Corrosion Inhibition of Mild Steel in a H$_2$SO$_4$ Solution
by Piper Guineense Squeezed Extract

S.O. Anuchi* and N.C. Ngobiri

Department of Pure and Industrial Chemistry, University of Port Harcourt, Port Harcourt, Nigeria

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Abstract

The inhibitive action of Piper guineense (uziza leaf) extract on the corrosion of mild steel in a 2 M H$_2$SO$_4$ medium has been studied using weight loss method. The collected leaf samples were rigorously grounded and squeezed, with the resultant gel extract used for the weight loss determination at 1.0%, 2.0%, 3.0%, 4.0% and 5.0% v/v concentrations, respectively. Therefore, rectangular mild steel coupons in a 2 M H$_2$SO$_4$ solution were also employed to determine the amount of weight loss in the absence and presence of Piper guineense extract at temperatures of 303 K, 313 K and 323 K. The results show that mild steel corrosion inhibition increases with increasing concentrations of Piper guineense extract, showing greater efficiency at higher temperatures of 313 K and 323 K. Moreover, Piper guineense extract can effectively perform as a green and non-toxic inhibitor for mild steel corrosion in acidic environments.

Keywords: Piper guineense, corrosion inhibitor, weight loss, adsorption and acidic medium.

Introduction

Corrosion is a serious economic challenge in the oil production industry. In Nigeria, millions of naira are lost each year because of corrosion [1]. However, corrosion inhibitors are widely used among other preventive or control methods, to mitigate metallic corrosion; they are substances that, when added in low dosages to a corrosive environment, prevent or decrease the corrosion rate of metals or alloys [2]. Many known inhibitors contain heteroatoms such as O, N and S, and multiple bonds in their molecular structures. Examples include nitrogen based compounds (amides, amines, imidazolines or quaternary
ammonium compounds). These chemicals are adsorbed onto the metal surface, thus forming a protective layer (film) that prevents corrosive agents from contacting with the metal [3].

Owing to the strict environmental regulations on the use of these chemicals in offshore oil and gas fields, attention has been shifted to the development of green alternatives to manage corrosion issues [4]. One of the more effective green approaches is the use of plant extracts as corrosion inhibitors. Several research works have reported the use of plant extracts on the corrosion inhibition of metals in acidic media. For example, Gunasekaran and Chauhan [5] studied the inhibition effect of 20%, 50% and 88% of an aqueous extract of Zenthoryhm alatum leaves on mild steel corrosion in H$_3$PO$_4$, using weight loss and electrochemical impedance spectroscopy techniques (EIS). It was concluded that 88% of the aqueous extract showed the highest inhibition efficiency at 70 ºC. In turn, Abiola et al. [6] investigated the inhibitive action of citrus paradise fruit juice on mild steel in a HCl solution at a temperature range of 30 ºC to 50 ºC, using weight loss technique. They observed that the inhibition efficiency decreases with increased temperatures. Patel et al. [7] studied the inhibitive action of Wrightiatinctoria, Clerodendrumphlomidis and Ipomoeatriloba plant extracts on mild steel in 0.5 M H$_2$SO$_4$ using weight loss, electrochemical impedance spectroscopy, electrochemical polarization and scanning electron microscopic methods. They observed that the inhibition efficiencies of the plant extracts increased with their higher concentrations, but decreased with higher temperatures.

Piper guineense is a West African plant species, cultivated in Mayala Island, India, Nigeria and other West African countries. The spice gotten from its dried fruits is commonly known as West African pepper or Ashanti pepper. The leaves of this plant species are known as uziza in Nigeria. The roots, fruits and leaves of this plant are used in the treatment of fever, asthma, abdominal pain and bronchitis [8]. Ebenso et al. [9] studied the inhibitive effect of ethanol extract of Piper guineense on the corrosion of mild steel in H$_2$SO$_4$, using thermometric, gasometric and gravimetric methods, but this study aims to investigate the inhibitive action of the squeezed of Piper guineense extract on the corrosion of mild steel in an H$_2$SO$_4$ medium.

Experimental

Material

The materials used for the study were mild steel sheets (composition of 0.05%-0.25% carbon and up to 0.4% manganese). The sheets were mechanically cut into different rectangular coupons, each one with the dimensions of 4 cm x 5 cm x 0.11 cm. At the edge of each mild steel coupon, a hole was made for proper immersion in an acidic solution. Therefore, proper scrubbing of all coupons with a brush was carried out, and then they were degreased by washing with deionized water, dried with acetone and stored in a desiccator before use. A 2 M H$_2$SO$_4$ solution was prepared and employed as the acidic medium.

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Plant (Piper guineense) sample preparation
Samples of Piper guineense were collected from the University of Port Harcourt Botanical garden in Port Harcourt, Rivers, Nigeria. Samples of the leaves were rigorously grounded and squeezed, with the resultant liquid gel emanating from them. The gel concentrate was properly sieved to obtain a clear semi-liquid concentrate. The filtrate was later stored in a clean glass bottle.

Methods
Weight loss determination
The method started with initial weight measurement of the pre-cleaned mild steel coupons using a weighing balance (Mettler Toledo, UK). This was followed by careful immersion of the coupons with the aid of synthetic threads into 100 mL of 2 M H$_2$SO$_4$ (aq) (blank solution). Afterwards, these coupons were further immersed into the acidic solution containing added Piper guineense extract with concentrations of 1.0%, 2.0%, 3.0%, 4.0% and 5.0% v/v, respectively; they were placed in a thermostatic water bath (P Selecta, France) maintained at 303 K, 313 K and 323 K, respectively. The weight loss was determined by retrieving the mild steel coupons at 2 hour intervals. Before measurement, each coupon was scrubbed with a light brush, rinsed with de-ionized water, cleaned and dried with acetone. The whole process was carried out in three replicates. The weight loss of mild steel coupons was evaluated in grams as the difference in their weight before and after immersion in the inhibitor/blank solution, using equation (1).

\[ \Delta W = (W_I - W_F) \]  

where \( W_I = \) initial coupon weight and \( W_F = \) final coupon weight.

From the weight loss determination, the inhibition efficiency (%I) of the extract was calculated using equation (2).

\[ \% \text{ Inhibition efficiency} = \frac{[\Delta W_{\text{BLANK}} - \Delta W_{\text{ADDITIVE}} \times 100\%]}{\Delta W_{\text{BLANK}}} \]  

where \( \Delta W_{\text{BLANK}} \) is the change in the mild steel weight in the inhibitor absence, and \( \Delta W_{\text{ADDITIVE}} \) is the change in the mild steel weight in the inhibitor presence.

Results and discussion
Effect of Piper guineense extract on the corrosion of mild steel
The weight loss variations with time for mild steel, in an acidic medium at 303 K, 313 K and 323 K, are shown in Fig. 1. From this figure, the weight loss is the highest at 323 K, and the lowest at 303 K. Therefore, the corrosion rate, which produces the weight loss of the mild steel coupons in the acidic medium, increases with higher temperatures. Weight loss values of mild steel in the acidic solution at 313 K and 323 K are far greater than those at 303 K under the same conditions. This implies that mild steel corrosion rate is greater at higher temperatures.
temperatures, which creates the need for very high temperatures conditions, so that plant extracts effectively inhibit corrosion.

**Figure 1.** Variations of weight loss (\(\Delta W\)) with time for mild steel coupons in a 2 M \(\text{H}_2\text{SO}_4\) solution at three different temperatures, in the inhibitor absence (no additives).

The corrosion rate of the mild steel coupons in the acidic medium was observed in the plant extract presence, at 303 K, 313 K, and 323 K, as shown in Fig. 2, Fig. 3 and Fig. 4, respectively.

**Figure 2.** Weight loss variations with time for mild steel coupons in a 2 M \(\text{H}_2\text{SO}_4\) solution containing different concentrations of Piper guineense extract, at 303 K.

From Fig. 2, it was clearly observed that, at 303 K, the weight loss of mild steel coupons considerably decreased with increasing concentrations of Piper guineense extract over time. Based on this, it can be inferred that Piper guineense extract can effectively perform as a corrosion inhibitor for mild steel in an acidic environment. This corresponds to the observation made by Ebenso et al. [9], using ethanol extract of Piper guineense.

Fig. 3 shows that, at 313 K, the increase in the concentration of Piper guineense extract from 1.0% to 5.0% v/v resulted in decreasing weight losses of mild steel coupons over the given time. In addition, mild steel corrosion rate in the presence of Piper guineense extract was lower than that obtained for the blank. This
indicates that the various used concentrations of Piper guineense extract inhibit mild steel corrosion in a H$_2$SO$_4$ solution.

**Figure 3.** Variations of weight loss with time for mild steel coupons in a 2 M H$_2$SO$_4$ solution containing different concentrations of Piper guineense extract, at 313 K.

**Figure 4.** Weight loss variations with time for mild steel coupons in a 2 M H$_2$SO$_4$ solution containing different concentrations of Piper guineense extract, at 323 K.

In Fig. 4, the weight loss of mild steel coupons in an acidic solution was found to decrease with a higher concentration of Piper guineense extract from 1.0 mL to 5.0 mL, at 323 K, indicating that it can inhibit mild steel corrosion at higher temperatures. This trend corresponds to the study of damsissa (Ambrosia Maritime L) plant extract on the corrosion inhibition of mild steel in a H$_2$SO$_4$ medium at 313 K, by Abdel-Gabelr et al. [10]. The inhibition efficiencies (%EI) variations for mild steel coupons in a 2 M H$_2$SO$_4$ solution containing different concentrations of Piper guineense extract at different temperatures are presented in Fig. 5.

It can be seen from Fig. 5 that an increase in the concentration of Piper guineense extract from 1.0% to 5.0% v/v resulted in a corresponding higher inhibition efficiency. In addition, as the temperature of the acidic/corrosive medium increased from 303 K to 323 K, the extract inhibition efficiency also increased; this indicates that the IE is enhanced with higher temperatures, irrespectively of
the extract concentration in the acidic medium. This trend contrasts with the observations made by Ebenso et al. [9] on the use of ethanol extract of Piper guineense; they found out that its inhibition efficiency values decreased with an increase in temperature from 303 K to 333 K. Consequently, they concluded that the extract inhibition mechanism is based on physical adsorption, rather than chemical adsorption, and that its inhibition efficiency values increase with higher temperatures [11, 12 and 13].

**Figure 5.** Inhibition efficiency variations for mild steel coupons in a 2 M H$_2$SO$_4$ solution containing different concentrations of Piper guineense extract at different temperatures.

**Conclusion**
From this study, we safely concluded that 1.0%, 2.0%, 3.0%, 4.0% and 5.0% v/v of Piper guineense extract inhibit mild steel corrosion in a 2 M H$_2$SO$_4$ acidic solution at 303 K, 313 K and 323 K, respectively. This plant extract can effectively inhibit corrosion at increased concentrations and temperatures. In addition, the samples preparation method may influence inhibition efficiency values, with respect to temperature and concentration. Finally, Piper guineense extract is a good green corrosion inhibitor for mild steel in an acidic environment.

**References**