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Ikuve T
Department of Mechanical Engineering, FIDEI Polytechnic Gboko, Nigeria

Amine JD
Department of Mechanical Engineering, Joseph Sarwuan Tarka University, Makurdi, Nigeria

Ashwe A
Department of Mechanical Engineering, Joseph Sarwuan Tarka University, Makurdi, Nigeria

Corresponding Author:
Ikuve T
Department of Mechanical Engineering, FIDEI Polytechnic Gboko, Nigeria

Corrosion inhibition effect of *Raphia palm* extract on mild steel in citric acid

Ikuve T, Amine JD and Ashwe A

Abstract
The inhibitive effect of Raphia palm extract on the corrosion of mild steel in citric acid was investigated 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%. The maximum inhibition efficiency of 94.74% was attained at 2.0 v/v% concentration in 0.5 M $C_6H_8O_7$. Results obtained from Weight Loss Measurements showed that the inhibition efficiency varies with the inhibitor concentration as well as the immersion period; at higher concentrations and an increase in the immersion period, the inhibition efficiency remains almost constant, whereas at lower concentrations and an increase in the immersion period the inhibition efficiency decreases. The decrease in inhibition efficiency with an increase in the immersion period is attributed to increased desorption and decreased adsorption of the inhibitor on the metal surface. Electrochemical studies showed that the inhibitor is of mixed type with a slight predominance of cathodic character for citric acid. Scanning Electron Microscopy (SEM) studies confirm the formation of a protective layer on the metal surface.

Keywords: corrosion inhibition, mild steel, *Raphia palm* extract, citric acid

1. Introduction
Corrosion is partial or complete wearing away, dissolving, or softening of any substance by chemical or electrochemical reaction with its environment [1]. The study of mild steel corrosion in acidic media has gained importance particularly in acidic media. According to [2], acid solutions enhance the rate of metal dissolution and in most cases are indirectly responsible for failure of metallic materials. Citric acid is a mild acid which when exposed to mild steel can cause stress corrosion cracking of the steel [3]. Acid solutions are used for the removal of scales, rust, and other materials in many industrial processes and therefore require to be restrained against corrosive attack on equipment.

The use of corrosion inhibitors, according to [4] is one of the most effective methods of protecting metal surfaces against corrosion in an acidic environment. Corrosion inhibitors are substances, which when added in small concentrations to corrosive media decrease or prevent the reaction of metal with the media [5]. Inhibitors are useful additives to cooling systems, refinery production units, boilers, etc. Different compounds have been investigated as inhibitors in bid to minimise or prevent metal corrosive attacks. 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 [5]. This justifies the search for cheaper natural inhibitors. The use of plant extracts for inhibition of mild steel corrosion in acidic media has been widely reported, [6, 7, 5].

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 [8].

Raphia palm fruit peels and pulp are edible, and contain tannins**,** and phytochemical screenings carried out by [9]**,** revealed the presence of alkaloids, saponins, flavonoids and phenols in the pulp and the seeds. There are many research reports on naturally occurring products as corrosion inhibitors on metals in aggressive solutions [10]**.** This study focused on evaluating the corrosion inhibition properties of *Raphia palm* fruit extract on mild steel in citric acid solution.

2. Experimentation

2.1 Materials

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

2.1.1 Preparation of plant extract and other materials

Ripe fruits of *Raphia palm* were harvested, 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 a 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 process, the obtained 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). The plant extract was screened for alkaloids, tannins, saponins, flavonoids, polyphenols, cardiac glycosides, anthraquinins, and phenobutinones, using Qualitative Phytochemical analysis in accordance with **[11]** and **[12]**. Gas Chromatography – Mass Spectrometry (GC-MS) was carried out using Shimadzu GCMS-QP 2010 Plus Model, Japan equipped with flame ionization detection (FID) and a CBP-5 capillary fused silica column (2.5 m × 0.25 mm i. d., 0.22 µm film thickness) **[13]**. Initially the instrument was programmed from 200 ^{0}C to 250 ^{0}C at 5 ^{0}C/min and held for 2.50 min and then programmed to 300 ^{0}C at 15 ^{0}C/min and held for 6.5 min for a total analysis period of 24 min, with the transfer line maintained at 280 ^{0}C**.** After programming, 5 µl of *Raphia palm* extract was injected into the system for analysis. The injection port temperature was held at 250 ^{0}C and operated in the split mode. The oven temperature was held at 70 ^{0}C then programmed at 10 ^{0}C /min and held for 5 min to 280 ^{0}C [14]. Other operating conditions were as follows: carrier gas, He (99.98%); pressure, 116.9 Kpa; total flow, 40.8 ml/min; column flow, 1.80 ml/min; purge flow, 3.0 ml/min; linear velocity, 49.2 cm/sec; split ratio, 1:20 [15, 14]**.**

To determine the functional groups in the plant extract, Fourier Transform Infrared Spectrometry was carried out using Shimadzu FTIR-8400S Fourier Transform Infrared Spectrometer. The sampling station used was equipped with a trough plate crystal. Before experimentation, the trough plate crystal and all tools were carefully cleaned with ethanol. After cleaning, a few drops of *Raphia palm* extract sample liquid were poured into the trough plate crystal with a plastic pipette, ensuring that the sample completely covered the exposed surface of the crystal. To analyse the sample, an infrared radiation comprising a range of frequencies was directed at the sample and single-beam spectra of the sample were collected. The single-beam spectra were then scanned between 4000 cm^{-1} and 500 cm^{-1} at a resolution of 2.0 and 10 numbers of scans. A detector was then used to read the intensity of the transmitted radiation at all frequencies scanned and the transmitted values were calculated and recorded using Happ – Genzel software. Data analysis was carried out by assigning the absorption frequency bands in the sample spectrum to appropriate normal modes or vibrations in the molecules [16]**.**

2.1.2 Preparation of specimens

The test specimens used for corrosion measurements were prepared in accordance with the American Society for Testing and Materials [17]**.** 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**.**

2.1.3 Preparation of electrolyte

The electrolyte used for experiments was prepared in accordance with **[18]**. In this procedure, a solution of 0.5 M $C_6H_8O_7$ was prepared by diluting analytical grade of citric acid with distilled water. To make 1 litre of 0.5 M $C_6H_8O_7$, 96 g of the citric acid reagent was added to 500 ml of distilled water in a 1000-mL volumetric flask while slowly stirring it **[14]**. Once the solution was at room temperature, it was diluted with more distilled water to exactly 1 litre mark on the flask. Preparation of the electrolyte was carried out in the Chemistry Laboratory of the Ahmadu Bello University, Zaria.

Table 1: Chemical Composition of Mild Steel

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

2. Methods

Corrosion measurements were carried out using Weight Loss and Potentio-dynamic Polarization techniques.

2.2.1 Weight loss measurements

The weight loss measurements were conducted in accordance with [19]. Mild steel specimens, prepared and weighed (w_1) were suspended and completely immersed in glass beakers containing 200 ml of 0.5 M $C_6H_8O_7$ 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 in triplicate at room temperature under static conditions for specimen immersion period of two, four, six, eight and ten days [20]. 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 equation 1 [21].

$$\eta_w(\%)=\left(\frac{W_2 - W_1}{W_2}\right)\times 100 \quad \ldots\ldots (1)$$

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

2.2.2 Electrochemical measurements

Potentiodynamic polarization measurements were conducted in accordance with [22]. The measurements were carried out using a conventional three-electrode cylindrical glass cell containing 200ml of 0.5 M $C_6H_8O_7$ 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). Each working electrode (5 mm × 5mm × 5mm) was mounted on a specimen holder using epoxy resin. The resin mixture was prepared by blending araldite with a hardener in the ratio of 10:1 and left for a period of 12 hours to properly set. After hardening, the specimen surfaces were mechanically polished with successive grades of silicon carbide paper starting from 200-grit up to 1000-grit on rotating grinding wheels, using water as lubricant. After polishing, the specimens were washed with distilled water, degreased with acetone, allowed to dry at room temperature and kept in a desiccator until required for use. 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 [23]. Polarization studies were conducted using an Autolab analyzer (PG STAT 204N) at a scan rate of 1mV s^{-1} in the potential range of 250mV below the corrosion potential to 250mV above the corrosion potential. 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 to 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 2 [24].

$$\eta_p(\%)=\left(\frac{I_2 - I_1}{I_2}\right)\times 100, \quad \ldots\ldots (2)$$

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 [25]:

$$\eta_R(\%)=\left(\frac{R_2 - R_1}{R_2}\right)\times 100 \quad \ldots\ldots (3)$$

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

2.2.3 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, specimens were polished with abrasives having particle sizes starting with 3 µm for coarse polishing and proceeding to 1 µm. The specimens were washed with distilled water, degreased with acetone and left to dry at 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 $C_6H_8O_7$ having 0.0 v/v% and 1.5 v/v% *Raphia palm* extract concentration respectively. The specimens were removed 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.

3. Results and Discussions

3.1 Characterization of Plant Extract

Phytochemical screenings of *Raphia palm* extracts shown in Table 2 reveals the presence of saponnins, cardiac glycosides, alkaloids, flavonoids, and phenobutinones. These compounds are heterogeneous organic compounds containing nitrogen, oxygen, sulphur and/or aromatic rings in their molecular structures [26]. Organic heterogeneous compounds containing these elements have been reported to be efficient corrosion inhibitors for metals [27]. Thus, it is assumed that the adsorption of these compounds on the surface of the mild steel used in this study is, responsible for the inhibition of the corrosion reaction. The good performance of the inhibitor may also be attributed to the synergy between the different compounds present in the extract.

Similarly, the Gas Chromatography and Mass Spectrometry (GC-MS) spectra of *Raphia palm* extract presented in Figure. 1 indicates that, *Raphia palm* extract comprises esters, stearic, palmitic, oleic, linoleic, myristic acids, etc. According to [28, 29, 30], fatty acids such as palmitic, stearic, oleic, linoleic, and myristic acids have good inhibitive properties for the corrosion of metals while the Fourier Transform Infrared Spectrometry (FT-IR) spectrum of *Raphia palm* extracts shown in Figure 2. indicates the presence of several compounds belonging to such functional

groups as hydroxyl (N-H), aliphatic and aromatic (C-H), alkyl (C=H), etc. groups. According to [31], compounds belonging to these functional groups contain alkaloids, flavonoids and oils which have efficient corrosion inhibition properties.

Table 2: Phytochemical Screenings of *Raphia palm* Extract

Chemical constituent Screening	
Saponins + +	
Cardiac glycosides	+ +
Alkaloids	+
Flavonoids	+
Phenobutinones	+
Tannins	–
Polyphenols	–
Anthraquinones	–

Notes: + +, present in greater quantity; +, present in smaller quantity; –, absent

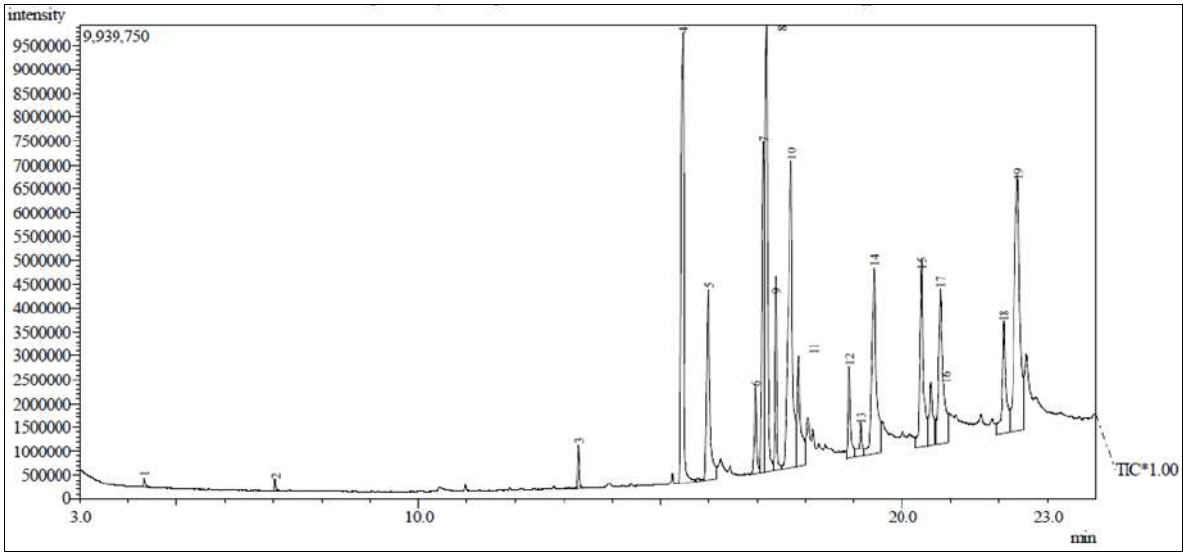

Fig 1: Representative Chromatogram of *Raphia palm* Extract

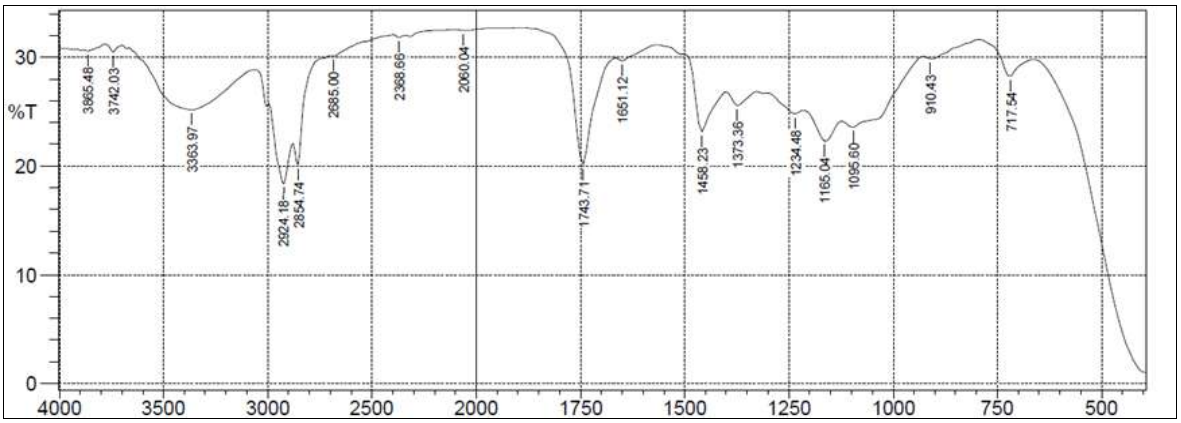

Fig 2: FT-IR Spectra of *Raphia palm* Extract

3.2 Weight Loss Measurements

The weight loss, and inhibition efficiency of mild steel coupons exposed to 0.5 M $C_6H_8O_7$ containing various concentrations of *Raphia palm* extract after two, four, six, eight and ten days respectively at room temperature, under static conditions are shown in Table 3. It was observed that, *Raphia palm* extracts inhibit the corrosion of mild steel in citric acid solution at all concentrations used in the study i.e. 0.1 – 2.0 v/v%. After the specimen immersion period of 10 days, maximum inhibition efficiency of 95.79% was attained at 2.0 v/v% concentration of the inhibitor in 0.5 M $C_6H_8O_7$. The variation of inhibition efficiency with an increase in immersion period indicates that at higher concentrations, the inhibition efficiency, η_w remained constant with the increase in immersion period, whereas at lower concentrations, η_w decreases with an increase in immersion periods. Similar sort of findings was reported elsewhere [32, 33, 29, 34]. The decrease in inhibition efficiency with increase in immersion period, according to [35] is due to increased desorption and decreased adsorption of the inhibitor on the surface of the metal.

Table 3: Corrosion Parameters Obtained from Weight Loss of Mild Steel in Aqueous Solution of 0.5 M $C_6H_8O_7$ Containing Various Concentrations of *Raphia palm* Extract at Different Intervals.

Inh. Conc. (v/v%)	Interval (number of days)									
	2		4		6		8		10	
	WL	IE	WL	IE	WL	IE	WE	IE	WL	IE
0.0	0.034	–	0.054	–	0.076	–	0.084	–	0.095	–
0.1	0.012	64.71	0.018	66.67	0.025	67.11	0.027	67.86	0.033	65.26
0.5	0.008	76.47	0.012	77.78	0.016	78.95	0.021	75.00	0.023	75.79
1.0	0.007	79.41	0.010	81.48	0.013	82.89	0.015	82.14	0.017	82.11
1.5	0.006	82.25	0.009	83.33	0.011	85.53	0.013	84.52	0.015	84.21
2.0	0.003	91.18	0.004	92.59	0.005	93.42	0.004	95.24	0.004	95.79

3.3 Potentiodynamic Polarization Measurements

Potentiodynamic anodic and cathodic polarization plots for mild steel specimens in 0.5 M $C_6H_8O_7$ 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 Tafel slopes (β_a), cathodic Tafel slopes (β_c), polarization resistance (R_p), and corrosion rate are listed in Table 4.

An analysis of the polarization curves (Figure. 3) indicates that upon the addition of *Raphia palm* extract, the corrosion potential shifts towards more positive direction. The increase in concentration of the extract also leads to a corresponding decrease in corrosion current densities [14]. However, there is no significant difference between the anodic and the cathodic overvoltage, which indicates that the extract acts as a mixed-type inhibitor. Also, as shown in the data of Table 4, the increase in the concentration of the extract resulted to a decrease in I_{corr}, β_a, β_c, and C_R values with corresponding increase in R_p and the inhibition efficiency. In addition, it can be observed that the change in β_a and β_c and the values of E_{corr} are approximately constant. 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.

3.4 Inhibition Efficiency

Values of inhibition efficiency calculated from weight loss measurements obtained after specimen immersion period of 2 days, potentiodynamic polarization (PP), and Linear Polarization Resistance (LPR) measurements are presented in Table 4. An analysis of SEM micrographs of mild steel specimens immersed in 0.5 M $C_6H_8O_7$ (Figure. 4) reveals that in the absence of *Raphia palm* extract, the mild steel surface is characterized by a very rough surface with pitted areas. This shape is typical of pitting corrosion. In the presence of the extracts, the rough surface is visibly reduced, indicating the formation of a protective film on the surface of the metal. This establishes that, *Raphia palm* extracts exhibit good inhibitive properties for the corrosion of mild steel in citric acid solution. However, there is still some evidence of pits on the metal surface.

The values of inhibition efficiency calculated from weight loss measurements obtained after specimen immersion period of 2 days, potentiodynamic polarization (PP), and linear polarization resistance (LPR) measurements shown in Table 5 are in good agreement with each other. This indicates that the inhibition efficiency is dependent on the type and nature of inhibitor used rather than the method adopted for measurement.

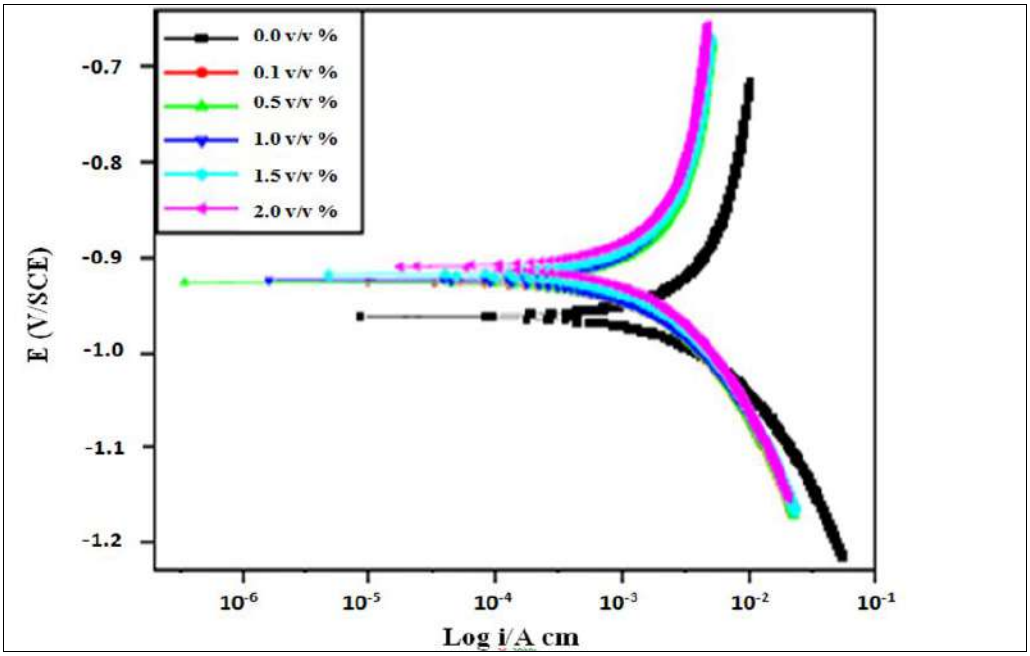

Fig 3: Polarization Curves for Mild Steel in 0.5 M $C_6H_8O_7$ Containing Various Concentrations of *Raphia palm* Extract

Table 4: Kinetic Parameters Derived from Polarization Curves of Mild Steel Corrosion in Aqueous solution of 0.5 M $C_6H_8O_7$ Containing Various Concentrations of *Raphia palm* Extracts

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	– 0.9915	0.000340	0.21809	0.23029	3.95	14.32
0.1	– 0.9732	0.000120	0.10752	0.11285	1.38	41.54
0.5	– 0.9701	0.000071	0.10607	0.11030	0.82	71.63
1.0	– 0.9560	0.000067	0.09765	0.10341	0.79	75.01
1.5	– 0.9526	0.000061	0.05957	0.05393	0.70	89.72
2.0	– 0.9415	0.000029	0.03645	0.03216	0.36	146.12

Table 5: Values of Inhibition Efficiency for 0.5 M $C_6H_8O_7$ Calculated from Weight Loss, Potentiodynamic Polarization, and Linear Polarization Resistance Measurements

Inh. Conc. (v/v%)	Inhibition Efficiency (%)		
	WL	PP_{av}	LPR
0.1	64.71	64.89	65.53
0.5	76.47	79.18	80.01
1.0	79.41	80.15	80.91
1.5	82.25	82.17	84.04
2.0	91.18	91.19	90.20

3.5 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 $C_6H_8O_7$ 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 a corroded and inhibited metal surface respectively.

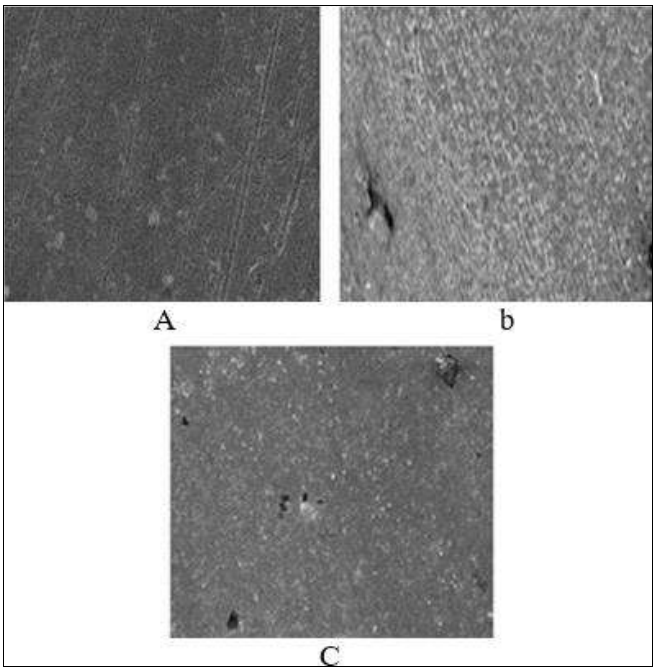

Fig 4: SEM Micrographs obtained at 1500x Magnification of (a) polished mild steel; (b) mild steel immersed in 0.5 M $C_6H_8O_7$ and (c) mild steel immersed in 0.5 M $C_6H_8O_7$ Containing 1.5% *Raphia palm* extract

3.6 Mechanism of Corrosion Inhibition

In the absence of the 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 [36].

4. Conclusions

Extracts from fruit peels of *Raphia palm* comprises several compounds most of which are fatty acids such as palmitic acid, linoleic acid, oielic acid, stearic acid, etc. and saponins, glycocides, alkaloids, flavonoids, and phenobutinones. The adsorption of these compounds on mild steel surface reduces the surface area available and inhibits corrosion.

Weight Loss measurements showed that *Raphia palm* extract inhibits the corrosion of mild steel in citric acid solution at all concentrations used in the study, i.e. 0.1 – 2.0 v/v%. The maximum inhibition efficiency of 94.74% was attained at 2.0 v/v% concentration of extract in 0.5 M $C_6H_8O_7$. The inhibition efficiency was found to vary with concentration of extract as well as period of immersion; at higher concentrations, the inhibition efficiency remained constant with an increase in immersion period, whereas at lower concentrations the inhibition efficiency decreased with an increase in immersion period. The decrease in inhibition efficiency, with an increase in immersion period is attributed to increased desorption and decreased adsorption of the inhibitor on the metal surface. Electrochemical results showed that the inhibition efficiency increases with an increase in concentration of the inhibitor. The Scanning Electron Microscopy (SEM) studies confirm the formation of a protective layer of *Raphia palm* extract on the surface of the metal.

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.

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