$	24	38.2	37.9	0.3	0.099	91.70
$F_2$	48	38.2	37.7	0.5	0.083	93.13
$F_3$	72	38.1	37.0	1.1	0.121	90.04
$F_4$	96	38.2	37.3	0.9	0.075	94.41
$F_5$	120	38.1	37.2	0.9	0.060	88.83
$F_6$	144	38.1	36.1	2.0	0.110	89.62
$F_7$	168	38.2	35.5	2.7	0.128	88.04
F <sub>7</sub>	168	38.2	35.5	2.7	0.128	88.04

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### Conclusion

The analysed results of the tested coupons showed decrease in the corrosion rates and increase in the inhibition efficiencies with increased in concentration of the extract and immersion time. The low corrosion rates recorded is indicative of the inhibitor capability to effectively decreases the corrosion of low carbon steel in  $0.5M H_2SO_4$  acidic solution. The inhibitive behaviour of the extract is believed to be strongly contributed by the molecular and organic species in the inhibitor.

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