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Comparative analysis of corrosion inhibition on mild steel by parts of *anogeissus leiocarpus* in acidic medium

S. J. I. Ibejekwe^{a,*}, B. Eke Uche^b, Sunday Elaigwu^a, J. R. Waziri^a^aChemistry Department, School of Sciences, FCE Pankshin, P.M.B. 027, Pankshin, Plateau, Nigeria^bChemistry Department, Faculty of Physical Sciences, University of Ilorin, P.M.B. 1515, Ilorin, Nigeria

ABSTRACT

This research investigates the corrosion inhibition by parts of *Anogeissus leiocarpus* on mild steel in 0.5 M H₂SO₄ under temperature conditions of 30-60 °C and exposure time of 3, 6 and 9 h using weight loss, and Potentiodynamic polarization (PDP) methods. The inhibition efficiencies of the parts follows the trend: root (91.73 %) and leaf (89.64 %). The result shows with increase in the concentration (0.2 g, 0.4 g, 0.6 g and 0.8 g) of the methanol extract, inhibition efficiency increases. Temperature and exposure time increase reveals increase in corrosion rate, hence decrease in inhibition efficiency (IE %). The gas chromatography mass spectroscopy (GCMS) result for the root and leaf reveals the phytoconstituents like Hexadecanoic acid, ethyl ester (30.42 %), DL-alpha-Hydroxylysine (18.27 %), Glycerol 1-palmitate (17.63 %), 9-Octadecenoic acid (Z)-, methyl ester (29.45 %), Benzene, 2-methoxy-4-methyl-1-(1-methylethyl)-(21.78%), and Norcodeine (11.21 %) respectively may be responsible for the variation in the inhibition efficiencies between root and leaf.

Keywords: *Anogeissus leiocarpus*, Potentiodynamic polarization (PDP), Weight loss, Gas chromatography mass spectroscopy (GCMS).

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1. INTRODUCTION

Corrosion is an electrochemical reaction between the corroding agent (which can be oxygen and water, or any corrosive material), and the material being corroded. Materials like metals and their alloys are indispensable materials for the design and fabrications of water treatments plants, heat exchangers, dye-bath, drill bits, food processing equipment, hollow pipes for fluid transportation, distribution and storage, etc. filter membrane, etc. However, it is

observed over time that scales of inorganic materials or products develop on the internal walls of these components and reduced the efficiency or productivity. On the other, removing these unwanted products via acid cleaning or acidizing process with dilute mineral acids (hydrogen chloride and sulphuric acid) is ideal but significant corrosion effects are introduced on the metal surface on the process. The resultant effects of metal corrosion are enormous; it causes tremendous damage to life-span and integrity of industrial tools of metallic component especially in oil and gas sectors and other chemical processing industries [1, 2], creates pollution problems which poses great health challenge to man and his environment and results to waste of economic

*Corresponding Author Tel. No.: +234-806-0868-701.

e-mail: johnibejekwe@gmail.com (S. J. I. Ibejekwe)

resources in routine corrective and preventive maintenance and material loss [3].

Since corrosion is undesirable occurrence, several mitigation techniques such as anodic and cathodic protection, utilization of corrosion resistant materials, coatings, and the usage of inhibitors have been employed for reducing or preventing corrosion. Among the explored mitigation techniques, the use of organic green corrosion inhibitors (OGCIs) appears to be gaining substantial attention because it's cheap, harmless, readily available, and environmentally accommodative [4, 5]. Natural corrosion inhibitors are more biodegradable and available compared to synthesized inhibitors [6]. OGCIs reduce or prevent corrosion when added to corrosive environments in small concentration [7]. Corrosion inhibitors in general can minimize or prevent corrosion by creating a protective film on the surface of metal which obstructs direct metal – corrosive agents connection. However, synthetic inhibitors such as Chromates, some imidazole based inhibitor are expensive and non-ecofriendly thus the shift to OGCIs [8–10].

Anogeisuss leiocarpus (marke) plays a very important role in medicinal science and it has high medicinal value due to its anti-fungal, anti-bacterial and other nutritional benefits. Thus, it has been in use in many parts of the world as remedy and cure for many known diseases [11–13]. This present study is to draw a comparison between the root and leaf regarding their inhibition efficiency, effects of temperature and exposure time on mild steel in acidic medium. Furthermore, this work seeks to reveal some phytoconstituents that may have direct bearing on corrosion inhibition due to their high concentration as shown by Gas chromatography mass spectroscopy (GCMS) result [14, 15].

2. EXPERIMENTAL SECTION

2.1. PREPARATION OF MATERIALS

The metal used for the research was mild steel low carbon grade with the following composition: 0.226 % C, 0.115 % Si, 0.297 % Mn, 0.032 % P, 0.010 % S, 0.034 % Cr, 0.023 % Ni, 0.0054 % Al, 0.0096 % Cu, 0.0035 % Co, 0.0098 % Nb, 0.0036 % V, 0.0031 % Pb, 0.0056 % Sn, 0.015 % As, 0.0048 % Ca, 0.0064 % Ce, 0.0049 % Zr, 0.0022 % La and 99.2 % Fe. The mild steel specimens for gravimetric experiment were cut into coupons with dimensions 2 x 2 x 0.14 cm size using mechanical cutter. The metals were abraded and polished with fine emery paper, washed with distilled water, degreased with ethanol and dipped in acetone to prevent corrosion. The coupons were kept in desiccators to dry prior to experiment. *Anogeisuss leiocarpus* used was obtained from Shere, Jos east and was taken to Federal college of Forestry Jos with voucher number FHJ839. The root and leaf was washed thoroughly with distilled water to remove dirt, peeled to remove the thick back, sliced into pieces, dried thoroughly, pulverized to fine powder particle, stored in an air tight container and kept for corrosion studies. The prepared *Anogeisuss leiocarpus* sample (500 g) was introduced in 1000 ml of methanol in a beaker and allowed to stand for 72 h and kept in an aerated condition. And the obtained filtrates were further subjected to evaporation at 352 K, in order to make them free of methanol. The extract stock solutions were used to prepare different extract concentrations, by dissolving 0.2, 0.4, 0.6 and 0.8 g of it in 50 cm³ of 0.5 M H₂SO₄ for gravimetric and electrochemical analyses [14]. The

mixture was thoroughly filtered with filter paper and the solution obtained was used to prepared inhibited solution for corrosion studies.

2.2. CORROSION INHIBITION EFFECTS MEASUREMENT USING WEIGHT LOSS STUDIES.

This was carried out by carefully immersing the coupons which has been accurately weighed into a 100 ml beaker containing 100 ml of 0.5 M H₂SO₄ in the absence and presence of the different inhibitors concentration (g/l): 0.2, 0.4, 0.6 and 0.8) for an exposure period of 3 to 9 h with temperature ranging from 303 to 333 K. After the exposure time was reached, the samples were removed from the solution, washed with distilled water, dried and reweighed to the accuracy of four decimal places with which the Corrosion rate (CR) and inhibition efficiency were calculated using the equations:

$$CR = \frac{w}{A \times t}, \quad (1)$$

and

$$\%IE = \frac{w_1 - w_2}{w_1} \times 100, \quad (2)$$

where w , A and t are weight loss (mg), exposed area (cm²) and minimum time (h), respectively, while w_1 and w_2 indicate the mild steel original weight and weight loss in either an uninhibited solution (blank) or an inhibited solution with the *Anogeisuss leiocarpus* root and leaves extract.

2.3. CORROSION MEASUREMENT USING ELECTROCHEMICAL STUDIES, POTENTIODYNAMIC (PDP).

The electrochemical studies were performed using a VERSAS-TAT 400 complete dc voltammetry and corrosion system model with V3 Studio software. The mild steel was cut into a 1 cm² square area which was exposed to the corrosive media, with and without inhibitors, as working electrode, and an Ag/AgCl rod as counter electrode. A saturated calomel electrode (SCE) was used as reference electrode, and it was connected by a Luggin's capillary. The experiments were undertaken at room temperature (303 K). The working electrode was immersed in a test solution for 1 h, until a stable open circuit potential was attained. The Tafel analysis study was set from a cathodic potential of -250 mV to an anodic potential of +250 mV, with respect to the corrosion potential, at a sweep rate of 1 mV/s. The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential, to obtain the corrosion current densities (i_{corr}). Each experiment was carried out three times to estimate the electrochemical parameters reproducibility and average values which are reported [15].

$$R_p = \frac{\beta_a \beta_c}{2.303(\beta_a + \beta_c) i_{corr}}. \quad (3)$$

3. RESULTS AND DISCUSSION

3.1. CORROSION STUDY FROM WEIGHT LOSS

The inhibition efficiency and corrosion rate for mild steel in 0.5 M H₂SO₄ solution at 303, 313, 323 and 333 K in the absence and presence of leaf and root extract are given in Table 1. From

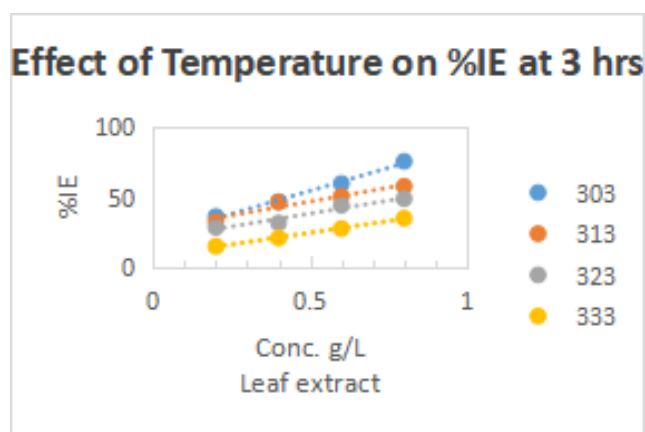
Table 1. Average values of C.R. (mpy) of mild steel gained from ML measurements, %IE, and θ of CA and CR with various concentrations at diverse temperatures.

Temp (K)	Time (h)	Con (g/l)	C.R.	θ	% IE	C.R.	θ	% IE	C.R.	θ	% IE	C.R.
303	3	BK	5.8917	-	-	10.7083	-	-	21.7000	-	-	34.2750

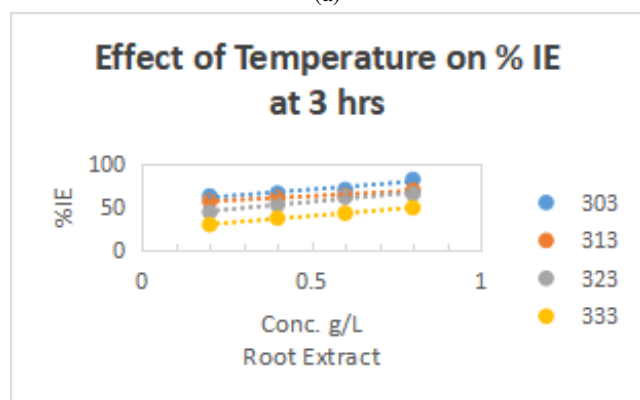
Key: P/P – Plant parts, Con. – concentration, %IE – Inhibition efficiency, Bk – Blank, C.R. – Corrosion rate, θ – Surface coverage.

Table 2. Potentiodynamic polarization (PDP) for Leaf and Root at 303 K

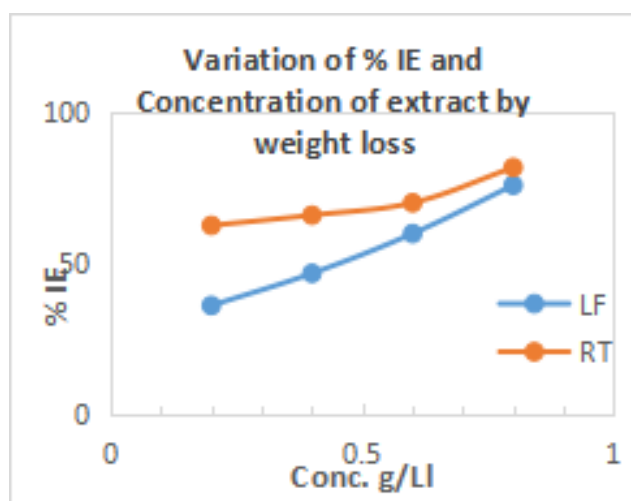
Inh Conc (ppm)	Ecorr (mV(SCE))	β_a (mV/dec)	β_c (mV/dec)	icorr ($\mu\text{A}/\text{cm}^2$)	CR (mppy)	% IE	θ
Bk	0	349.692	342.6	3292	64844.258	75.0706	-
LV	200	459.162	318.3	582.4	29945.078	53.82	0.5382



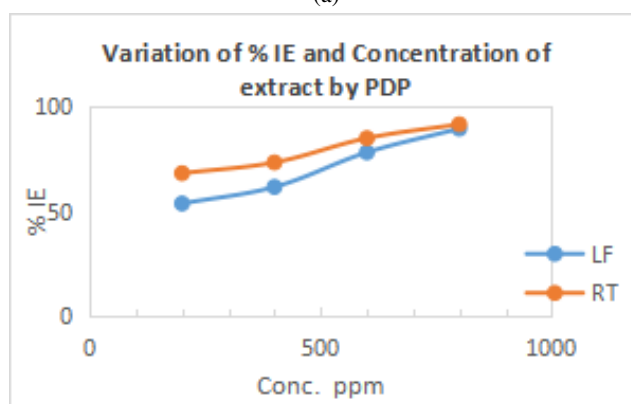
(a)



(b)



(a)

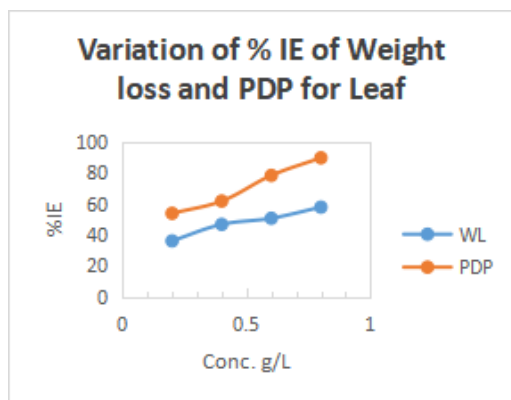


(b)

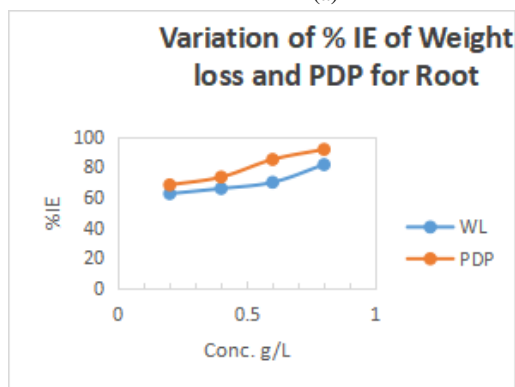
Figure 1. (a) Temperature effect on % IE on leaf. (b) Temperature effect on % IE on root.**Figure 2.** Variation of % IE and concentrations of extract by weight loss methods.

Table 1, the corrosion rate is higher in the blank solutions compared to the inhibited; which is as a result of the mitigating effect of the phytoconstituents present in both the leaf and root extract on mild steel surface. At fixed temperature, the corrosion rate decreases as the concentration of extract increases 0.2 to 0.8 g/L and thus increase in the % IE. This suggests that as the concentration of the extract increases, there is an increase in the number of adsorption of the extract constituents onto the surface of the mild steel which makes a barrier for mass transfer and prevents of further corrosion [16]. Therefore, the examined leaf and root are regarded as proficient inhibitors for the corrosion of mild steel in 0.5 M H_2SO_4 solutions. On the other hand, with increasing

temperature at fixed inhibitor concentration, the values of weight loss and C.R. was increased for both extracts, and % IE was decreased. This is in line with results obtained by [17, 18] when ciprofloxacin drug was used as an eco-friendly inhibitor. This is because of increase in the average kinetic energy of the reacting molecules [9]. In this context, the change in %IEs of the examined extracts with their concentrations acquired at 303 K temperature for 3 hours is illustrated in Figure 1(a, b). Similar plots were achieved at other temperatures but not shown here. This behavior can be attributed to the acceleration of the hydro-



(a)



(b)

Figure 3. Variation of % IE for PDP and weight loss methods.

gen evolution reaction in acidic medium with rising temperature and thus reduction in inhibitor adsorption. This suggested the mechanism of physical adsorption of the inhibitor molecules on the electrode surface [15].

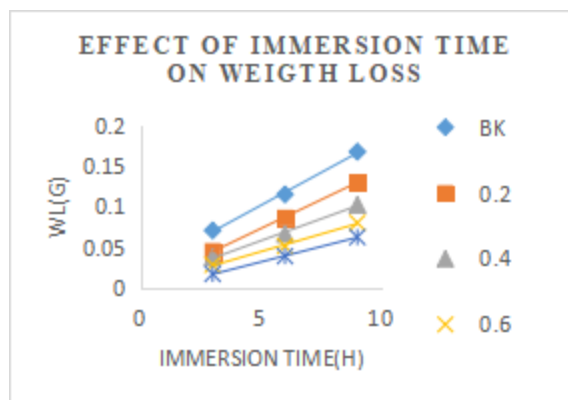
In consistence with PDP technique, the values of %IE, acquired from ML measurements, the root extract was also in general higher than those of the inhibitor, leaf as illustrated in Figures 2 and 3.

However, from the literature it was revealed that the examined leaf and root exhibited higher %IEs compared to reported organic dyes for the corrosion of mild steel in sulfuric acid solutions [16, 19–21].

The inhibition efficiency increases with increase in concentration of the extract of both leaf and root. This is attributed to the increase in the fraction of the mild steel surface covered (θ) by the adsorbed constituents of the leaf and root as the concentration of the phytoconstituent increases. The inhibition efficiency increases progressively as the concentration of extract increases up to 0.8 g/L for both weight loss PDP techniques, Figure 4.

3.2. EFFECT OF IMMERSION TIME

The results of effect of immersion time on CR and IE are shown in Table 1. The CR and IE were calculated at 3 hours interval for the total period of 9 hours in blank H_2SO_4 and in the presence of optimum concentration of both extracts (0.8 g/L) at 303 K. The corrosion rate decreases as the concentration of extract and immersion time increases. The continuous decrease in corro-



(a)



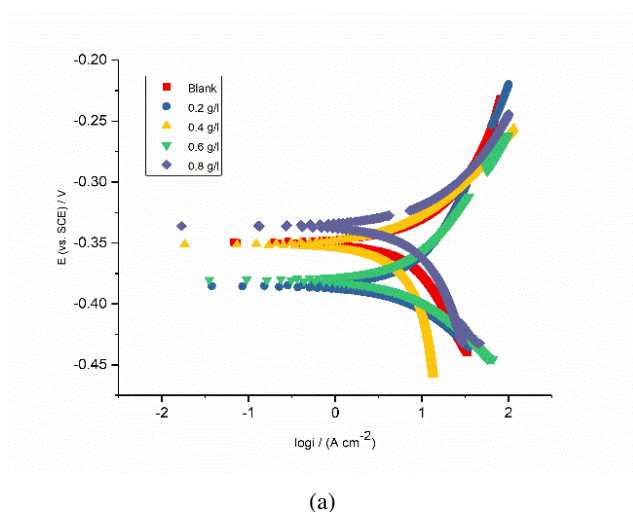
(b)

Figure 4. Weight loss against immersion time for mild steel in 0.5 M H_2SO_4 solution without and with various concentrations of: (a) Leaf and (b) Root at 303 K.

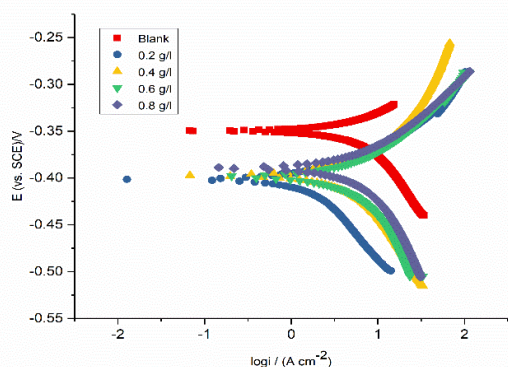
sion rate could be attributed to the formation of oxide film which shields the mild steel surface from having direct contact with the acidic environment. However, investigation have revealed that these protective oxide film can be destroy if the immersion time is extended to days thus resulting increase in corrosion rate and decrease in % IE [9].

3.3. POTENTIODYNAMIC POLARIZATION (PDP)

The PDP curves of mild steel in 0.5 M H_2SO_4 solution at 303 K without and with various concentrations of root and leaf are shown in Figure 5. The average values of the corrosion parameters, viz. corrosion potential (E_{corr}), anodic and cathodic Tafel slopes (β_a and β_c), corrosion current density (i_{corr}), polarization resistance (R_p), %IE, and surface coverage (θ) of the examined extracts, were determined and are listed in Table 2. From Figure 5(a,b) and the data inserted in Table 2, it can be noticed that adding the extracts to the corrosive medium transformed both anodic and cathodic branches of the polarization curves to lower current densities. This behavior indicated delay of both anodic and cathodic reactions and then inhibition of mild steel corrosion. The value of E_{corr} for mild steel in the corrosive medium was shifted to positive directions as a result of adding the root and leaf extracts, revealing that these compounds act as mixed type inhibitors with a major anodic type [17, 22]. The value of β_a in the corrosive medium did not change noticeably with ad-



(a)



(b)

Figure 5. The influence of concentration of extract on the polarization parameters in the absence and presence of methanol extract of *A. leiocarpus* (a) leaf (b) root tafel plot.

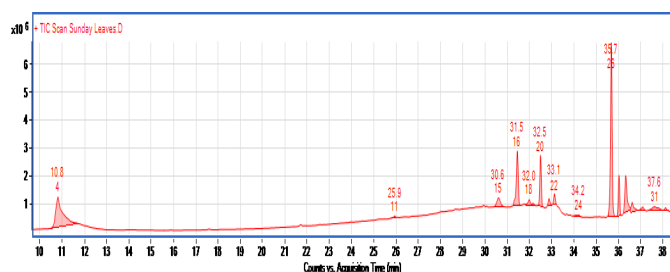


Figure 6. Gas chromatogram (GC) of the methanol extract of *Anogeissus leiocarpus* leaf.

dition of the extracts inhibitors while the β_c value was gradually increased. This suggested that the adsorbed molecules did not affect the anodic metal dissolution and enhanced the cathodic hydrogen evolution. Also, the value of i_{corr} of mild steel in the corrosive medium was reduced with increasing the concentration of the extracts indicating protection impacts. However, the obtained value of R_p of the corrosive medium was found to increase with increasing root and leaf concentrations, indicating a decrease in the corrosion rate of mild steel in the presence of the extracts. The results gotten showed that, under same investiga-

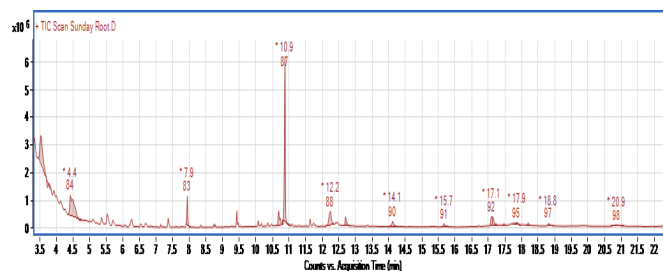


Figure 7. Gas chromatogram (GC) of the methanol extract of *Anogeissus leiocarpus* root.

tional conditions, the %IE of the root was higher than that of the inhibitor leaf. This behavior may be attributed to the high presence of Hexadecanoic acid, ethyl ester (30.42 %) in root as shown by the GCMS result which enhances the inhibition performance in plants.

3.4. GAS CHROMATOGRAPHY MASS SPECTROSCOPY (GCMS)

Figures 6 and 7 are the GCMS analysis. The prominent compounds among 17 compounds found in leaf are 9-Octadecenoic acid (Z)-, methyl ester (29.45 %), Benzene, 2-methoxy-4-methyl-1-(1-methylethyl)- (21.78%) and Norcodeine (11.21 %). While that of root with 16 compounds are Hexadecanoic acid, ethyl ester (30.42 %), DL- α -Hydroxylysine (18.27 %) and Glycerol 1-palmitate (17.63 %) in higher proportion. The high percentage of Hexadecanoic acid, ethyl ester may be the reason for the high inhibitions efficiency. This investigation has shown that due to the long carbon chain, it forms protective film on metals by absorbing onto the surface by hydrophobic and hydrophilic interaction enhanced by the ester group attached to the carbon chain [16].

4. CONCLUSION

This research have shown that both root and leaf are good corrosion inhibitor in comparison to organic dyes in an acidic medium. The higher the concentration of both extract, the higher the inhibition efficiency and vice versa. Increase in temperature and exposure time does result in corrosion rate increase. The reason for higher % IE of root compare to that of leaf is due to the high presence of Hexadecanoic acid, ethyl ester (30.42 %) as revealed by the GCMS chromatogram [15].

DATA AVAILABILITY

The data will be available on request from the corresponding author.

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