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Comparative Analysis for Corrosion Inhibition on Mild Steel by Seed and Stem of *Anogeisuss leiocarpus* in Acidic Medium

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ABSTRACT

This research work was investigated to compare corrosion inhibition by parts of *Anogeisuss leiocarpus* on mild steel in 0.5 M H_2SO_4 under temperature conditions of 30-60 ℃ and exposure time of 3-, 6- and 9-hours using weight loss, and PDP. The inhibition efficiencies of the parts follow the trend: seed (95.65 %) and stem-bark (88.09 %). Increase in the concentration (0.2 g/L, 0.4 g/L, 0.6 g/L and 0.8 g/L) of the methanol extracts resulted in % IE increase but decreased with temperature increase thus, increase in corrosion rate. The GCMS analysis of the extract reveals that 9-Octadecenoic acid (Z)-, methyl ester (45.2%), Hexadecanoic acid, methyl ester (18.09 %), 9,12Octadecadienoic acid (Z,Z)-, methyl ester (17.38 %), Methyl stearate (12.43 %), Maltitol (18.74), 2Quinazolineacetic acid, 6-chloro-1,2,3,4-tetrahydro-2-(methoxycarbonyl)-4-oxo-3-phenyl-, methyl ester (11.42 %), Ethyl Oleate (16.21%), and Oleic Acid (10.95 %) are prominent compounds. The Phytochemicals screening shows that following metabolites like saponinns, tannins, flavonoids, carbohydrates, steroids and cardiac glycosides are present in the extract. These metabolites may be responsible for inhibition.

KEYWORDS: *Anogeisuss leiocarpus,* weight loss, PDP, mild steel, IE %, Metabolites, GCMS.

1. INTRODUCTION

Long chain organic compounds have been reported to be efficient corrosion inhibitors due to their ability to cover the surface of mild steel and because of its hydrophobic nature thus preventing the metal surface from reacting with moisture within the environment. In addition, some of these compounds are attracted to functional groups such as nitrogen, sulphur and oxygen in conjugated systems thus blocking active sites or forming a protective barrier on steel surfaces [1,2,3]

Recent investigation have shown that most herbal plants for treatment of some diseases by traditionalist among our locals[4,5] are good sources of green inhibitor. *Anogeisuss leiocarpus* is one such medicinal plants used for the treatment of diseases like toothache, diarrhea, respiratory infections, jaundice, hepatitis, haemorrhoids and headache [6,4]. This plant is common in the middle-belt and in the far north of Nigeria[7,4]. The purpose of this investigation is to draw a comparative study on extracts from the seeds and stem of *Anogeisuss leiocarpus* to ascertain which has the highest inhibition efficiency, % IE on mild steel in 0.5M H_2SO_4 solution using weight loss and Potentiodynamic polarization (PDP) methods. This will also x ray the phyto-compounds present in both methanol extracts through photochemical analysis.

Fig 1: *Anogeisuss leiocarpus*, (DC) Guill & Perr Shere, Jos East, Plateau State

2. EXPERIMENTAL METHODS

2.1 **Gas Chromatography-Mass Spectroscopy (GC-MS)**

The sample was analyzed using Agilent technologies 7890A GC and 5977B MSD. The experimental conditions were set as follows: Hp 5-MS capillary standard non-polar column; Dimension: 30 m; ID: 0.25 mm; Film thickness: 0.5 µm. Flow rate of mobile phase (carrier gas: He) was set at 1.0 ml/min. Oven temperature was raised from 298 K to 313 K at 278 K/min, and injection volume was 1 µl. Samples dissolved in methanol were fully scanned at the range of 40-650 m/z and the results were compared by using NIST mass spectral library search programme.

2.2. Preparation of Specimen.

The composition of the mild steel used in this investigation are 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. This mild steel used in this research were obtained from building materials, Bukuru express, Jos south, Plateau state. And 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.

2.3. Preparation of Plant Extract.

Anogeisuss lieocarpus used was obtained from Shere, Jos east and was taken to Federal college of Forestry Jos for identification with voucher number FHJ839. The Seed and Stem 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* samples (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. 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 g, 0.4 g, 0.6 g and 0.8 g of it in 50 cm^3 of 0.5 M H_2SO_4 for gravimetric and electrochemical analyses [8].

2.4. Weight loss Measurement

This was carried out by carefully immersing the coupons which has been accurately weighed into a 100 mL beaker containing 100 mL 0f 0.5 M H_2SO_4 in the absence and presence of the different inhibitors concentration (g/L): 0.2 g/L, 0.4 g/L, 0.6 g/L and 0.8 g/L for an exposure period of 3 to 9 hours with temperature ranging from 303 K 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:

$$CR = \frac{wl}{A \times T} \times 1000 \left(mgcm^{-2}hr^{-1}\right) \quad (1)$$

And

$$w^1 - w^2$$

$$\%IE = \frac{\quad}{w_1} \square 100 \quad (2)$$

where w, A and t are weight loss (mg), exposed area (cm^2) 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 leiocapus* seed and stem extract [8].

2.5. Potentiodynamic Polarization Measurement

The electrochemical studies were performed using a VERSASTAT 400 complete dc voltammetry and corrosion system model with V3 Studio software. The mild steel was cut into a 1 cm^2 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 (303K). The working electrode was immersed in a test solution for 1hr, 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 1mV/s. The linear Tafel segments of the anodic and cathodic curves were extrapolated to corrosion potential, to obtain the corrosion current densities (icorr). Each experiment was carried out three times to estimate the electrochemical parameters reproducibility and average values which are reported [8, 9].

$$\%IE = \frac{I^0_{corr} - I_{corr}}{I^0_{corr}} \times 100 \quad (3)$$

3. RESULTS AND DISCUSSION

3.1 Gas Chromatography-Mass Spectroscopy (GC-MS)

For the seed extract, 8 compounds were identified and their retention time, percentage area, compound name, are listed in Table 1a. 9-Octadecenoic acid (Z)-, methyl ester (45.2 %), Hexadecanoic acid, methyl ester (18.09 %), and 9,12-Octadecadienoic acid (Z,Z)-, methyl ester (17.38) are the abundant component. Table 1b shows the GCMS analyses for the stem extract and revealed 16 compounds with Maltitol (18.74 %) as the major component. Double bonds and heteroatoms are found in the structures of the identified compounds contain double bonds and oxygen, which suggests the use of the seed and stem extract of *Anogeisuss leiocarpus* as corrosion inhibitor.

Table 1a. GCMS Analysis for seed extract of *Anogeisuss leiocarpus*

Peak	Compound	RT	Area	Area %	Area Sum %
1	Diglycerol	2.9	536667.87	7.95	3.6
2	Hexadecanoic acid, methyl ester	13	2700493.51	40.02	18.09
3	Eicosanoic acid	13.5	67923.46	1.01	0.46
4	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	14.7	2594057.17	38.45	17.38
5	9-Octadecenoic acid (Z)-, methyl ester	14.8	6747134.33	100	45.2
6	Methyl stearate	15	1854770.42	27.49	12.43
7	Palmitoleic acid	15.2	301625.86	4.47	2.02
8	Eicosanoic acid, methyl ester	17.7	123060.39	1.82	0.82

Table 1b. GCMS Analysis for stem extract of *Anogeisuss leiocarpus*

Peak	Compound	RT	Area	Area %	Area Sum %
1	3,4-Methylenedioxyamphetamine	11.2	6782570.38	35.93	6.73
2	4H-1,2-Diazepine, 5-(4-chlorophenyl)-3,7-diphenyl-	22.4	3291218.38	17.43	3.27
3	5β-Pregnane-3α,20α-diol	26.3	7042330.5	37.31	6.99

4	Maltitol	27.4	18877674.7	100	18.74
5	2-Quinazolineacetic acid, 6-chloro-1,2,3,4-tetrahydro-2-(methoxycarbonyl)-4-oxo-3-phenyl-, methyl ester	30	11506740.8	60.95	11.42
6	Pipercallpsine	31.8	582017.85	3.08	0.58
7	Benzoic acid, 2,4-bis[(trimethylsilyl)oxy]-, trimethylsilyl ester	32.5	720848.66	3.82	0.72
8	Methanone, (2-methoxyphenyl)phenyl-	33.3	2954761.84	15.65	2.93
9	Ethaneperoxoic acid, 1-cyano-1-[2-(2-phenyl-1,3dioxolan-2-yl)ethyl]pentyl ester	33.7	1692350.33	8.96	1.68
10	Hexadecanoic acid, ethyl ester	33.9	5306309.57	28.11	5.27
11	2-Chloroethyl oleate	35.7	1306238.92	6.92	1.3
12	6-Octadecenoic acid	36.1	1067713	5.66	1.06
13	Oleic Acid	36.5	11031508.9	58.44	10.95
14	Ethyl Oleate	36.9	16323225.9	86.47	16.21
15	Octadecanoic acid, ethyl ester	37.3	6870794.72	36.4	6.82
16	Acetyldigitoxin	37.7	5370652.71	28.45	5.33

Table 2: Phytochemical Analysis of *Anogeisuss leiocarpus* (Seed and Stem-bark).

Parameters	Seed	Stem
Alkaloid	-	-
Saponins	++	+++
Tannins	+++	++
Flavonoids	+++	+++
Carbohydrates	+++	++
Steriods	++	++
Terpenes	++	-
Anthraquinones	-	-
Cardiac glycosides	++	+

Key: + = Trace amount
++ = Moderate amount
+++ = Appreciable amount
– = Absence

Tables 1 and 2, revealed that methanol extract of *Anogeisuss leiocarpus* contains phytochemical constituents reported as good corrosion inhibitors[10]. The corrosion inhibition of mild steel by methanol extract of *Anogeisuss leiocarpus* was due to the complex chemical compositions of some phytochemical constituents that contain hetero-atoms which forms chemical bonds between the iron in the mild steel and the extract[11,12]. Research in recent times has shown that effective organic compounds used for corrosion inhibition contains hetero-atoms like phosphorous, sulphur, oxygen and nitrogen[13,14]. These hetero-atoms have lone pair of electrons which forms a protective film on metal surfaces thereby decreasing the rate of corrosion[15]. In addition, phyto-componds of long hydro-carbon chains were also found in Anogeisuss leiocarpus which are hydrophobic (water resistant) in nature[16]. This suggests good potentials of the extract of *Anogeisuss leiocarpus* as a corrosion inhibitor.

Table 3: Average Values of C.R. (mpy) of Mild Steel Gained from ML Measurements, %IE, and θ of seed and stem with Various Concentrations at Diverse Temperatures.

			Temperature (K)											
P/P	**Time**		**303**			**313**			**323**			**333**		
Seed	3	Con (g/l)	C.R	θ	% IE	C.R	θ	% IE	C.R	θ	% IE	C.R	θ	% IE
		BK	5.8917			10.7083			21.7000			34.2750		
		0.2	3.1917	0.4583	45.83	5.6000	0.4770	47.70	15.2417	0.2976	29.76	28.3333	0.1734	17.34
		0.4	2.0083	0.6598	65.98	4.4250	0.5868	58.68	13.2500	0.3894	38.94	23.4583	0.3156	31.56
		0.6	1.4765	0.7494	74.94	3.6750	0.6568	65.68	11.5000	0.4700	47.00	19.0167	0.4452	44.52
		0.8	0.8833	0.8501	85.01	3.1833	0.7027	70.27	10.3667	0.5223	52.23	18.5500	0.4588	45.88
	6	BK	4.7917			9.8292			13.4333			30.1000		
		0.2	2.8833	0.3983	39.83	6.0750	0.3819	38.19	9.9625	0.2584	25.84	26.5083	0.1193	11.93
		0.4	2.3458	0.5104	51.04	5.3167	0.4591	45.91	9.0583	0.3257	32.57	23.2542	0.2274	22.74
		0.6	1.5894	0.6683	66.83	4.5000	0.5422	54.22	8.4458	0.3713	37.13	20.4042	0.3221	32.21
		0.8	1.1792	0.7539	75.39	3.2500	0.6693	66.93	7.3000	0.4566	45.66	17.0375	0.4340	43.40
	9	BK	4.6528			8.4500			11.5250			28.3472		
		0.2	3.0417	0.3463	34.63	5.3639	0.3652	36.52	8.9750	0.2213	22.13	26.9583	0.0490	4.90
		0.4	2.7222	0.4149	41.49	4.8222	0.4293	42.93	8.2944	0.2803	28.03	24.0361	0.1521	15.21
		0.6	2.3611	0.4925	49.25	4.2139	0.5013	50.13	7.8250	0.3210	32.10	22.1861	0.2173	21.73
		0.8	1.0389	0.5618	56.18	3.3806	0.5999	59.99	6.9361	0.3982	39.82	18.3250	0.3536	35.36
Stem	3	BK	5.8917			10.7083			21.7000			34.2750		
		0.2	3.0889	0.4757	47.57	5.7750	0.4607	46.07	14.0083	0.35.45	35.45	24.2000	0.2939	29.39
		0.4	2.1833	0.6294	62.94	4.5500	0.5751	57.51	11.7583	0.4581	45.81	23.2167	0.3226	32.26
		0.6	1.8917	0.6789	67.89	4.1833	0.6093	60.93	10.2667	0.5269	52.69	21.2967	0.3787	37.87
		0.8	1.7167	0.7089	70.86	3.9333	0.6327	63.27	9.3917	0.5672	56.72	19.2167	0.4393	43.93
	6	BK	4.7917			9.8292			13.4333			30.1000		
		0.2	2.6000	0.4574	45.74	7.1694	0.2706	27.06	10.0708	0.2503	25.03	22.2625	0.1607	16.07
		0.4	2.2250	0.5357	53.57	6.0375	0.3858	38.58	8.9083	0.3368	33.68	21.0125	0.3019	30.19
		0.6	1.9750	0.5878	58.78	5.6208	0.42.82	42.82	7.7958	0.4197	41.97	19.6917	0.3458	34.58
		0.8	1.5083	0.6852	68.52	4.8083	0.5108	51.08	7.1708	0.4662	46.62	17.5292	0.4118	41.18
	9	BK	4.6528			8.4500			11.5250			28.3472		
		0.2	2.7167	0.4161	41.61	6.1917	0.2673	26.73	8.5611	0.2572	25.72	26.2500	0.0740	7.40
		0.4	2.3481	0.4953	49.53	5.7500	0.3195	31.95	7.8639	0.3177	31.77	24.6222	0.1314	13.14
		0.6	2.0639	0.5564	55.64	4.8917	0.4211	42.11	7.1000	0.3839	38.39	19.7000	0.3050	30.50

0.8	1.8750	0.5970	59.70	4.3778	0.4819	48.19	6.6250	0.4252	42.52	17.7833	0.3727	37.27

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

Table 3, gives a general picture of the relationships that exist between the following parameters; corrosion rate, surface coverage, inhibition efficiency, temperature effect, exposure time effect and effect of concentration.

Figure 2, reveals the effect of temperature on inhibition efficiency. The inhibition efficiency decreases with increase temperature and this indicates physical adsorption[17,18]. The kinetic energy of the extracts increases due to the increase in temperature, thus, making adsorption insufficient between extract and surface of the mild steel at the binding sites[19,20].

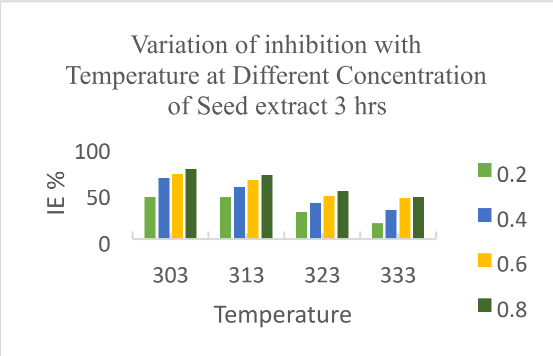


Fig 2a: IE % and Temperature

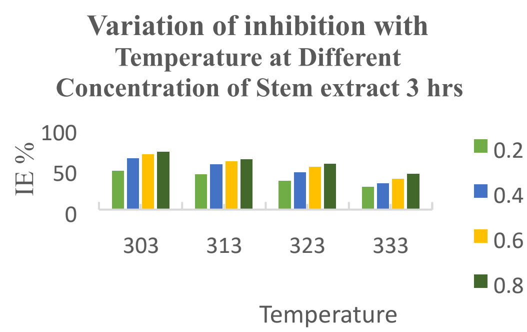


Fig 2b: IE % and Temperature

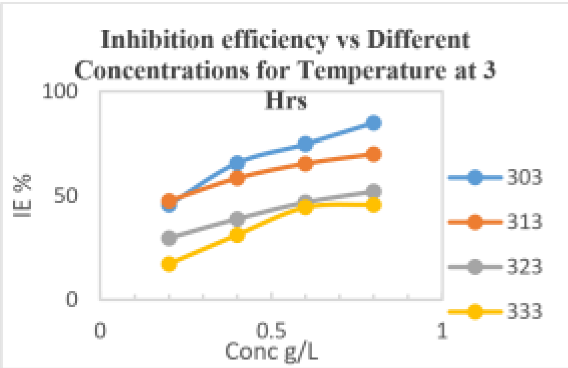


Fig 3a: IE % and Concentration.

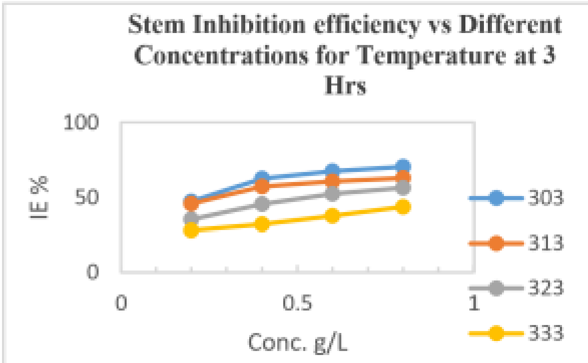
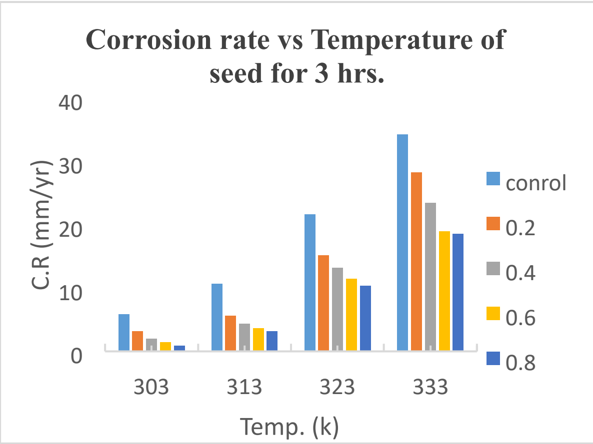


Fig 3b: IE % and Concentration.

For specific temperature, Fig 3, it was observed that with increase in the concentration of the extracts, the inhibition efficiency also increased to maximum of 70.86 % and 85.01 % for stem-bark and seed respectively. This implies that both extracts exhibited a good anticorrosion effect on the surface of the mild steel by forming films. The result clearly showed that the inhibition mechanism involves blocking of mild steel surface by inhibitor molecules via adsorption [21].

Also the corrosion rate decreases with increase in the inhibition efficiency due to increasing adsorption coverage of inhibitor molecules on the steel surface with their concentrations, which decreased the dissolution rates of mild steel [17]. But fig 4 revealed that with temperature increase, the corrosion rate also increased. The reason for this is due to acceleration of the hydrogen evolution reaction in acidic medium with rising temperature and thereby reducing the rate of inhibitor adsorption. This again is an indicator of physical adsorption mechanism of the extracts molecules on the electrode surface [18].



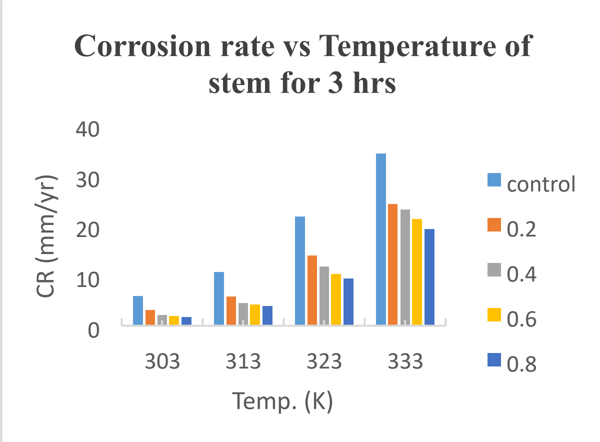


Fig 4a: Corrosion rate vs Temperature

Fig 4b: Corrosion rate vs Temperature

3.2 Potentiodynamic Polarization Graph

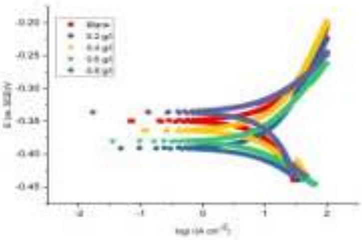


Fig 5a: Stem-bark

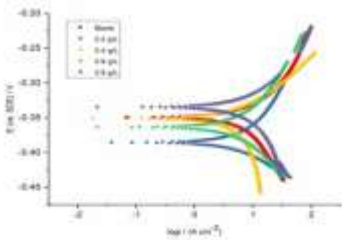


Fig 5b: Seed

Table 4: Potentiodynamic Polarization
Average Corrosion Parameters Acquired from PDP Curves in the Corrosion of Mild Steel in 0.5 M H_2SO_4 Solution without and with Various Concentrations of Seed and Stem 303K.

	Inch Conc (ppm)	E_{corr} - (mV(SCE))	β_a (mV/dec)	-β_c (mV/dec)	I_{corr} (μA/ cm^2)	CR (mppy)	% IE	θ
BK	0	349.692	342.6	3292480.256	64844.258	75.0706	-	-
SE	200	385.501	174.8	91.0	11428.698	13.2311	82.38	0.8238
	400	351.127	91.7	1127.8	11501.781	13.3157	82.26	0.8226
	600	363.394	183.3	175.7	7398.728	8.5681	88.59	0.8859
	800	334.694	191.9	1295.2	2820.725	3.2645	**95.65**	0.9565
ST	200	343.306	155.3	405.1	20498.559	23.7313	68.39	0.6839
	400	363.989	207.3	141.7	15905.291	18.4137	75.47	0.7547
	600	380.232	115.2	80.9	9366.628	10.8438	85.56	0.8556
	800	396.118	101.6	65.6	7722.951	8.9409	**88.09**	0.8809

The result for inhibition efficiency in table 4 was computed using equation 3. Thus *Anogeisuss leiocarpus* extract is an organic inhibitor which has no heavy metal and is environmentally safe to use for minimizing corrosion of mild steel metals in acidic medium.

4. CONCLUSION

The methanol extracts for both seed and stem revealed high percentage of 9-Octadecenoic acid (Z)-, methyl ester (45.2 %), Hexadecanoic acid, methyl ester (18.09 %), 9,12-Octadecadienoic acid (Z,Z)-, and methyl ester (17.38 %), Maltitol (18.74 %) and Ethyl Oleate (16.21 %). These account for corrosion inhibition on the surface of the mild steel. The seed extract have higher inhibition efficiency compare to the stem due to the high presence of 9-Octadecenoic acid (Z)-, methyl ester (45.2 %) for both PDP and weight loss methods. Increase in concentration of both extract resulted in increase in IE %. Temperature and exposure time increase resulted in decrease in IE %, thus indicating physical adsorption mechanism.

CONFLICT OF INTERESTS

The authors declare no conflict of interests.

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