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INHIBITION OF CORROSION ON MILD STEEL USING *PIPER GUINEENSE* (UZIZA) EXTRACT IN SULPHURIC ACID MEDIUM OF VARYING PH VALUE

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ABSTRACT

Many attempts are being made to curb the menace of corrosion in the industry. Recently, green inhibitors were widely considered due to their comparative advantage over other means of corrosion control and prevention. In this research, the inhibition of mild steel corrosion in sulphuric acid solution of varying pH values by Piper guineense extract as an eco-friendly and commercially available inhibitor was studied at room temperature by weight loss technique. Weight loss and corrosion rate reduces with increase in pH value. The extract exerts its inhibitive properties by being adsorbed on the surface of the mild steel. The adsorption characteristics of the extract were found to obey physical adsorption mechanism. It was also found that the weight loss obeyed the first order rate model with rate constants (k) ranging from 0.1015-0.1238 and 0.1003-0.1302; and goodness of fit (R^2) ranging from 0.9690-0.9835 and 0.9586-0.9912 for the uninhibited and inhibited coupons respectively. This study has revealed that the extract of piper guineense, effectively inhibit mild steel corrosion in sulphuric acid (H_2SO_4) media with different pH values.

Keywords: ***Piper guineense (uziza), mild steel, weight loss, corrosion inhibition, adsorption***

1. INTRODUCTION

Excessive corrosion attack is a known re-occurrence on iron and steel surfaces deployed for use in aqueous and very aggressive environments. The corrosion process can be significantly suppressed by incorporation of certain substances into the environment in other to inhibit the corrosion reaction and reduce the corrosion rate. The inhibitors act at the interface created by corrosion products between the metal and the aqueous aggressive solution, and their interaction with the corroding metal surface, often leads to modification in either the mechanism of the electrochemical process at the double layer or in the surface available to the process. The high cost of most common corrosion inhibitors, coupled with recent concerns about the environment provide sufficient motivation for the development of new classes of inexpensive yet effective additives, specifically designed to address future environmental and safety needs.

In recent times, considerable effort has been devoted to study the corrosion inhibiting effect of some natural products, particularly biomass extracts. The reason for this is that the abundant phytochemical constituents of biomass extracts have considerable potential as inexpensive, non-toxic, readily available and renewable sources of a wide range of organic chemicals of prospective industrial significance [10].

Various scientist and researchers around the world have reported several plant extracts; ochradenusbaccatus, cassia italic [1] extracts,jathrophacurcas leaves extract [5],sienna italic extract[3], *piper guineense* seed extract [4], phyllanthusamarus extract [11], wrightiatinctoria, clerodendrumphlomidis and ipomoeatribola extracts [12], dodonaeaviscosa leave extract [7],apricot juice [2],

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pentaclethramacrophyllabentham extract [6], telfairiaoccidentalis [8],as good and promising corrosion inhibitors.

The aim of this research was to investigate the inhibitive and adsorptive characteristics of *Piper guineense*extract for the corrosion of mild steel in sulphuric acid media of varying pH values, as a cheap, non-toxic, easily biodegradable and environmentally safe substance that could be used for inhibition purposes.

2. MATERIALS AND METHODS

2.1 Extraction of Plant (*Piper Guineense*)

Samples of *Piper guineense*(uziza) were obtained, washed in running water, sun dried and ground into powder. The extraction was carried out in a Soxhlet Extractor (Figure 1) for 2hours each at a temperature of about 100^{o}C using 30g of ground stock of *Piper guineense*and 150ml of hexane. The resulting solution was allowed to cool to room temperature, filtered and stored. Thereafter, different concentrations of the test solution were prepared by dissolving 10ml of the extract in 200ml of the corrosive medium.

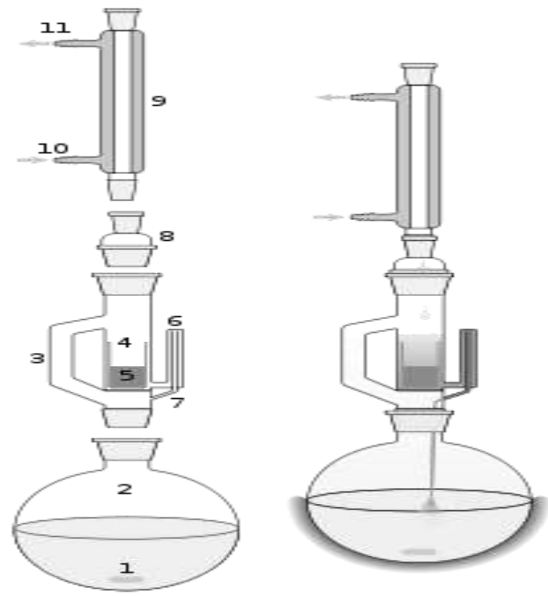

Figure 1: Schematic Representation of a Soxhlet extractor

1: Stirrer bar **2:** Still pot (the still pot should not be overfilled and the volume of solvent in the still pot should be 3 to 4 times the volume of the Soxhlet chamber) **3:** Distillation path **4:** Thimble **5:** Solid **6:** Siphon top **7:** Siphon exit **8:** Expansion adapter **9:** Condenser **10:** Cooling water in **11:** Cooling water out.

2.2 Phytochemical Analysis Of*Piper Guineense*(Uziza) Extract

This analysis was carried out to determine the phytochemicals contained in the *Piper guineense* (uziza) extract used for the inhibition process in this experiment.

2.3. Preparation of Specimen

A commercially available grade of mild steel with composition as follows: Cr-0.01, Ni-0.06, Cu-0.32, Mn-0.6, C-0.15, S-0.01, Si-0.3, and the balance Fe, identified and obtained locally was employed for this study. The sheet of mild steel was mechanically press cut (using abrasive cutter) into coupons of 3cm x 3cm x 0.1cm dimensions. A small hole of about 4mm diameter near the upper edge of the coupon was made to help hold with hooks and suspend them into the corrosive medium.

The coupons were mechanically polished successively with the help of emery papers, thoroughly cleaned with distilled water, dried and stored in moisture-free desiccators prior to use.

2.4 Preparation Of Corrosive Medium

The corrosive medium used for this experiment was sulphuric acid solution. It was prepared by appropriate dilution of analytic grade of the acid reagent with distilled water. Different sulphuric acid solution with different pH values were prepared. A pH meter was used to constantly check the pH value during the mixture. The temperature at which the solution was prepared was 27.6^{o}C.

2.5 Weight Loss Measurement

In the weight loss experiment, two sets of four (4) plastic containers of 250ml capacity were labelled (pH3 – pH6 and, with and without inhibitors) each containing 200ml of test solution at room temperature (about 27.6^{o}C). The coupons were immersed in the test solutions with the help of glass hooks and monitored after every 3days (72hours). The weights of the coupons were noted before immersion. After every immersion time of 3days, the coupons were removed, polished with brush, washed in distilled water, dried and reweighed. From the initial and final weights of the coupons, the losses of weights were calculated, and the corrosion rate (millimetre penetration per year) was computed from the following Equation (1);

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Corrosion rate, $CR = \frac{534\,W}{DAT}$ (1)

where W is the weight loss (g), D is the density of the coupon (7.85/cm^3), A is the surface area of the coupons (cm^2) and T is the immersion time (days). The efficiency of the inhibitor was also computed using Equation (2);

$$\%IE = \frac{W_o - W}{W_o} x100 \quad (2)$$

where W_o, is the corrosion rate in the absence of inhibitor, and W is the corrosion rate in the same environment with the inhibitor added.

2.6 Modelling Weight Loss, Corrosion Rate And Inhibitor Efficiency

Linear and non-linear regression techniques were employed in modelling corrosion rate and inhibitor efficiency based on the data set obtained from the experimental procedures using the Data Analysis Tool Pak provided by Microsoft Excel 2013. The applicability and reliability of the models obtained were tested on the basis of the Pearson's correlation coefficient (R) and the coefficient of determination also referred to as the goodness of fit (R^2).

2.6.1 Application of the first order rate model

Let the rate of weight loss be proportional to the weight loss at any given time,

Then: $\frac{dC_t}{dt} \alpha C_t$ (3a)

for which

$$\frac{dC_t}{dt} = kC_t \quad (3b)$$

where C_t is the weight loss at any given time, t and k is the 1st order rate constant equivalent to the rate of weight loss with time.

By rearranging Equation (3b) and integrating both sides, one obtains

$C_t = C_o e^{kt}$ (4) where C_o is the weight loss at time $t = 0$.

With Equation (4) the weight loss at any particular time *(t)* could be obtained. The strength or productive power of Equation (4) is determined by a term known as the coefficient of determination or goodness of fit (R^2). The value of R^2 lies between 0 and 1 i.e. $0 \leq R^2 \leq 1$. The closer the value of R^2 to 1 the more reliable the model in question is.

3. RESULTS AND DISCUSSION

3.1 Result of Phytochemical Analysis

The result of the phytochemical analysis done on the *Piper guineense* extract used for this experiment is shown in Table 1. [6] Reported similar experimental results to the current work.

3.2. Weight Loss Measurements

The adsorptive and inhibitive effect of *Piper guineense* extract on mild steel corrosion in sulphuric acid solution (H_2SO_4) of varying pH values was studied at room temperature (about 27.6^{o}C) using weight loss technique.

The results obtained from weight loss measurements indicated that the addition of *Piper guineense* extract into the corrosive medium (H_2SO_4) caused a significant reduction in the corrosion of mild steel coupon. The obtained variations of weight loss with time of exposure in uninhibited and inhibited coupons are represented in Figure 2 and Figure 3, showing that the presence of *Piper guineense* extract in the solution clearly reduced the weight loss of the coupons at varying exposure time as can also be seen in the work of [4]. The weight loss reduces as the pH value increases. The weight loss increases as the time of exposure increases; the reason for this behavior could be due to the loss of corrosion inhibition potency as the exposure time increases [9].

Table 1: Phytochemical Analysis of *Piper Guineense* (Uziza) Extract

S/N	SAMPLES	Protein	CHO	Lipid	Ascorbic Acid	Penicillin	Free fatty Acid	Ca^{2+} Mg/100	P^+ Mg/100	Sodium Mg/100	Fe Mg/100	K Mg/100	TANNINS Mg/100	SAPONINS Mg/100

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1.	UZIZA LEAF	16.8	48.5	20.5	22.9	$4.50X10^{-5}$	2.05	2.10	0.2	0.07	0.05	3.9	0.25	1.495
2.	UZIZA LEAF	16.9	48.6	20.6	22.8	$4.50X10^{-5}$	2.10	2.09	0.2	0.08	0.05	4.0	0.26	1.52

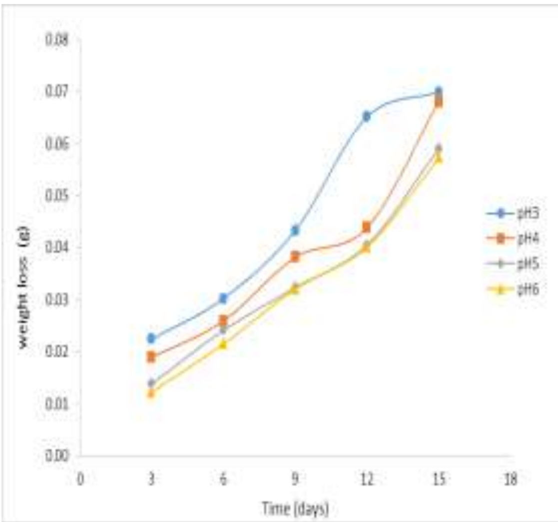

Figure 2: Variation Of Weight Loss With Time Of Exposure For Uninhibited Mild Steel Coupon In 200ml H_2SO_4 Solution

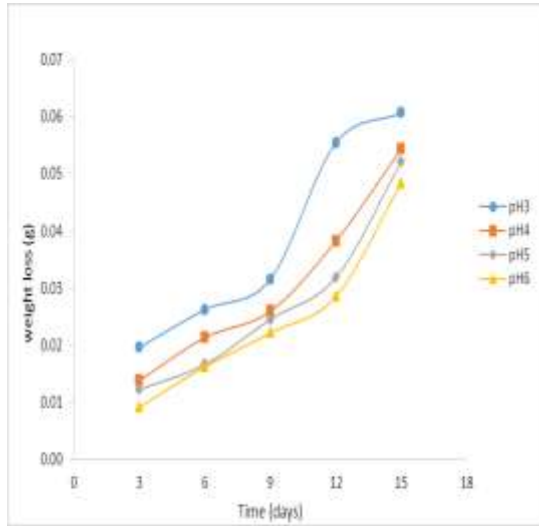

Figure 3: Variation Of Weight Loss With Time Of Exposure For Inhibited (Using Piper GuineenseExtract) Mild Steel Coupon In 200ml H_2SO_4 Solution

3.3 Corrosion Rate And Inhibition Efficiency

The Calculated values for the rate of corrosion (CR) are as shown in Table 2 and Table 3 below. It indicates that the addition of the inhibitor retards the rate of corrosion of the mild steel coupon. The corrosion rate reduces as the pH value increases. The calculated inhibitor efficiency is as shown in Table 4. It show that the inhibitor efficiencydecreases as the time of exposure increases. This could be as a result of the dissolution or loss of the inhibitor as the time of exposure increases and thus unable to properly inhibit the corrosion process.

Table 2: Corrosion Rate Of Coupons Without Inhibitor

Time (days)	Corrosion rate (mpy)			
	pH3	pH4	pH5	pH6
3	0.0265	0.0221	0.0163	0.0144
6	0.0178	0.0153	0.0142	0.0127
9	0.0170	0.0150	0.0127	0.0126
12	0.0192	0.0129	0.0120	0.0118
15	0.0165	0.0161	0.0139	0.0135

Table 3: Corrosion Rate of Coupons with Inhibitor

Time (days)	Corrosion rate (mpy)			
	pH3	pH4	pH5	pH6

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3	0.0231	0.0163	0.0114	0.0107
6	0.0155	0.0126	0.0098	0.0096
9	0.0124	0.0102	0.0096	0.0087
12	0.0164	0.0113	0.0094	0.0084
15	0.0143	0.0128	0.0123	0.0114

Table 4: Inhibition Efficiency

Time (days)	Inhibitor efficiency (%)			
	pH3	pH4	pH5	pH6
3	13	26	12	25
6	13	18	31	25
9	27	32	24	30
12	15	13	21	29
15	13	20	12	15

3.4. WeightLossSimulationForUninhibited Mild Steel Coupon

By applying the Equation (4) on the experimented data the following models were developed (Table 5).

Table 5: Weight Loss Models For Uninhibited Mild Steel Coupons

Figure 4 shows the weight of uninhibited coupons if the exposure time increases beyond the experimental time. This simulation was based on the established models in Table 5.

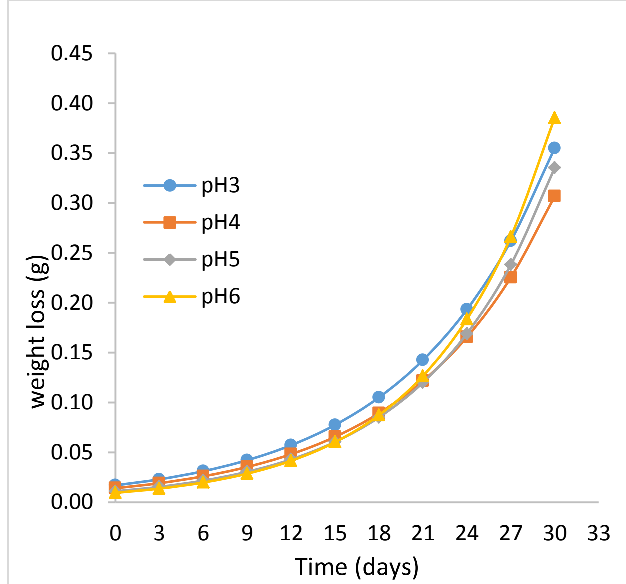

Figure 4: Weight Loss SimulationAnd Prediction Of Uninhibited Mild Steel Coupons

3.5 Weight Loss Simulation Of Inhibited Mild Steel Coupons

By applying the Equation (4) on the experimented data the following models were developed (Table 6).

Table 6: Summary of Established Models For Inhibited Coupons

pH	MODEL	C_o	K	R^2
3	$C_t = 0.0143e^{0.1003t}$	0.00143	0.1003	0.9586
4	$C_t = 0.0102e^{0.1109t}$	0.00102	0.1109	0.9907
5	$C_t = 0.00832e^{0.1185t}$	0.00832	0.1185	0.9912

pH	MODEL	C_o	K	R^2
3	$C_t = 0.0169e^{0.1015t}$	0.0169	0.1015	0.9690
4	$C_t = 0.0140e^{0.1029t}$	0.0140	0.1029	0.9835
5	$C_t = 0.0109e^{0.1142t}$	0.0109	0.1142	0.9748
6	$C_t = 0.0094e^{0.1238t}$	0.0094	0.1238	0.9749

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6	$C_t=0.00663e^{0.1302t}$	0.00663	0.1302	0.9800

Figure 5 shows the weight of inhibited coupons if the exposure time increases beyond the experimental time**.** This simulation was based on the established models in Table 2.

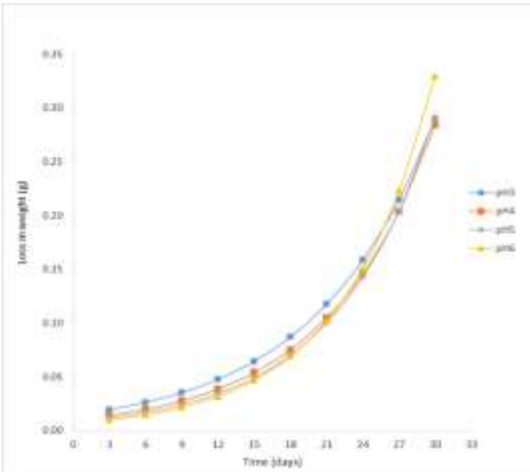

Figure 5: Weight Loss Simulation/Prediction Of Inhibited Mild Steel Coupon

4. CONCLUSIONS

The phytochemical constituents of the extract of leaves of *Piper guineense*used for this study are; protein, carbohydrates, lipids, ascorbic acid, penicillin, free fatty acid as oleic acid, calcium, potassium, sodium, iron, tannins and saponins. This study has revealed that the extract of *Piper guineense*, effectively inhibit mild steel corrosion in sulphuric acid (H_2SO_4) media with different pH values. The compound exerts its inhibitive properties by being adsorbed on the surface of the mild steel. The adsorption characteristics of the compound have been found to obey physical adsorption mechanism. Weight loss of the mild steel coupons increased with an increase in exposure time. The weight loss and corrosion rate reduces with an increase in pH value. Inhibitor efficiency decreases as the exposure time increases; this is due to the loss of corrosion inhibition potency as the exposure time increases.

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