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Corrosion Inhibition Study of *Raphia palm* Extract on Mild Steel in Phosphoric Acid Solution

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Abstract

The anti-corrosive study of *Raphia palm* extract on mild steel in phosphoric acid solution was conducted at room temperature, under static conditions using Weight Loss, Electrochemical, and Scanning Electron Microscopy (SEM) techniques. *Raphia palm* extract was found to inhibit the corrosion of mild steel at all concentrations used for the study ie, 0.1 v/v % - 2.0 v/v %. Results obtained from Weight Loss measurements showed a maximum inhibition efficiency of 84.47 % which was attained at 2.0 v/v % concentration of *Raphia palm* extract in 0.5 M H_3PO_4. The inhibitory action against the mild steel is attributed to adsorption of phytochemical components of the *Raphia palm* extract on the metal surface which limits the dissolution of the metal surface by blocking the active corrosion sites, thereby, reducing the rate of corrosion. Electrochemical studies showed that the inhibitor is of mixed type and the anodic reaction controls the rate of corrosion. Scanning electron microscopy (SEM) studies confirm the formation of a protective layer of *Raphia palm* extract on the metal surface.

Keywords: Corrosion Inhibition; *Raphia palm* Extract; Mild Steel; Phosphoric Acid

Introduction

Metallic corrosion is the surface wastage that occurs when metals are exposed to corrosive environments. The protection of metals against corrosion by phosphoric acid, according to [1] has been the subject of much study since it is used in many industrial processes. Acid solutions are generally used for the removal of undesirable scales, rust, and other materials in several industrial processes and therefore require to be restrained against corrosive attack on equipment.

According to Satapathy et al. [2], the use of inhibitors is one of the most practical methods for preventing unexpected metal dissolution and consumption in acidic media. Corrosion inhibitors are substances which when added in small concentrations to corrosive media decrease or prevent the reaction of metal with the media [3].

Different organic and inorganic compounds have been investigated as inhibitors to control or prevent metal from corrosive attacks [2]. However, most of the synthetic organic compounds and chemicals used as inhibitors have been found to have hazardous effects on both humans and the environment [3], hence the motivation to develop non-toxic and environmentally friendly natural products as corrosion inhibitors. The use of these natural products, such as plant extracts for inhibition of metallic corrosion in phosphoric acid has been reported [1,4,5,6,7]. Plant extracts have become important because they are environmentally acceptable, inexpensive, readily available and renewable sources of materials, and ecologically acceptable. Moreover, they can be extracted by simple procedures with low cost [8]. Plant products are viewed as a sustainable alternative to the synthetic products.

Raphia palm (Raphia farinifera) scientifically classified into the family Arecaceae, is a tropical palm tree occurring in lowland riverain, swamp forests, around human habitations and other moist locations at altitudes of 50-1000m. In Nigeria, it is represented by about five species commonly found in the Middle Belt part of Nigeria. The trunk of this species is up to 10m tall, 1m in diameter, and is sheathed in persistent leaf bases. Raphia farinifera is monocarpic in nature; flowering and fruiting only once, followed by death, although the plant itself keeps living due to the development of new suckers. The plant develops flowers when the tree is in the range 20-25 years old, and it takes a further 5-6 years from flowering to ripe fruit, all fruits ripening together. The fruits are oblong to ovoid, 5-10cm in length, with imbricate, glossy, golden-brown scales. The leaves are used for thatching, hut construction, furniture, fences, sweeping-brushes, and several other commercial and domestic uses [9]. *Raphia palm* extracts have been reported [10-17,] to have pharmacological properties. On a negative side, Ohimain et al. [18], reported the use of *Raphia palm* extract as a bait to stupefy and catch fish.

Literature has shown that the inhibitive effect of some plant solutions is due to the adsorption of molecules of phytochemicals present in the plant on the surface of the metal [19,20]. Phytochemical screenings carried out by Ikuve et al. [21], on *Raphia palm* fruits revealed the presence of saponins, cardiac glycosides, alkaloids, flavonoids and phenobutinones in the peels and have been reported to have efficient corrosion inhibition properties.

Mild steel is a very versatile ferrous alloy metal that is used in a wide range of industrial food applications because of its combined excellent mechanical properties, ease of fabrication, excellent welding ability, and low purchasing cost; six times lesser than stainless steel [22]. However, mild steel is susceptible to corrosion in acidic solutions leading to substantial loss of metals. This article investigates the corrosion inhibition properties of *Raphia palm* extract on mild steel in a phosphoric acid solution.

Experimentation

Materials

The materials that were used for this work included *Raphia palm* fruits, mild steel strip, and a solution of phosphoric acid.

Preparation of Plant Extract

Ripe fruits of *Raphia palm* which were harvested from the wild were unshelled, exposing the nuts from which the peels were removed and shade-dried for 5 days to enhance preservation. The dried peels were then ground into powder of particle size ≤ 150μm to facilitate higher extract yield due to higher surface-to-volume ratio using a Soxhlet Extractor. To obtain the extract solution, 200 g of the *Raphia palm* powder was weighed and reflux-extracted continuously with absolute methanol in a Soxhlet Extractor for 24 hrs. At the end of the extraction process, the extract solution was concentrated by drying in an oven at 75 ℃ for 1 hr. The stock solution was then prepared by diluting the *Raphia palm* extract with n-heptane in the ratio 3:1 (i.e. n-heptane: *Raphia palm* extract).

Preparation of Specimens

The test specimens used for corrosion measurements were prepared in accordance with [23]. The material that was used for preparing specimens for corrosion measurements was mild steel, having the chemical composition as shown in Table 1. In this procedure, coupons were made from a cold rolled mild steel bar that was free of rust spots. The steel bar was sheared to rectangular-shaped coupons of length 10 mm, width 10 mm, and thickness 5 mm. All sharp edges on each coupon specimen were deburred using a file. Prior to all measurements, the steel specimens were mechanically polished with emery papers of grades: 220, 400, 800 and 1000; thoroughly washed with distilled water; degreased with acetone; dried at room temperature; and weighed (w_1) to the nearest 0.1 mg on an analytical balance. After weighing, the specimens were kept in a desiccator until ready for use. Preparation of the specimens was carried out at the tools machine shop of the Ahmadu Bello University, Zaria.

Element% comp.	**C** 0.193	**Si** 0.267	**Mn** 0.75	**P** 0.046	**S** 0.032	**Cr** 0.168	**Ni** 0.119
Element% comp.	**Mo** 0.012	**Al** 0.0044	**Cu** 0.223	**Co** 0.011	**Ti** <0.0010	**Nb** <0.0030	**V** 0.0019
Element% comp.	**W** 0.022	**Pb** >0.028	**B** 0.0015	**Sn** 0.024	**Zn** >0.032	**As** 0.0087	**Bi** <0.0020
Element% comp.	**Ca** 0.0013	**Ce** <0.0030	**Zr** 0.0016	**La** <0.0010	**Fe** 98.0		

Table 1: Chemical Composition of Mild Steel

Preparation of Electrolyte

The electrolyte used for experiments was prepared in accordance with [24]. In this procedure, a solution of 0.5 M H3PO4 was prepared by diluting analytical grade of 85.5 % phosphoric acid of 14.8 Molar concentration and specific gravity 1.70 with distilled water. To make 1 litre of 0.5 M H_3PO_4 34 ml of the phosphoric acid reagent was added to 500 ml of distilled water in a 1000-ml volumetric flask while stirring it slowly. The solution was then topped with more distilled water to make up a litre. Preparation of the electrolyte was carried out in the Chemistry Laboratory of the Ahmadu Bello University Zaria.

Methods

Raphia palm extract has been reported [21] to have good inhibitory effect on mild steel corrosion in citric acid solution. The inhibitory action is attributed to the adsorption of the inhibitor molecules at the metal/solution interface, forming a protective film. The experimental methods employed in this study included Weight Loss, Electrochemical and Scanning Electron Microscopy (SEM) techniques.

Weight Loss Measurements

The weight loss measurements were conducted in accordance with [25]. In this procedure, carefully prepared and weighed (w_1) mild steel specimens were suspended and completely immersed in glass beakers containing 200 ml of 0.5 M H3PO4 solution with various concentrations of *Raphia palm* extract of 0.0 v/v % (without extract), 0.1 v/v %, 0.5 v/v %, 1.0 v/v %, 1.5 v/v % and 2.0 v/v

%. The corrosion measurements were carried out at room temperature under static conditions for specimen immersion period of two, four, six, eight and ten days. At the end of each run, the samples were withdrawn from the test solution, rinsed with distilled water, cleaned with acetone, dried and weighed (w_2) again. The weight loss was determined by the difference between the initial and the final weights of each test specimen. To study the weight loss measurements, the inhibition efficiency (η_w)was calculated using the following equation [26]:

$$\eta_w\,(\%) = \left(\frac{W_2 - W_1}{W_2}\right) \times 100$$

Where, W_1 and W_2 are weight loss of the uninhibited and inhibited mild steel specimens respectively.

Electrochemical Measurements

Potentiodynamic polarization measurements were conducted in accordance with [27]. The measurements were carried out using a conventional three-electrode cylindrical glass cell containing 200ml of 0.5 M H3PO4 solution and various concentrations of *Raphia palm* extract of 0.0 v/v % (without extract), 0.1 v/v %, 0.5 v/v %, 1.0 v/v %, 1.5 v/v % and 2.0 v/v % at room temperature, under stationary conditions. Mild steel specimens were used as working electrodes (WE), while platinum foils were used as counter electrodes (CE), and saturated calomel electrodes as reference electrodes (RE). Prior to each experiment, each working electrode was allowed to corrode freely and its open circuit potential (OCP) was recorded as a function of time up to 15 minutes as described elsewhere [2]. Polarization studies were conducted using Autolab analyzer (PG STAT 204N) at a scan rate of 1mV s–1 in the potential range of ± 250mV. All potentials were recorded with reference to the saturated calomel electrode (SCE). Linear polarization Resistance measurements were carried out in the potential range of ± 10 mV at a scan rate of 1 mVs^{-1}. CH-instrument beta software was used for evaluating the experimental data.

To study the polarization curves, the inhibition efficiency was calculated using the following equation [28]:

$$\eta_p\,(\%) = \left(\frac{I_2 - I_1}{I_2}\right) \times 100,$$

Where, I_1 and I_2 are the corrosion current densities of the uninhibited and inhibited mild steel specimens respectively.

The inhibition efficiency obtained from linear polarization resistance studies was calculated using the relationship [29]:

$$\eta_R\,(\%) = \left(\frac{R_2 - R_1}{R_2}\right) \times 100$$

Where, R_1 and R_2 are polarization resistance of the uninhibited and inhibited mild steel specimens respectively.

Scanning Electron Microscopy (SEM)

Surface analysis was carried out using scanning electron microscope model PHENOM Prox. In this procedure, mild steel bar shared into dimensions of 5 mm × 5 mm × 5 mm were used. The mild steel specimens were initially ground to a 600-mesh finish on grinding wheels, using water for lubrication. After grinding, the specimens were polished with abrasives having particle sizes starting with 3 µm for coarse polishing and proceeding to 1 µm. The specimens were then washed with distilled water, degreased with acetone and left to dry under room temperature. After drying the specimens were attached on plastic hooks with nylon strings and immersed in 2 glass beakers containing 100 ml of 0.5 M H_3PO_4 having 0.0 v/v % and 1.5 v/v % *Raphia palm* extract concentration respectively. The specimens were removed from the test solutions after 2 days, mounted on a stub of metal with adhesive and coated with 60 nm of gold and loaded on the scanning electron microscope. Morphological examination was conducted at 5 KV.

Results and Discussions

Weight Loss Measurements

The effect of *Raphia palm* extract concentration and immersion period on weight loss for mild steel corrosion in 0.5 M H_3PO_4 is shown in Figure 1. Similarly, the effect of extract concentration and immersion period on inhibition efficiency for mild steel corrosion in 0.5 M H_3PO_4 is presented in Figure 2. It can be seen from the graph of Figure 1 that in the presence of the extract, the weight loss decreased with increase in immersion period. On the other hand, there was an increase in weight loss with corresponding increase in immersion period in the absence of the extract. It was also observed that *Raphia palm* extract inhibited the corrosion of mild steel in phosphoric acid solution at all concentrations used in the study, except the blank solution (i.e., 0.0 v/v %). After specimen immersion period of 10 days, maximum inhibition efficiency of 84.47 % was attained at 2.0 v/v % concentration of the inhibitor in 0.5 M H_3PO_4 as shown in Figure 2. The inhibitory action against the mild steel corrosion can be attributed to adsorption of the phytochemical components of the plant extract on the metal surface. This adsorption limits the dissolution of the metal by blocking the active corrosion sites on the metal surface, thereby, decreasing the weight loss of the metal with a corresponding increase in inhibition efficiency as the concentration increases. Similar sorts of findings have been reported elsewhere [30,19].

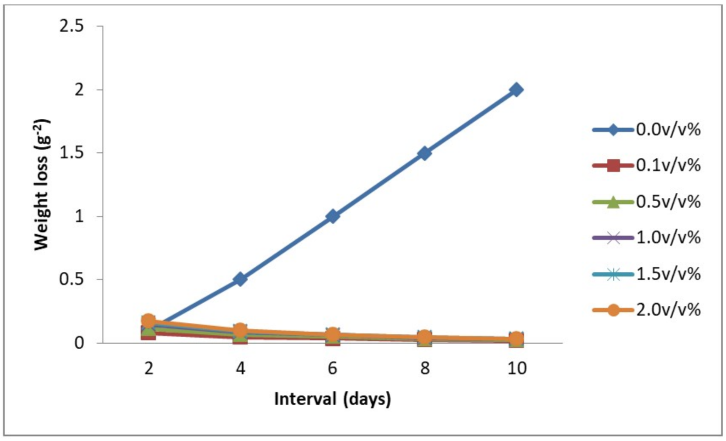

Figure 1: Effect of extract concentration and immersion period on weight loss

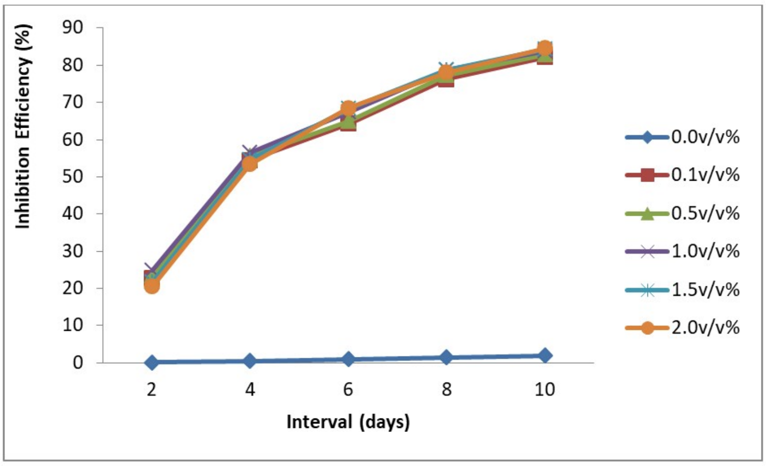

Figure 2: Effect of extract concentration and immersion period on inhibition efficiency

Potentiodynamic Polarization Measurements

Potentiodynamic anodic and cathodic polarization plots for mild steel specimens in 0.5 M H_3PO_4 containing various concentrations of *Raphia palm* extract are shown in Figure 3. The respective kinetic parameters including corrosion current density (I_{corr}) corrosion potential (E_{corr}), anodic Tefel slopes (β_a), cathodic Tefel slopes (β_c) polarization resistance (R_p), and corrosion rate are listed in Table 2. An analysis of the polarization curves (Figure 3) indicates that upon addition of *Raphia palm* extract, both the anodic and cathodic curves shift to lower current densities indicating a decrease in corrosion rate. The remarkable change in values of both the anodic and cathodic Tafel constants, (β_a), and (β_c), respectively shows that the extract acts as a mixed-type inhibitor. The increase in concentration of the extract also leads to a corresponding decrease in corrosion current densities [31]. Also, as shown in the data of Table 2, the increase in concentration of the extract resulted to a decrease in (I_{corr}), β_a, β_c, and CR values with corresponding increase in Rp and the inhibition efficiency. In addition, it can be observed that the values of β_a tend to a more positive direction than the β_c values. This indicates that the anodic reaction controls the rate of corrosion. On a general note, the addition of *Raphia palm* extract decreases the corrosion rate, and the inhibition efficiency increases with increase in concentration of the extract.

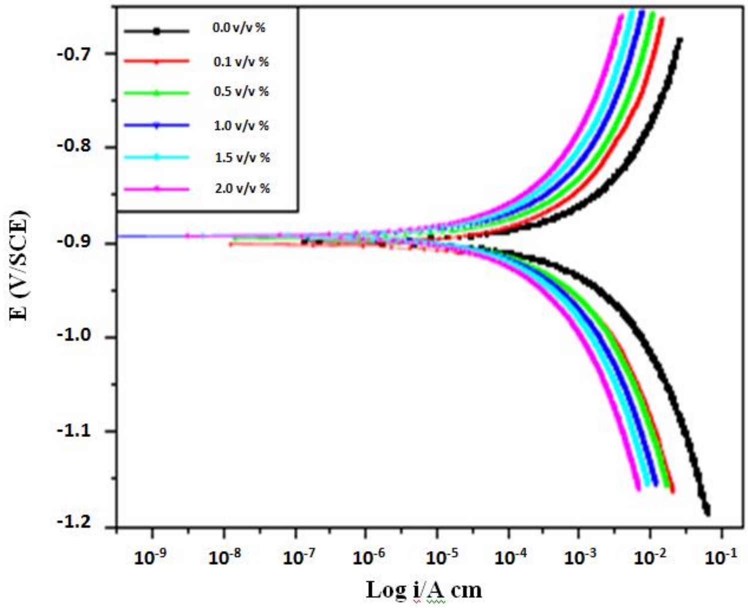

Figure 3: Polarization Curves for Mild Steel in 0.5 M H_3PO_4 Containing Various Concentrations of *Raphia palm* Extract

RPE Conc. (v/v %)	E_{corr} (V)	I_{corr} (µA/cm2)	β_a (mV/dec)	β_c (mV/dec)	CR (mm/yr)	R_p (Ω)
0.0	– 0.9168	0.00246	0.54513	0.26438	29.59	31.43
0.1	– 0.9110	0.00190	0.49618	0.20816	22.13	41.03
0.5	– 0.8885	0.00113	0.41582	0.17320	13.15	68.65
1.0	– 0.8573	0.00088	0.36746	0.16850	10.28	87.59
2.0	– 0.8503	0.00057	0.36746	0.11664	7.01	132.31
2.0	– 0.8425	0.00046	0.32467	0.10531	5.29	159.27

Table 2: Kinetic Parameters Derived from Polarization Curves of Mild Steel Corrosion in Aqueous solution of 0.5 M H_3PO_4 Containing Various Concentrations of *Raphia palm* extract

Surface Morphology

The scanning electron microscopy (SEM) micrographs of the surfaces of mild steel specimens obtained at 1500X magnification after immersion period of 2 days in 0.5 M H_3PO_4 at 0.0 v/v % and 1.5 v/v % of inhibitor concentration are shown in Figure 4. Figure 4a shows a freshly polished surface while Figures 4b and 4c show corroded and inhibited metal surfaces respectively. An inspection of the inhibited specimen (Figure 4c) shows that the roughness is remarkably reduced, showing fewer pits and cracks. This shows that *Raphia palm* extract exhibits good inhibitive properties for the corrosion of mild steel in solutions of phosphoric acids.

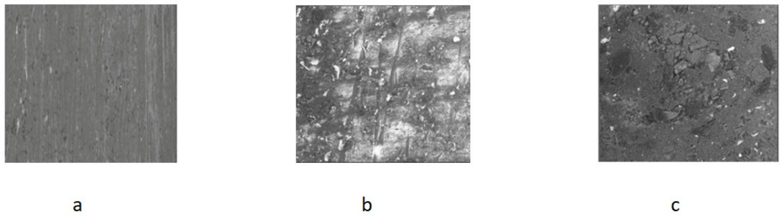

Figure 4: SEM Micrographs obtained at 1500x Magnification of (a) polished mild steel; (b) mild steel immersed in 0.5 M H_3PO_4 and (c) mild steel immersed in 0.5 M H_3PO_4 Containing 1.5 % *Raphia palm* extract

Mechanism of Corrosion Inhibition

In the absence of corrosion resistance, the solution is in contact with the metal surface and the porous surface film. In the presence of corrosion resistance, the open locations in the porous layer are blocked due to adsorption of the inhibitor molecules at the metal/solution interface, forming a protective film as described elsewhere [32].

Limitations and Constraints

According to Kigigha et al. [12], *Raphia palm*s are among the most diverse and geographically wide-spread palms. Also, studies conducted by Youkparigha et al. [33], indicate that there are variations in the phytochemical contents of various species of *Raphia palm* extracts. This implies that the inhibitory properties of extracts from the various species may also vary. In addition, as with most organic materials, the life span of *Raphia palm* extract cannot be sustained, hence its inhibitory property over time may not be guaranteed. Furthermore, this study is limited to determining the inhibitory properties of the overall phytochemical contents of *Raphia palm* fruit extracts for the corrosion of mild steel in 0.5 M H3PO4. To identify the chemical compound in *Raphia palm* extract that provides more protection for mild steel corrosion in acidic media, it is essential to separately evaluate and compare the inhibitive characteristics of all the chemical compounds present in the extract.

Conclusion

The article concludes a comprehensive evaluation of corrosion inhibition properties of *Raphia palm* fruit extract on mild steel in 0.5 M H_3PO_4. Weight Loss measurements showed that *Raphia palm* extract inhibits the corrosion of mild steel in phosphoric acid solution at all concentrations used in the study, i.e., 0.1 – 2.0 v/v %. Maximum inhibition efficiency of 84.47 % was attained at 2.0 v/v % concentration of extract in 0.5 M H3PO4. The inhibitory action against the mild steel corrosion can be attributed to adsorption of the phytochemical components of the plant extract on the metal surface. This adsorption limits the dissolution of the metal by blocking the active corrosion sites on the metal surface, thereby, decreasing the weight loss of the metal with a corresponding increase in inhibition efficiency as the concentration increases.

Electrochemical results showed the mixed-mode of the inhibitor. In addition, the studies indicate that the anodic reaction controls the rate of corrosion.

The Scanning Electron Microscopy (SEM) studies confirm the formation of a protective layer of *Raphia palm* extract on the surface of the metal.

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