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# EXTRACTS OF <u>PREMATURE</u> DACRYODES EDULIS SEED AND THEIR CORROSION INHIBITION POTENTIALS ON MILD STEEL IN 1 M HCI PRESENCE

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## ABSTRACT

Gravimetric and electrochemical techniques (weight loss, effects of difference in temperatures, and electrochemical verifications) were carried out on premature *Dacryodes edulis* seed extract in IM HCI as corrodent at different temperatures and time (24 hours immersion time at room temperatures, 1 hour immersion time at  $40^{\circ}$ C and 1 hour immersion time at  $70^{\circ}$ C). The results showed that inhibition efficiency increased with increase in concentration of inhibitor. The result from the potentiodynamic polarization and electrochemical impedance also suggest that the premature *Dacryodes edulis* seed extract is a cathodic inhibitor and the mechanism of physisorption and chemisorption were observed from results obtained from the calculation of heat of adsorption, as well as from analysis of the results provided from the electrochemical experiments. Hence premature *Dacryodes edulis* seed extract has corrosion inhibitor.

## INTRODUCTION

According to *Corrosionpedia* (2018), acidic corrosion is a gradual destruction and wearing away of a material (e.g., metal) due to acidic compounds present in the environment. The acids may be those coming from the soil, industrial atmosphere, water or toxic chemicals that react with the material they come in contact with. Amitha and Bharathi (2011) defined corrosion as the deterioration of a metal by chemical attack or reaction with its environment.

When metals are exposed to aggressive environments such as, the use of acid solutions for pickling, chemical and electrochemical etching of metal, industrial acid cleaning, cleaning of oil refinery equipment, oil well acidizing and acid descaling, they usually lead to loss of the metal due to corrosion (Ating *et al.*, 2010, Bishir *et al.*, 2017). It has been estimated that approximately 5% of any industrialized nation's income is spent on corrosion prevention, maintenance and replacement of products lost or contaminated due to corrosion reactions (Abiola, 2002). Industrial operations such as oil well acidification, acid pickling, acid cleaning and acid descaling are operating in acidic environment (Swathi *et al.*, 2017).

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Fernando *et al.*, (2014) described corrosion inhibitors as chemical substances which are added in small concentrations to an environment and retard the occurrence of corrosion reactions. The process of using inhibitors to control corrosion is termed corrosion inhibition. Some investigations have in recent times been made into the corrosion inhibiting properties of natural products of plant origin and they have been found to generally exhibit good inhibition efficiencies (Okafor and Ebenso, 2007). African pear (*Dacryodes edulis*) seed has always been suspected as one whose extracts are possible corrosion inhibitors for metals including mild steal.

*Dacryodes edulis* is a fruit tree native to Africa, sometimes called safou (Cameroon), atanga (Equatorial Guinea and Gabon), ube (Nigeria) (en.wikipedia). However, many authors have given this fruit differing interpretations and descriptions which to a large extent is a function of their research locality, as well as their area of research. According to Vivien and Foure (1996), *Dacryodes edulis* is an oleiferous fruit native to Southern Nigeria and perhaps to Cameroon. Its current geographic range is from Central Africa to Sierra Leone in the West, to Uganda in the East, and to the Northern part of Angola in the South (Troupin, 1950). It is a dioecious, shade loving, evergreen tree. It is an indigenous fruit tree in the Gulf of Guinea and Central African Countries (Troupin, 2000), but due to the popularity of the nutritious fruit for consumption, the plants cultivation or distribution extended to Sierra Leone, Uganda, Angola, Zimbabwe and Nigeria. The plant does not grow in the wild and this make its natural area of distribution to be obscure (Verheji, 2002).

Okafor (1983) in Nigeria identified two main variants of *Dacryodes edulis*: var. edulis and var. parvicarpa. The fruit of var. edulis is larger and the tree has stout, ascending branches, while those of var. parvicarpa has smaller fruit and slender, drooping branches. Youmbi et al. (1989) identified two morphological parts in markets in Cameroon.

The generic name "Dacryodes" is derived from the Greek word "Dakruon" meaning "Tear", referring to resin droplets on the bark surface of its members, while "edulis" means edible, signifying the value of the nutritious fruit in the plants cultivation (Anonymous, 2010). The plant belongs to the family Burseraceae whose members are characterized by an ovary of 2 to 5 cells, prominent as in ducts in the bark, wood and intrastaminal disk (Chunduff, 2004). According to (Verheji, 2002), the genus "Dacryodes" consist of about 40 species but (Rehm, 2004) indicated 80 species to cover subspecies, varieties, forms and cultivars. However, two varieties are acceptable: Var. edulis and Var. parvicarpa whose conical fruit is smaller with the pulp. Var. edulis exhibit verticillate or subverticillate branching, while the branching is slender and opposite or bifurcate in Var. parvicarpa (Okafor, 2003; Kapsue and Tchiegang, 2006; National Research Council, 2006). Dacryodes edulis has common names like African pear in English, Eben in Ibibio, Ube in Igbo, Safoutier in French (Burkill, 2005). But since the premature Dacryodes edulis is our product of interest, it is worthy to note their names too. In English, it is known as premature African pear, Ikpong Eben in Ibibio and Ube uvuru ocha in Igbo languages respectively.

In the present work, the corrosion inhibition action of **premature** *Dacryodes edulis* seed extracts on mild steel is to be investigated in 1M HCl.

## EXPERIMENTAL

#### **Preparation of Specimens**

before the commencement of corrosion studies, the mild steel sample was prepared by cutting into pieces of the same measured dimensions of 1 mm x 50 mm x 40 mm. Holes were drilled in the coupons to enable the insertion of a hanger and thereafter, wet polished with silicon carbide abrasive paper (from grade 800 - 1600), degreased in absolute ethanol, rinsed in acetone, weighed and stored in moisture-free desiccator.

#### Method of extraction

For the stock solution used in this experiment, the plant extracts were prepared by reflux boiling of 20 g of the dried, ground, filtered, and weighed sample of *Dacryodes edulis* for 3 hours in a round-bottomed flask, allowed to cool at room temperature, and filtered using an instantly weighed white handkerchief; and stored in a white plastic bottle. From this stock solution, inhibitor test solutions were prepared with concentrations of 100, 500, and 1000 mg L<sup>-1</sup> mixed with the corrodent. The electronic weighing instrument used is OHAUS brand.

#### Weight Loss Determination

Gravimetric titration analysis was conducted on mild steel coupons of stated dimensions. In the weight loss experiment, four reaction vessels were labeled BL for blank, and  $A_1 - A_3$  for samples containing 100, 500, and 1000 mg L<sup>-1</sup> masses of the inhibitor in 1.0 M of the corrodent. The mild steel coupons were carefully weighed and given appropriate tagging to avoid mix up. They were then suspended (under total immersion conditions) in the reaction vessels containing 200 ml (in a 250ml beaker each) of the test solutions with the aid of twine and broom sticks. All tests were made in aerated solutions. In the first experiments for each corroding sample, the coupons were retrieved after 24 hours.

The other experiments were for the effects of elevated temperatures in which the enabling ranges were for 40 and 70 °C, respectively, for 1 hour. In each case, the retrieved coupons were scrubbed with bristle ("smokers") brush under water, dipped in absolute ethanol, rinsed with water, dried in acetone, and reweighed.

Each test was run in duplicate and the mean values of the measured parameters presented.

The weight loss was a simple difference between the initial mass and the final mass retrieved from the corrodent solutions.

The evaluated results were for

#### (i) Inhibition efficiency, $\eta$ (I.E.) in percentage, given by

$$\eta\% = (1 - \frac{\Delta M_{SP}}{\Delta M_{IN}}) \times 100 \dots I$$

where  $\Delta M$  = mass loss; SP = sample; IN = inhibitor; and

#### (ii) Corrosion rate, $\rho$ , given by

$$\rho = \frac{KW}{DAT} \dots 2$$

where K = rate constant (534 mpy; mils per year, 1 mil =  $10^{-3}$  in); W = mass loss in mg; D = density of the mild steel coupon (7.85 g cm<sup>-3</sup>); T = immersion time(h); A = exposed area of the coupon (0.1911cm<sup>2</sup>).

### Electrochemical measurements

Electrochemical tests were conducted in a conventional three-electrode corrosion cell, using a V3 Potentiostat/Galvanostat, coupled to a PC, running on the Powersuite and Powersine software. A graphite rod and saturated calomel electrode (SCE) served as counter and reference electrodes respectively. Test metal specimens were fixed in epoxy resin with a surface area of  $1 \text{ cm}^2$  exposed to the test solution. Measurements were in aerated and unstirred solutions at the end of 1800s of immersion at  $30\pm1$  °C. Each test was run in triplicate and the mean values of the measured parameters presented.

Impedance measurements were carried out using signal amplitude perturbation of 5 mV at the corrosion potentials ( $E_{corr}$ ) and frequency range 100 kHz - 0.1 Hz. The resulting impedance data were analyzed using Zsimpwin 3.0 software. Potentiodynamic polarization measurements were performed at a scan rate of 0.333 mV/s and potential range  $\pm 250$  mV versus corrosion potential.

#### **RESULTS AND DISCUSSION**

#### **Gravimetric Results**

#### **KEY FOR TABLES 1 – 5**

 $C_1$  = Concentration of inhibitor  $\Delta m$  = Mass loss

I.E	=	Inhibition efficiency
C.R	=	Corrosion rate
Log C.R	=	Log of corrosion rate

 Table 1: Table of average values for 24 hours immersion at room temperature for both blank and inhibited

solutions							
$C_1(mg/L)$	$\Delta m(g)$	I.E. (%)	C.R. Mm/yr				
Blank	0.0868	-	1.2874				
100	0.0517	40.4377	0.7668				
500	0.0340	60.8294	0.5042				
1100	0.03225	62.8456	0.4783				

From the table of values above, the values of mass loss ( $\Delta m$ ) and corrosion rate (C.R) decreased as the inhibitor concentration (C<sub>1</sub>) increased but the inhibition efficiency (I.E) increased as the concentration of inhibitor (C<sub>1</sub>) increased. This obviously is a direct proportionality in that if more inhibitor solution is added, then corrosion rate will decrease as that is exactly the purpose of the addition of the inhibitor.

Table 2: Table of values showing result for 1 hour immersion at temperature of 40<sup>0</sup>C for both blank and inhibited solutions

$C_1$ (mg/L)	Δm(g)	I.E. (%)	C.R. Mm/yr
Blank	0.0179	-	6.3718
100	0.01445	19.2737	5.1437
500	0.0136	24.0223	4.8411
1100	0.01315	26.5363	4.6809

Table 3: Table of values showing result for 1 hour immersion at temperature of 70<sup>o</sup>C for both blank and inhibited solutions

blank and minored solutions						
$C_1 (mg/L)$	<b>Δm</b> (g)	I.E. (%)	C.R. Mm/yr			
Blank	0.19505	-	69.4315			
100	0.10245	47.4750	36.4689			
500	0.0891	54.3194	31.7167			
1100	0.07095	63.6247	25.2559			

From the values in Tables 2 and 3 above respectively, the mass loss ( $\Delta$ m) and corrosion rate (C.R) decreased from the value realized when the solution was blank, and the decrease continued as the concentration of inhibitor (C<sub>1</sub>) increased from 100 to 1100 mg L<sup>-1</sup>, and the values of inhibition efficiency (I.E) increased as the concentration of inhibitor (C<sub>1</sub>) increased correspondingly. It can be stated rightly that the addition of the inhibitor decreased the total surface area available for the corrosion reaction to take place.

Table 4: Table of values showing the result for 1 hour immersion at temperatures of 40<sup>°</sup>C and 70<sup>°</sup>C with their corresponding log of corrosion rate for both blank and inhibited solutions

<b>C</b> <sub>1</sub>	40 <sup>°</sup> C				70°C			
(mg L <sup>-1</sup> )	Δm (g)	I. E. (%)	C.R. (mm yr <sup>-1</sup> )	Log (CR)	Δm (g)	I.E. (%)	C.R. (mm yr <sup>-1</sup> )	Log (CR)
Blank	0.0179	-	6.3718	0.8042	0.19505	-	69.4315	1.8416
100	0.01445	19.2737	5.1437	0.7113	0.10245	47.4750	36.4689	1.5619
500	0.0136	24.0223	4.8411	0.6849	0.0891	54.3194	31.7167	1.5013
1100	0.01315	26.5363	4.6809	0.6703	0.07095	63.6247	25.2559	1.4024

The need to calculate the log of the corrosion rate is to ascertain the differences observed in the value of corrosion in another mathematical expression with the bid to determine how significant these differences might be. From the values realized, it is clear that the significance is appreciable and is a proof that the presence of this inhibitor in the experiment is effective enough to be regarded as a recognized and proven one.

Further Calculations made were:

1) **Apparent Activation Energies (Ea):** Activation energy may be defined as the minimum energy required to start a chemical reaction. It can also be described as the minimum energy which must be available to a chemical system with potential reactants to result in a "chemical reaction". Thus, the calculation of apparent activation energies for mild steel corrosion was deduced from Arrhenius equation:

2) Heat of Adsorption ( $Q_{ads}$ ): The heat of adsorption,  $Q_{ads}$ , is the heat evolved when a given amount of a substance is adsorbed. It is another thermodynamic parameter which describes the adsorption mechanism and is connected to the degree of surface coverage,  $\theta$ , ( $\eta\% = 100 \times \theta$ ), through this relation:

Where  $\theta_1$  and  $\theta_2$  are the values of the degree of surface coverage of temperature  $T_1$  and  $T_2$  respectively

Table 5: Table of values showing the result of heat of adsorption  $(Q_{ads})$  and activation energy  $(E_a)$  for mild steel corrosion at temperatures of 40<sup>o</sup>C and 70<sup>o</sup>C respectively

$C_1$	40°C		70°C		
$(mg L^{+})$	Activation	Heat of Adsorption	Activation	Heat of	
	Energy (kj/mol)	(kj /mol)	Energy (kj/mol)	Adsorption (kj /mol)	
Blank	242.03491	-	506.06346	-	
100	288.04964	-0.13035	415.00329	0.11860	
500	342.32741	-0.19852	398.26654	0.11802	
1100	345.21239	-0.19257	357.13323	0.14053	

From the table of values above, the values of activation energy  $(E_a)$  and heat of adsorption  $(Q_{ads})$  at temperature  $40^{\circ}C$  were less than the values of activation energy (Ea) and heat of adsorption  $(Q_{ads})$  at temperature of  $70^{\circ}C$ . The heats of adsorption (Qads) at temperature of  $40^{\circ}C$  were negative values while that of  $70^{\circ}C$  were positive values. These observations are significant in that they are indicators of the nature of adsorption reactions taking place that are the precursors of the resultant outputs.

#### Potentiodynamic polarization measurement (PDP)

Potentiodynamic polarization curves for mild steel in 1 M HCl containing different concentrations of premature *Dacryodes edulis* seed extracts at 30 °C are depicted in Figure 1.

The changes observed in polarization curves after addition of the inhibitor is the criteria to classify inhibitors as cathodic, anodic, or mixed-type inhibitor. This Potentiodynamic polarization experiment was undertaken to establish the difference in the effects of premature *Dacryodes edulis* seed extracts on the anodic and cathodic corrosion reactions. The values of the corrosion current density in the absence  $(i_{corr,bl})$  and presence of inhibitor  $(i_{corr,bl})$  were used to estimate the inhibition efficiency from polarization data as follows:

I.E. % = 
$$\left(\frac{I_{corr\ (bl)} - I_{corr\ (in\ hi)}}{I_{corr\ (in\ hi)}}\right) \times 100.....6$$

#### Where I<sub>corr(h)</sub> and I<sub>corr(inh)</sub> represents the corrosion current density in the absence and presence of the inhibitor, respectively.

Potentiodynamic polarization curves for mild steel in 1 M HCl containing 100 and 1000 mg/L, respectively, of *Dacryodes edulis* at 30 °C are represented in Figure 1 below. The metal coupon dissolved rapidly and there was no form of resort to retardation within the studied potential range. The behavior of the polarization curves in 1 M HCl environment (Figure 1) is an indication that the presence of premature *Dacryodes edulis* seed extracts as inhibitor shifts the cathodic and anodic curves towards the area of lower current densities, just as the addition of premature *Dacryodes edulis* seed extracts into the acidic solution has only slight noticeable effect on the corrosion potential ( $E_{corr}$ ) in each case. Results as shown in Table 6 reveal that the highest displacement in  $E_{corr}$  value in the studied acidic solutions was less than 85 mV, an indicator that premature *Dacryodes edulis* seed extracts can be regarded as a mixed type inhibitor.

The data in Table 6 show that the corrosion current density decreased significantly in the presence of *Dacryodes edulis* inhibitor compared to the uninhibited solution and also decreased with an increase in the concentration of the extract. The inhibition efficiency was calculated using the equation:



FIGURE 1: Potentiodynamic polarization curves of mild steel in 1 M HCl in the absence and presence of premature Dacryodes edulis seed extract

 Table 6: Polarization parameters for mild steel in 1M HCI in the absence and presence of premature Dacryodes edulis seed extract

System	E <sub>corr</sub> (mV)	j <sub>corr</sub> (µA/cm <sub>2</sub> )	IE (%)
Blank (1 M HCl)	÷ 455.1	269	
100 mg L <sup>-1</sup>	÷ 483.1	39.9	85.2
1000 mg L <sup>-1</sup>	÷ 489.3	25.9	90.4
0			

Electroche mical	System	$R_{s} (\Omega cm^{2})$	$R_{ct} (\Omega cm^2)$	N	IE (%)
Impedance	1  M HCl	1.659 1.817	54.2 98 7	0.89	- 34 5
Spectroscop y	1000 mg/L AP	2.318	262.6	0.89	81.5
Table 7:					

Electrochemical impedance parameters for mild steel in 1M HCI in the absence and presence of premature *Dacryodes edulis* seed extract.

The electrochemical impedance spectroscopy measurements was carried out in order to reveal the kinetics of the electrochemical processes taking place at the metal/solution interface, and how they were modified by premature *Dacryodes edulis* seed extract inhibitor.



FIGURE 2: Impedance spectra of mild steel in 1 M HCl in the absence and presence of premature Dacryodes edulis seed extract

Electrochemical Impedance Spectroscopy was also employed to study the corrosion response of mild steel in 1M HCl in the presence and absence of the inhibitor. Figure 2 represents the Nyquist plots of mild steel specimens in 1 M HCl. The plots showed clearly that the impedance response of metal specimens had a marked difference in the presence and absence of the inhibitor. The semicircles showed slight irregularity which may be attributed to the roughness or non homogeneous nature of the metal surface (Hassan *et al.*, 2007). The capacitance loop intersects the real axis at higher and lower frequencies. At high frequency end the intercept corresponds to the solution resistance (Rs) and at lower frequency end corresponds to the sum of Rs and charge transfer resistance (Rct). The difference between

the two values gives Rct. The value of Rct is a measure of electron transfer across the exposed area of the metal surface and it is inversely proportional to rate of corrosion (Kashman *et al.*, 1969).



#### 4.2 Discussion

Tables 1 – 4 showed the corrosion parameters in 1M hydrochloric acid containing various concentrations of premature *Dacryodes edulis* seed extract with different exposure time and temperature. The result from different tables reveal that the values of loss in mass ( $\Delta$ m) and corrosion rate (C.R) significantly decreased with the increase in inhibitor concentration (C<sub>1</sub>) and the inhibition efficiency (I.E %) increased with the increase in inhibitor concentration (C<sub>1</sub>).

Also, the role of temperature on the corrosion rate of each of the gravimetric analysis was noted. In 24 hours immersion time at room temperature, the corrosion rate is lower (slow) than that of 1 hour immersion time at  $40^{\circ}$ C and the corrosion rate at 1 hour immersion time at  $70^{\circ}$ C. (i.e. rate of corrosion in 24 hours immersion time at room temperature < rate of corrosion in 1 hour immersion time at  $40^{\circ}$ C < rate at corrosion in 1 hour immersion time at  $70^{\circ}$ C.) This result shows that the ability of the inhibitor to prevent corrosion depends on the concentration of the inhibitor in the corroding medium and temperature with different immersion time. This is mainly due to the fact that concentration, temperature and time are factors that affect the rate of any chemical reaction in general and corrosion in particular.

Similarly, the result from the calculation of heat of adsorption  $(Q_{ads})$  and activation energies  $(E_a)$  of mild steel at temperatures of 40°C and 70°C respectively are shown in table 5. The result presented the values of activation energy (E<sub>a</sub>) and heat of adsorption ( $Q_{ads}$ ) at temperature of 40<sup>o</sup>C to be less than the activation energy ( $E_a$ ) and heat of adsorption ( $Q_{ads}$ ) at 70<sup>o</sup>C – reason is because temperature can alter the rate of a chemical reaction. Further discoveries made were that the heats of adsorption (Qads) at  $40^{\circ}$ C were negative values while that of 70°C were positive values. The negative values clearly indicate that the adsorption of premature Dacryodes edulis seed extract on the surface of mild steel coupon is exothermic. Hence, physical adsorption of the inhibitor cations occurred on the mild steel surface at  $40^{\circ}$ C. Also, from the knowledge of dependence of adsorption on temperature, it is clear that compounds which obey physical adsorption have plots of isotherms at the different temperatures studied and that the reaction is a spontaneous process. The heat of adsorption  $(Q_{ads})$  at of temperature of  $40^{\circ}\!C$  were consequently, seen to be physisorption values while that of 70°C were chemisorption as was observed from the values of  $Q_{ad}$  (Ehiemere *et al.*, 2021). This is well understood as molecules are known to be trapped in precursor states (physisorption) before chemisorption. A general survey of the result at  $40^{\circ}$ C and  $70^{\circ}$ C respectively reveals, ashas been noted by many authors, that the mechanism of corrosion inhibition is due to the formation and maintenance of a protective film on the metal surface and that additives cover the anodic and cathodic sites through uniform adsorption following Langmuir Isotherm.

Further criteria to classify inhibitors as cathodic, anodic, or mixed-type inhibitor, is a function of the changes observed in polarization curves after addition of the inhibitor. The potentiodynamic polarization experiments for mild steel in the presence of 1M HCI with and without inhibitor in table 7 showed that

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### CONCLUSION

Several natural products such as plant extract, amino acids, and biopolymers have been reported to be efficient corrosion inhibitors (Raja and Sethuraman, 2008). They are the rich sources of ingredients such as fatty acids which have very high inhibition efficiency (Swathi *et al.*, 2017). Preliminary phytochemical study of crude untreated seed extracts of *Dacryodes edulis* show the presence of phenols, carboxylic acids, tannins, saponins, steroids, flavonoids, alkaloids, terpenoids and cardiac glycosides. If the crude sample is separated into acidic, basic and neutral metabolites, the acidic metabolite shows the presence of tannins, saponins, flavonoids, cardiac glycosides phenols and carboxylic acids in varying proportions. The basic metabolite shows the presence of steroids and terpenoids (Nwokonkwo, 2014). According to Rani and Basu (2011), the presence of saponin, tannin, phlobatin, anthraquinone, cardiac glycosides, flavanoid, terpene, and alkaloid has inhibition potentials. The adsorption of the inhibitor on mild steel surface is exothermic, spontaneous, and best described by Langmuir adsorption model (Obot and Obi-Egbedi, 2010).

Many theories to substantiate the mode of action of these green inhibitors have been put forth by several workers. Many have suggested that organic substances, which form onium ions in acidic solutions, are adsorbed on the cathodic sites of the metal surface and interfere with the cathodic reaction. The adsorption of these molecules could take place via interaction with the vacant d-orbitals of iron atoms (chemisorption) (Rani and Basu, 2011).

Premature *Dacryodes edulis* seed extracts acts as an inhibitor for corrosion of mild steel in 1M HCI solution and it can be concluded that inhibition efficiency increased with increase in the concentration of the inhibitor and corrosion rate increased with the decrease in the concentration of the inhibitor. Also, higher temperatures aid greater corrosion rate as seen in the gravimetric analyses carried out.

Similarly, corrosion inhibition is afforded by both physical and chemical adsorption components of premature *Dacryodes edulis* seed extract on the mild steel surface; therefore, Langmuir adsorption isotherms are obeyed. Hence, results from all the techniques employed are in reasonably good agreements and points to the fact that *Dacryodes edulis* seed extract is a good and efficient inhibitor within the range of concentrations investigated.

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