

Synergized Plants Extract as Corrosion Inhibition Enhancer for Galvanised Steel in 1.0 M HCl Media

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Abstract

*The enhancement of corrosion inhibition of galvanised steel by synergised leave extracts of *Sida acuta* (S.A.) and *Jatropha curcas* (J.C.) in 1.0 M HCl solution was investigated using the gasometric technique at 30 °C and 50 °C. Results obtained showed that complimenting plant extracts with galvanisation of steel enhanced the inhibition of Zinc in the protection of steel. Fourier transform infrared analysis confirmed the O-H functional group of alcohol at 3851 and 3436 cm^{-1} band. Surface morphology of the steel, synergised were studied with a scanning electron microscope (SEM) and electron dispersive X-ray spectroscopy (EDX). Analysis revealed the protection of the internal grains of the metal from dissolution into corrodents provided by the adsorbed film the extract. Inhibition efficiency improved from 57.57 % to 95.45 %, corrosion rate 0.454 cm^3/hr to 0.093 cm^3/hr for zinc-coated steel only to extract enhanced-zinc coated steel. Langmuir isotherm best explained the adsorption mechanism of extract of zinc surface. Zinc coated steel has a half-life less than $0.04531539 \times 10^{10}$ minutes, while effective enhancement of Zinc coated steel with extract gave a half-life of $0.075132088 \times 10^{10}$ minutes. This implies that the enhancement of galvanised (Zinc coated) steel with synergised extract improved the inhibitory characteristics of the composites over that of zinc coat alone, which corrodes in place of steel.*

Keywords: galvanised steel, synergised leave extract, *Sida acuta*, *Jatropha curcas*, half-life

1. Introduction

Steel corrosion is a fundamental industrial concern that has received a considerable amount of academic attention (Amitha and Bharathi, 2012). Corrosive attack on the steel pipe actually occurs from external, due to their subjection to highly oxygenated environments such as air, water, soil, and particularly those containing appreciable acidity content (Muhammad *et al.*, 2018) and also, from the acidic waste of manufacturing solid and liquid waste and fertiliser usage on farmlands. Even when they are exposed to oxygen is not available, especially in an anaerobic environment; some bacteria make corrosion still occur. It is a case where steel pipe conveying fluids are buried or made to unavoidable pass-through unpleasant environments such as river estuaries and waterlogged soil, (Richard and Ivanildo, 1997; Stefan, 2008). The use of galvanised steel pipe for civil and waterworks from inception to distribute potable water to the populace has suffered setbacks due to corrosion of the pipe, leading to leakages. The water pipes' internal wall is protected by residual constituent like calcium carbonate of the bye product of treatment, primarily



when calcium base is used to soften the hard water. However, these may cause a pile up of fur, which reduces the inflow of water, (Noble, 2013). These resulted from substandard external coating and ageing. However, such gave room for alternative substitution of the galvanised steel pipe to ceramic pipes for low-pressure applications that depend on the force of gravity for flow. Some ceramics made of sodium silicate and clay has been used to form a pipe. Polyvinylchloride (PVC) pipes of recent were more preferred. The non-corroding resistance to chemicals and ease of workability are the characteristics that made PVC advantageous over galvanised steel pipes (Twort *et al.*, 2000). The breakages of these alternative pipes due to high impact from civil works of external force, climate influence, scale formation and contamination of conveyed treated water by the dissolving pipe brought about the revisit of the steel pipe material (Mohebbi, 2011).

The use of corrosion inhibitors is one of the most practical methods for corrosion protection of steel, especially in acidic media (Ebenso *et al.*, 2010). The protection of metal surfaces against corrosion is an essential phenomenon in various technical industries, especially manufacturing and construction. Inhibitors are practical means of preventing corrosion in an aggressive environment, either acidic or basic media.

Galvanized (Zinc coated) steel is a classic example of cathodic protection of steel. The Zinc corrodes preferentially and protects the steel. Zinc acts as a sacrificial anode in contrast, Loto *et al.* (2019). Tin, which is more corrosion resistant than Zinc, is sometimes undesirable as a coating because it is usually cathodic to steel. At perforations in the Tin coating, the deterioration of the steel is accelerated by galvanic action. Magnesium is often connected to underground steel pipes to suppress their corrosion (the magnesium preferentially corrodes). Cathodic protection are also obtained by protonated organic extract constituent in solution that got adsorbed on cathodic sites of the metal retard the hydrogen gas release and the molecules component of the solution impinge the anodic dissolution of the metal (Popova *et al.*, 2003).

This action removes silver, and it is not very good for silver plates because the plating is eventually removed. Many of the stains on silverware are due to silver sulphide. A simple electrochemical cleaning method consists of placing the silver in an Aluminium jar containing water and baking sodas and not sodium chloride (Kuntzleman, 2016).

Some plant extracts act as an alternative to either mechanical or chemical means to inhibit corrosion of metals such as Zinc in acidic media (El-Etre *et al.*, 2005; Abiola and James, 2010; Abdel-Gaber *et al.*, 2012). Although Abiola and James, 2010 reported the effects of Aloe vera extract on the corrosion of Zinc in HCl solution using weight loss methods. El-Etre *et al.*, (2005) reported the effect of *Lawsonia inermis* leaf extract of the Henna plant on the corrosion inhibition of carbon steel, nickel and Zinc in acidic, neutral and alkaline solutions employing polarisation technique. All of these studies suggest the fact that green inhibitors can be cheaply sourced and applied without contaminating or of any human and environmental. They are organic compounds containing electron donor atoms, particularly nitrogen, sulfur and oxygen in their functional groups with aromatic and heterocyclic rings with corrosion-inhibiting capability.



The leaf extract contains various phytoconstituents and nature of conjugated bonds, so corrosion inhibition, in this case, could not be traced to particular species out of lots present in the extract. Since some of these constituents, including tannins, organic and amino acids, alkaloids, proteins, flavonoids, and organic pigments and their acid hydrolysis products are known to exhibit inhibiting action, this however made it difficult to assign the inhibitive effect by adsorption to a particular constituent (Zucchi and Omar 1985; Kliskic *et al.*, 2000; Martinez and Stern, 2001; Quraishi, 2004).

Given these inhibiting effects of acid extracts of some plants, origin has been attributed to protonated species resulting from the hydrolysis products. Yukiyoishi *et al.* (2012) reported that there might have been intermolecular esterification between J.C. and S.A. extracts. Because saponin was present in both J.C. and S.A. leave extracts, many works had been earlier done on the protection of the galvanised steel pipe with chromate and phosphate-based inhibitors. This is furtherance to an alternative solution to the corrosion menace of the zinc coat that quickly lost its protection on steel. This study expects elongating the life span of the steel pipe using a green inhibitor to complement the zinc coat. This synergised plant extract has earlier been incorporated into the recipe of Alkyd resin base primer as substituted to the primer of zinc chromate additives (Sodiya *et al.* 2016).

2. Materials and Method

2.1 Metal Elemental Composition Analysis

The composition of the metal samples considered for this study was determined using Metal Spectrometer Analyser of Model ARL Quanbo Desk OTD 226 (Optical emission), a Thermo Scientific make of Switzerland.

2.2 Metal Sample Preparations

A galvanised steel pipe was sliced with a lathe machine into coupons of 18.0 mm x 9.0 mm x 3.0 mm. Duplicate sets of samples were prepared. In the first set, the concave side (inner) of the metal and edges which could influence the corrosion was polished with 600 emery paper. The convex galvanised surfaces were left as received to avoid reduction or removal of the zinc coat.

The coupon thickness was determined with the Mitutoyo brand of analog micrometer screw gauge, while other dimensions were also confirmed using Mitutoyo digital venier gauge of 2 decimal places. Surface treatment of the coupons was done by degreasing in ethanol and dried with acetone. They were then stored in moisture free desiccators to prevent rusting or contamination before use, (Noor, 2005).

2.3 Extraction from Plant Leaves

Fresh leaves of *Jatropha curcas*. 10 g of the pulverised leaves were weighed into a 250 ml conical flask separately containing 100ml ethanol using analytical balance Ohaus PA4102. The flask was corked correctly and left to stand for 48 hours with occasional shakings. The resultant mixture was



filtered, and the residue thoroughly washed with ethanol. The residue (husk) was pre-soaked again in 100 ml ethanol for 24 hours. The mixtures were filtered, and both filtrates added together. The use of a rotary evaporator concentrated the extract recovered the solvent. The concentrated extract was evaporated to oily solid on water bath by gently heating at 75°C to expel the leftover ethanol finally. The exact process was carried out on *Sida acuta* simultaneously. The pH of *Sida acuta* was 6.07, and *Jatropha curcas* was 6.58 in an ethanolic solvent of pH 8.06 at 25 °C. It implies that S.A is slightly more acidic than the J.C. but were in the same pH range. Their dissolution in acid solution did not alter the properties of the solutions at synergization.

2.4 Extraction Yields of Plant Extracts

The extraction yield, also known as soluble yield, was done according to Zhang *et al.* (2007). There was an improvement in the yield of the same plant leaves in ethanolic extraction. *Jatropha curcas* gave a 6.26 %, and *Sida acuta* gave 4.48% compared with the 4.2% and 5.6 % respective yield obtained in the methanolic and ethanolic solvent for *Raphanus sativus* (Safia *et al.*, 2013)

2.5 Preparation of stock solution and various concentrations of *Sida acuta* plant extracts

1 g of the extract was dissolved in 1 litre of 1.0 M HCl to make 1g/dm³ of the extract solution. From those further dilutions were made to obtain 0.1 g/dm³, 0.35 g/dm³, 0.6 g/dm³ and 0.85 g/dm³ All reagents were of Analar grade from (BDH Chemicals, England) and distilled water was used to prepare all solutions.

2.6 Weight Loss Determination

Weighed coupons were immersed in 50 ml of 1.0 M HCl as control and 1.0 M HCl containing different concentrations (0.1 gdm⁻³, 0.35 gdm⁻³, 0.60 gdm⁻³, 0.85 gdm⁻³ and 1.0 gdm⁻³) of leave extract (*Sida acuta*) at 30 °C and 50 °C for 2 hours. The coupons were removed from the corrodent and dropped in water to discontinue corrosion. The panels were scrubbed with a brittle brush under running tap water to remove the corrosion products. It was later rinsed in ethanol and, after that, in acetone before finally air drying and weighed. Each reading was recorded up to the fourth decimal place using Ohaus PA4102 electronic analytical balance. The difference in weight of the compounds was taken as the weight loss (Abdeli *et al.*, 2010, Karthikaiselvi and Subhashini, 2014). The experiment was repeated with a concentration of 8 gdm⁻³ of J.C. and synergised with various S.A. concentrations from 0.1 to 1.0 gdm⁻³ at ratio 4:1. The weight loss of the coupons was evaluated in grams as the difference in the weight of the coupons before and after the test (Orubite and Oforka, 2004):

$$W = (W_i - W_f) \quad \text{Eq. 1}$$

Where ΔW = weight loss of coupon, W_i = Initial weight of coupon, W_f = Final weight of coupon. Each reading reported is an average of two experimental recordings to avoid deviations due to errors that might be encountered in the course of the experiments. The inhibition efficiency was estimated as (Rejendran *et al.*, 2009):



$$I. E\% = \left[\frac{(W_u - W_i)}{W_u} \right] \times 100 \quad \text{Eq. 2}$$

Where: I.E. = Inhibition Efficiency
 W_u = Weight of Uninhibited
 W_i = Weight of Inhibited

$$\theta = \frac{I.E\%}{100} \quad \text{Eq. 3}$$

Where θ = surfaced coverage

2.7 Gasometric Procedure

50 ml of 1.0 M HCl solution (control) in Mylius Cell as described by Dawodu and Sodiya, (2015). The coupon was dropped into the corrodent and corked airtight with the application of Vaseline gel. The hydrogen gas evolved by downward displacement of water was recorded on the calibrated tube (inverted burette). Readings were taken at every 1 minute interval for 20 minutes at 30°C. The experiment was repeated at 50 °C using another coupon but the same concentration and set of Synergized extract concentrations. At the end of each Hydrogen gas Evolution (HE) experiment, the coupon was withdrawn from the tested solution, washed thoroughly with water followed by acetone and dried with air (Mamatha, 2011). Duplicate data readings were taken in and recorded to 0.1cm³. The expression below was used to calculate the Inhibition Efficiency of the plant extracts:

$$\% I. E = \left[1 - \left(\frac{V_{in}}{V_{uni}} \right) \right] \times 100 \quad \text{Eq. 4}$$

Where V_{in} = Volume of hydrogen gas released from inhibited solution
 V_{uni} = Volume of hydrogen gas released from uninhibited solution
 I. E. = Inhibition Efficiency

Further studies were made by correlation analysis to obtain volume of hydrogen (cm³) evolved against time (minutes) (Mathur and Vasudevan, 1982):

$$V_{in} = c \pm bt \pm at^2 \quad \text{Eq. 5}$$

$$R = \frac{\delta v}{\delta t} = b + 2at \quad \text{Eq. 6}$$

$$R = 2at \pm b \quad \text{Eq. 7}$$

Where V_{in} is the Volume of hydrogen gas released from inhibited solution
 Corrosion rate R deduced accordingly (Ajayi *et al.*, 2011)

3. Results and Discussion

3.1 Qualitative analysis of the Plant extracts (Phytochemical screening)



The results of the plant extracts qualitative analysis are as shown in Table 1. It confirms the presence of secondary metabolites effectiveness as corrosion inhibitors which had been earlier established elsewhere to possess inhibitory properties (Amitha *et al.*, 1998; Ebuehi and Okorie, 2009; Akilandeswari *et al.*, 2010; Ahirrao *et al.*, 2011; Raimi *et al.*, 2014). Out of twelve (12) tests carried out on each plant extracts, ten (10) phytoconstituents were detected for *Jatropha curcas* and seven (7) for *Sida actuta*. The reduction in the constituents' concentration or total absence of some secondary metabolites after inhibition such as alkaloids, flavonoids, and tannins from the spent extract (after some metals had been subjected to inhibition by the plant extract) confirmed that they had been taken up to inhibit the metal at absorption. The heteroatoms constituent in the bonding structure of the secondary metabolites indicated below have been reported to be responsible for inhibiting that process.

Table 1: Phytochemicals in J.C extract, S.A extract, mixture of J.C and S.A extract before and after use for corrosion test

Detects	J.C Leaf extract	S.A leaf extract	Mixed solution use	extract before use	Mixed solution After use
Saponins	++	+++	+		-
Flavonoids	++	+++	+		-
Tannins	+	+	+		-
Phlobatanins	Nd	Nd			-
Steroids	+	+			-
Terpenoids	+	+			Nd
Cardiac Glycosides	+	Nd			-
Glycosides	Nd	Nd			-
Alkaloids	++	++	+		+
Reducing Sugar	+	Nd	+		Nd
Phenol	+	Nd	+		-
Anthraquinones	Nd	Nd			-
Resins	+	+	+		+

JC: *Jatropha curcas*, SA: *Sida acuta*, +++ = Very deeply present:

++ = Deeply present, + = Present; Nd= Not detected, - = Not present

3.2 FTIR Analysis

Fourier Transform Infra-Red Spectroscopy (FT-IR) analysis shown in Fig. 1 confirmed the presence of functional groups in the mixed leaf extract. The O-H of carboxylic acid at 3851.06 cm^{-1} ¹ Strong band of 3436.86 cm^{-1} was assigned to stretching vibrational frequency of weak O-H.



Weak bands at 1081.4 and 1045.93 cm^{-1} depict C-O stretching vibrational band of alcohol, ethers and esters present in the extract. The broad band at 2076.12 cm^{-1} of N=C=S of isocyanate. These detected functional groups in the extract contained heteroatoms Oxygen, Nitrogen and Sulphur in the bonding structure of their secondary metabolites. They are reported to be binding centres with metals atoms at adsorption as inhibitors (Abiola, 2007; Abiola and Tobun 2010; Abiola *et al.*, 2011).

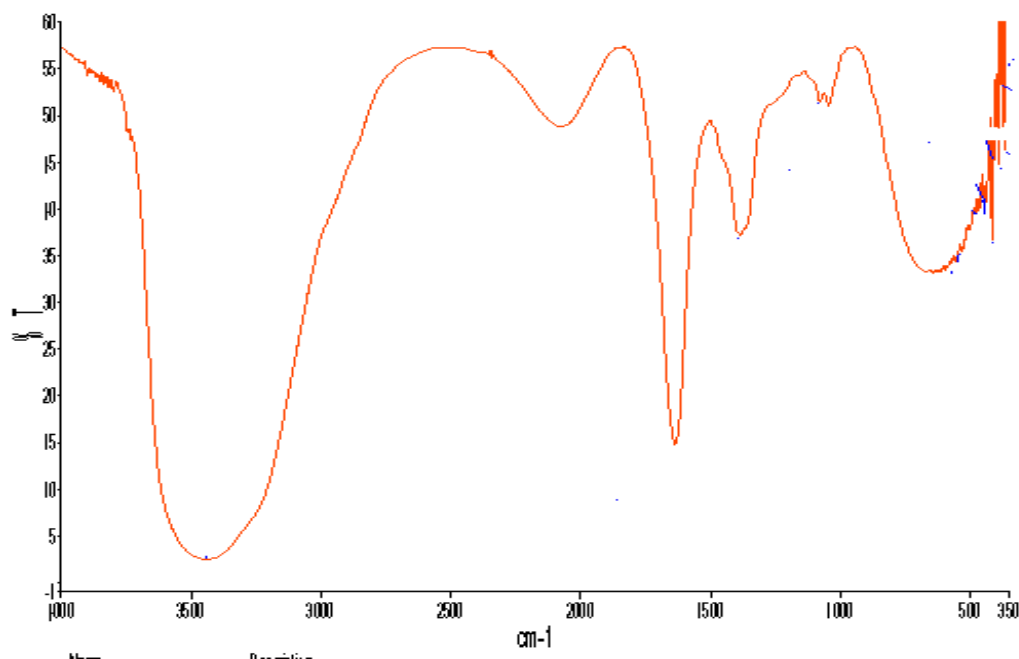


Fig. 1 Spectrum of mixture of *Sida acuta* with *Jatropha curcas* leaf extracts

3.3 Corrosion Rate.

The rate at which material corrodes is a function of the corrodent's temperature and concentration and the duration at which the material has contact with the corrodent. In Fig. 2b the amount of weight loss at the equivalent period at 50 °C was higher than that at 30 °C due to an increase in temperature that made the reaction faster. Fig. 3a and b indicated corrosion rate increase with concentration and temperature (between 30 °C to 50 °C) using equation 8.

$$\text{Log}R = B\text{Log}C_{\text{extract}} + \text{Log}k \quad (\text{Eq. 8})$$

Where B stands for the slope and the intercept is given as $\text{log } k$.



Where r is the corrosion rate, $\log r$ (mol/min) gives the log of corrosion rate, k is the specific reaction constant (mol min^{-1}), B is the reaction constant (mol^{-1}) (the negative value in this work infer decreasing slope due to the inhibitive effect of the extract on the metal) and C is the volume concentration of extracts (mol) or weight concentration (g/dm^3). Adopting this method gave a corrosion rate relationship with the extract concentration. Converting volume of hydrogen gas evolved in the presence of inhibitor from units of $\text{ml}(\text{cm}^3)$ to mol assuming hydrogen evolution took place at $1.01325 \times 10^{-5} \text{Pa}$, a conversion factor 3.83×10^9 which has been used in other studies for the same purpose was employed (Noor and Al-Moubaraki, 2008). In the same vein, a factor of 0.018700307 was employed in converting Volume of extracts in $\text{ml}(\text{cm}^3)$ to mole. This factor has been variously employed in different studies (Mathur and Vasudevan, 1982; Ajayi *et al.*, 2011). From the graph, the relationship between $\log r$ and $\log C$, which gave linear expression from where k and B were obtained?

3.4 Half-life of metal inhibited with Plant extract

The time it will take a mole equivalent of metal or specified amount of corrodible substance to damage or disintegrate by half of its original size denoted as:

$$t_{1/2} = \ln \frac{2}{k} \quad \text{Eq. 9}$$

Where $\ln 2 = 0.693147$ and k is the specific adsorption constant

From Fig 2a at 30°C of 1.0g/dm^3 , the value of $t_{1/2}$ obtained is 7.52×10^{11} mins and from Fig. 2b at 50°C , the value of $t_{1/2}$ obtained is 3.64×10^{10} mins.

A decrease in half-life with an increase in temperature was observed. The half-life of this galvanised steel enhanced with plant extract for corrosion inhibition was also affected by the concentration of extract. At 1.0g/dm^3 concentration of synergized extract that gave 95.45 % I.E. with the zinc coat at half-life ($t_{1/2}$) of 7.52×10^{12} mins. At 0.1g/dm^3 concentration of synergised extract, which gave 57.57 % I.E, the fraction of efficiency reduction is 0.603. The extract concentration decreases which resulted in a reduction in half-life. The little or no effect of extract inhibition of corrosion reaction on the metal will corrode fast and diminished in size in a shorter duration of time. This implies that zinc coat steel with no plant extract will have a half-life less than 4.53×10^{11} mins at an efficiency reduction of decimal of 0.6, while effective enhancement of zinc coat steel with plant extract will gave a half-life of 7.51×10^{12} mins.



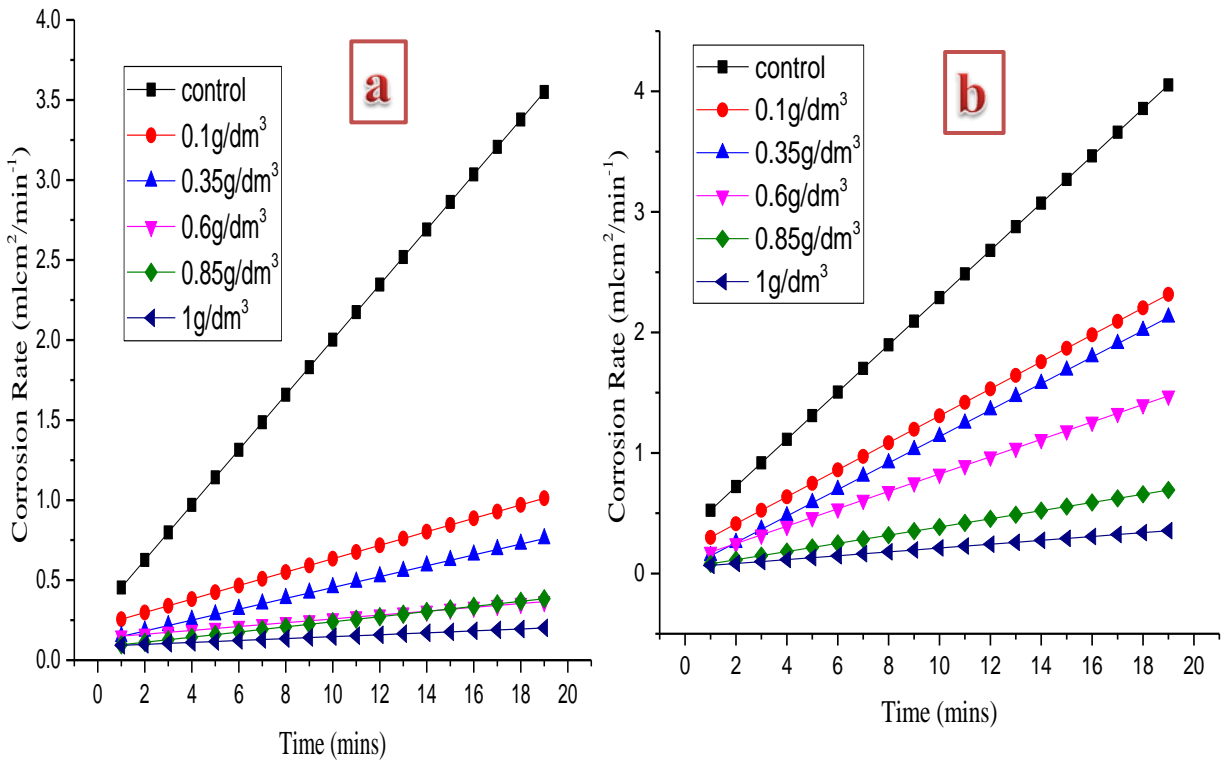


Fig. 1 Graph of corrosion rate (mlcm²/min⁻¹) of varying volume dilution of extracts with time intervals (minutes) for galvanised mild steel corrosion (a) at 30°C and (b) at 50°C



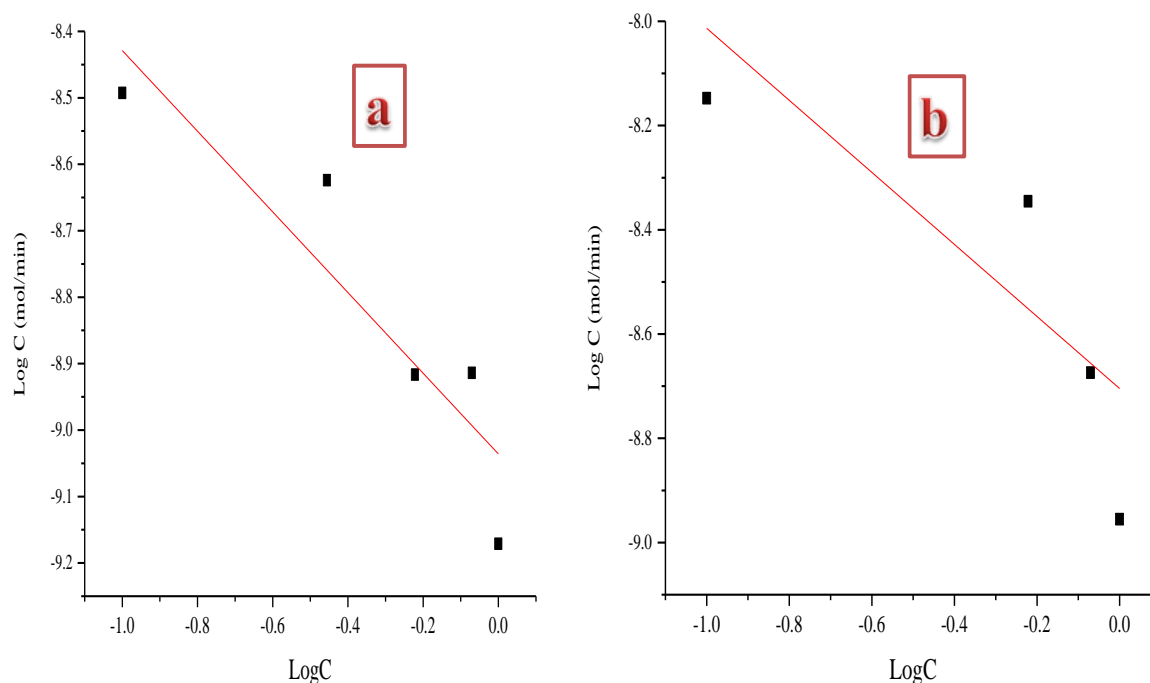


Fig. 2 The plot of Corrosion log rate r (mol/min) against the log of varying concentration C (mole) of synergised extract at x minutes for galvanised mild steel inhibition in the presence of 1.0 M HCl (a) at 30 °C. (b) at 50 °C.

3.5 Adsorption Studies

The mechanism of interaction at the interface between the zinc coat and the extract was best explained by Langmuir, among other isotherms where the adsorption site of the extracts can cover only one site at a time on the galvanised surface. The value of coefficient determination (R^2) and correlation coefficient (R) is subsequently at maximum tending to 1.0 as deduced from straight-line graphs of the plot of C/θ against C as depicted in Fig. 3.

$$\frac{\theta}{\theta - 1} = kC \quad (\text{Eq. 10})$$

Dividing both sides θk and then rearrange, we then have:

$$\frac{C}{\theta} = \frac{1}{k} + C \quad (\text{Eq. 11})$$

Where θ is the surface coverage, C stands for concentration and k is characteristics constant related to adsorption intensity or degree of favourability of adsorption. It's coefficient of determination R^2 is 0.996 at 30 °C and R^2 is 0.988 at 50 °C. The measure of the explanatory power of the model coefficient (R) is 0.998 and 0.955 at 30 °C and 50 °C respectively. This showed the favourability of adsorption to Langmuir adsorption isotherm at 30 °C than 50 °C.



The inhibiting action is performed via adsorption of the extracts on the zinc surface, which is physical in monolayer characteristics.

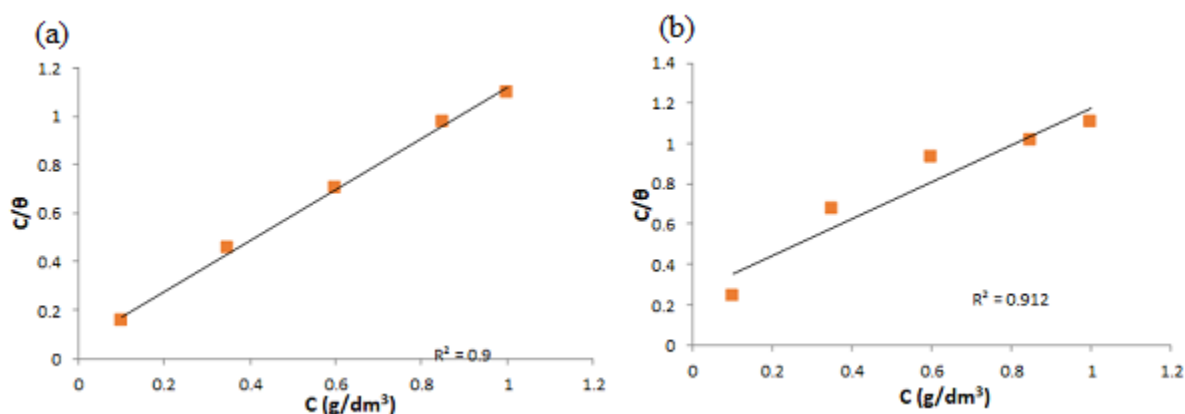


Fig. 3 Plot C/θ against Concentration C (g/dm³) showing agreement with Langmuir isotherm for Galvanised mildsteel corrosion in synergized plant extracts in the presence of 1.0 M HCl (a) at 30 °C (b) at 50 °C.

3.6 Surface morphological examinations

SEM image show the microstructural arrangement of the constituent of Zinc coat as it covers the surface of the steel as received newly in Fig. 4a. Fig. 4b is the EDX spectrum with high value of 93.31 % of Zn which shows the freshness of the material without any attack or inhibitory influence. The effect of the acid solution on the zinc coat in the absence of extract revealing depreciation of zinc coat is as shown in Fig. 4c. The surface of zinc metal is covered with patches of corrosion products. It has made much of the α -Zinc phase dissolve into the acid solution. Fig. 4d depicts the EDX spectrum revealed that α -Zn peak is prominent. A low value of Zinc at other regions of the material surface revealed the high loss of the material to the solution from that area coupled with high 59.24 % carbon content as a corrosion product. Fig. 4e shows that the SEM image is covered by the adsorbed extracts as it protected galvanised steel with mild depletion of material surface covered with oxide films. The effect of corrosion of the metal was very feasible. Fig. 4f EDX spectrum shows prominent Zinc peak as the major followed by carbon peak from the extract adsorbed on the steel in the presence of other impurities peaks such as aluminium, silicon and sulphur. Also, the presence of 29.64% of carbon and 6.23% oxygen as products of corrosion.



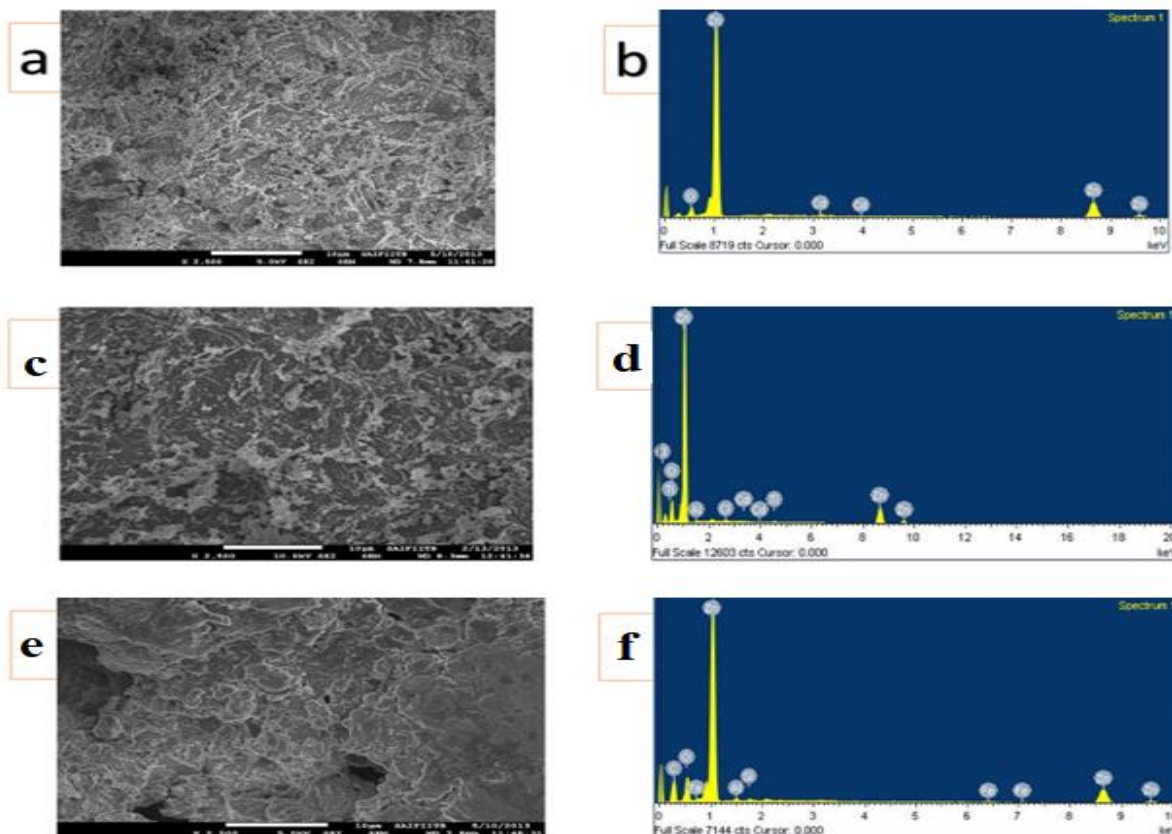


Fig. 4: SEM images of galvanised pipe surface (a) as received (b) as received (c) in HCl acid (d) in HCl acid (e) as protected with extract and EDX spectrum of galvanised mildsteel surface (f) as protected with extract

4. Conclusion

The morphological studies show that the galvanised surface was inhibited against corrosion by the synergised plant extract and thus reduced the rate of preferential discharge. The adsorption of the extract was best favoured by Langmuir isotherm. This observation is further confirmed through the studies of the half-life of studies. Zinc coated steel gave 4.53×10^{11} mins, while effective enhancement of Zinc coated with plant extract steel showed a half-life of 7.51×10^{12} mins. The enhancement of steel corrosion inhibition through the protection of zinc coat on the steel was achievable as the extract created an inhibition barrier for the corrosion process for the zinc coat on the steel. The plant extracts inhibited corrosion of the zinc coat on steel and thus elongated its life span of preferential corrosion for steel protection.

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