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Inhibition of Corrosion of Galvanized Steel Sheet in 1M HCl and H2SO4 by Plukenetia conophora Leaf Extract

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ABSTRACT

The inhibiting effect of Plukenetia conophora leaf extract on the corrosion of galvanized sheet in 1.0M H_2SO_4 and HCl solutions at ambient temperature was investigated in this study using gravimetric and gasometric methods. The corrosion rates of the galvanized sheets decreased with increased concentration of the inhibitors and exposure time. The study also revealed that as the concentration of Plukenetia conophora leaf extract increased, the inhibition efficiency increased by 48% in HCl and 56% in H_2SO_4 after 10 hours exposure during gravimetric and 5 mins exposure during gasometric measurement. The volume of the hydrogen gas evolved reduced with increased exposure time during the gasometric method. The presence of the extract slowed down the corrosion rate and the rate at which the hydrogen gas evolved. The formation of adsorption layer on the surface of the metal reduced the rate at which hydrogen gas was evolved, which was a function of the increment in concentration of the extract. It was concluded that Plukeneia conophora leaf extract could be used as a green inhibitor to slow down corrosion of galvanized sheet in the acidic media.

1. Introduction

Galvanized steel is most commonly used in applications such as building roofing, automotive parts and water pipeline systems owing to its good resistance to corrosion (El-Sayed et al., 2012; Yadava et al., 2007; Ajayi et al., 2011; Rosaline et al., 2011). Corrosion resistance of galvanized sheet is largely dependent on the protection obtained from the zinc coating technique (Shyamala and Kasthuri, 2011; Kartsonakis et al., 2012). However, many cases of failure of galvanized pipes and tanks have been found to be due to the aggressive nature of their application environments, leading to severe damage to galvanized steel sheets resulting from formation of rust (Zhang, 2005).

In most rural areas in developing countries, the predominantly used roofing material is corrugated galvanized steel, which are sheets of hot-dip galvanized mild steel, cold-rolled to produce a linear corrugated pattern. The sheet is coated with a thin zinc layer, which is formed by passing the metal through a molten bath of zinc at about 460°C. In an unpolluted aqueous solution, ZnO and $Zn(OH)_2$ are the most abundant corrosion products (Mokaddem et al., 2010; Vu et al., 2012). When exposed to the atmosphere, the pure zinc reacts with oxygen to form zinc oxide, which further reacts with carbon dioxide to form zinc carbonate ($ZnCO_3$), a usually dull grey, fairly strong material which stops further corrosion in many circumstances by protecting the substrate steel material (American Galvanizers Association, 2011).

Recently, researchers are showing more interest on the use of natural or green inhibitors, which are more environmentally friendly and non-toxic compared with synthetic inhibitors. Plant parts extracts such as the root, stem, bark and leaves have been investigated as potential replacement for synthetic inhibitors in many applications (Ajayi et al., 2011; Rosaline et al., 2011). These plant extracts include *Moringa oleifera* (Odusote et al., 2016), *Chromolaena odorata* (Ajayi et al., 2011), *Cassia auriculata* (Rosaline et al., 2011), *Ocimum Santum*, *Aegle marmelus* and Solanum trilobatum (Shyamala et al., 2011; Oguzie, 2008), Vernonia amygdalina (Ndibe et al., 2011) and Aloe vera (Al-Turkustani et al., 2010). In previous studies, the inhibition effects of Jatropha curcas leaf extract as a cheap, non-toxic and biodegradable corrosion inhibitor on mild steel in different media were investigated (Odusote and Ajayi, 2016, Ajayi et al., 2014; Odusote and Ajayi, 2013).

The aim of this study was to produce corrosion inhibitor from Plukenetia conophora (Walnut) leaf extracts and determine their effectiveness and inhibition efficiencies on galvanized steel sheet in concentrated tetraoxosulphate (VI) acid and hydrogen chloride acid solutions. Plukenetia conophora seed have been reported to possess good inhibiting properties on varieties of metals (Ayoola et al., 2011). However, little or no literature has been found on the use of the leaves extract as corrosion inhibitor in different media.

Table 1: Chemical composition of the galvanized sheet sample (wt%)

Elements	Fe	Zn	Cu	Al	Sn	Mn	Cr	Others
Weight (%)	99.40	0.3210	0.0100	0.0330	0.0010	0.2520	0.0018	0.2073

2. Experimental procedure

2.1 Specimen preparation

Rectangular specimens of galvanized sheet were mechanically press cut into 0.05 x 3cm^2 coupons. The specimens were metallographically prepared, after which they were degreased in ethanol and cleaned in acetone. The specimens were then allowed to dry and were kept in a desiccator. The composition of the galvanized sheet samples was analyzed using Optical Emission Spectrometer (OES) and the result is presented in Table 1.

2.2 Preparation of the Plant Extract

The fresh green leaves of Plukenetia conophora were collected locally, washed thoroughly with distilled water and air dried for five days. The dried leaves were pulverized using manual grinding machine. About 500 g of the dried leaves was soaked in a sufficient quantity of 99% ethanol in 1000 ml round bottom flask in order to cover the powder completely. The mixture was then refluxed together properly in order to agitate the constituent of the Plukenetia conophora to dissolve into the ethanol. The flask was then covered and left in a cool dried cupboard for 48 h. The contents of the flask were filtered using a filter paper, and the filtrate was concentrated using a water bath at 70°C to vaporize the ethanol. The concentrate was used to prepare the different concentrations of the plant extract needed (0.2, 0.4, 0.6, 0.8 g/l) with distilled water. These concentrations were kept in air-tight plastic containers to avoid contamination.

2.3 The phytochemical screening of the leaves

The phytochemical screening of the *Plukenetia conophora* leaves extract was carried out and the result is shown in Table 2. The result showed that the extract contained alkaloid, cardiac glycosides, saponins, tannins, anthraquinones, flavonoids, terpenoid, anthraquinones, oxalate, phenol and trypsin. These constituents have been reported to have good inhibiting effects on metals in several media (Ayoola *et al.,* 2011).

2.4. Weight Loss Method

The specimens were metallographically prepared, degreased in ethanol, washed in acetone, stored in desiccators and weighed using an electronic balance prior to immersion in 50 ml of 1.0M HCl and H_2SO_4 with different concentrations (ranges between 0.2 g/l - 0.8 g/l) of the leaf extracts. The control experiment was without any leaf extract. From the weight loss, the corrosion rate, inhibition efficiency (%IE) and surface coverage (θ) of the plant extract were calculated and reported for HCl and H_2SO_4 respectively, using the Equations 1-3 (Ayoola *et al.*, 2011; Eddy, 2009):

$$\text{Corrosion rate } (gcm^{-2} h^{-1}) = (W/DAT) \qquad (1)$$

Where W is the weight loss (g), D is the density of the sample in gcm^{-3}, A is the surface area of the galvanized sheet coupon in cm^2, and T is the time of exposure (hours).

Table 2: Phytochemical screening of the Plukenetia conophora leaves extract

Constituent	Alkaloids	Cardiac glycosides	Saponin	Tannins	Anthraquinones	Flavonoids	Terpenoids	Anthraquinone	Phenols
Bioassay	+++	+	++	+	+	+	+	+	++

The benchmark sample D was in operation for 3 years and shows little evidence of extensive damage on the surfaces close to the vane entries as seen in the previous samples. The protective oxide film is evident with a typical brown colouration dominating the surface colour. The extent of pitting seen on the surface is acceptable for an impeller operated over such a long period. Thus if operated within optimised conditions the impeller shows a greater chance of survival. In such a case the life cycle can be prolonged by improving the quality of the protective coating.

$$\text{I.E. } (\%) = \left(\frac{CR_B - CR_W}{CR_B}\right) X100 \qquad (2)$$

$$\text{Surface Coverage } (\theta) = \left(\frac{CR_B - CR_W}{CR_B}\right) \qquad (3)$$

Where CR_B and CR_W are corrosion rates in the absence and presence of the inhibitor.

2.5. Gasometric Method

The gasometric assembly used for the measurement of hydrogen evolution followed the procedure reported by Ajayi *et al.,* 2014. A reaction vessel was connected to burette through a delivery tube. 1.0 M HCl and 1.0 M H_2SO_4 solutions were separately introduced into the mylius cell, and the initial volume of air in the burette was recorded. The prepared galvanized sheets were dropped into the HCl and H_2SO_4 solution and the mylius cell was quickly closed. The volume of hydrogen gas evolved from the corrosion reaction was monitored by the volume change in the level of water in the burette. The change in volume was recorded every 30 s for up to 5 minutes in the acid solutions without inhibitor. Similar procedures were repeated with the acid solutions with inhibitors. The inhibition efficiency and the degree of surface coverage were determined using Equations 4 and 5 (Inzunza *et al.*, 2012).

$$\text{I.E. } (\%) = \left(\frac{V_{H0} - V_{H1}}{V_{H0}}\right) X100 \qquad (4)$$

$$\text{Surface coverage } (\theta) = \left(\frac{V_{H0} - V_{H1}}{V_{H0}}\right) \qquad (5)$$

Where V_{H0} and V_{H1} are volume of hydrogen gas evolved in the absence and presence of the inhibitors respectively.

3.0 Results and Discussion

3.1 Gravimetric Data

The curves for the variation of weight loss with exposure time for galvanized sheet specimen immersed in 1.0M H_2SO_4 and HCl solutions with varied concentrations of *Plukenetia conophora* leaf extract are presented in Figures 1 and 2. It was found from the figures that the weight loss of galvanized sheets increased with the period of exposure, but decreased as the concentration of the inhibitor increased. These were more significant differences from the control samples compared to those immersed in inhibitors. Figures 3 and 4 also indicate that the rate of corrosion of galvanized sheet decreased with increased concentration of the inhibitor and the period of exposure, possibly because of the fast rate at which hydrogen gas evolved when in the acidic environment, especially in HCl.

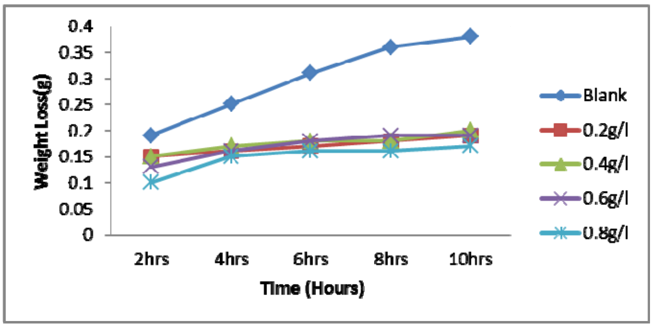

Figure 1: Variation of weight loss with time for corrosion of galvanized sheets in 1.0M H_2SO_4 using *plukenetia conophora* as inhibitor.

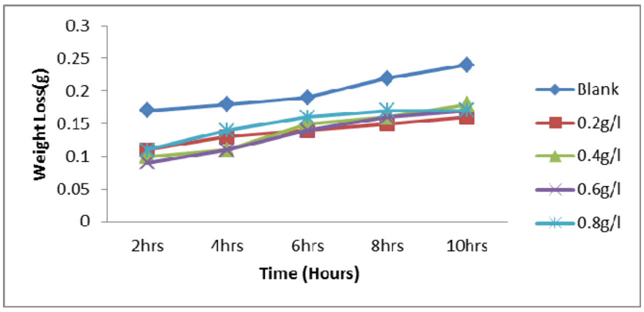

Figure 2: Variation of weight loss with time for corrosion of galvanized sheets in 1.0M HCl using *Plukenetia conophora* as an inhibitor.

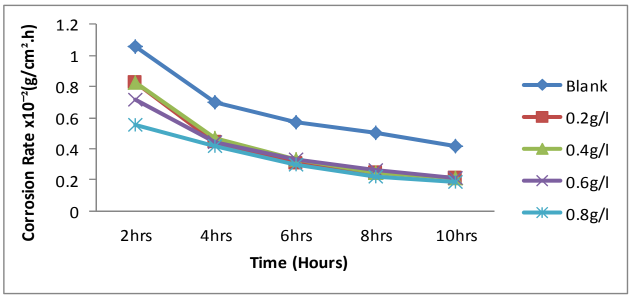

Figure 3: Variation in corrosion rate against time of immersion of galvanized sheet in 1.0M H_2SO_4 using *Plukenetia conophora* as an inhibitor.

This indicates that the rate of corrosion is dependent on the environment and the concentration of inhibitor. Hence, *Plukenetia conophora* leaf extract inhibited the corrosion of galvanized sheet in the acidic solutions. Furthermore, with increased concentration of the inhibitor, the surface coverage increased and the adsorption of the phytochemical constituents on the metal surface, thereby creating a barrier between the metal and the acidic media. This enhances the inhibition of the metal and resulted in less mass loss than the uninhibited metal.

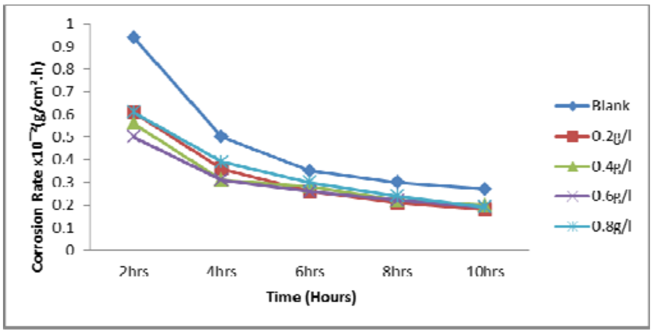

Figure 4: Variation in corrosion rate against time of immersion of the extracts in 1.0M HCl using *Plukenetia conophora* as an inhibitor.

Tables 3 and 4 show that HCl was more aggressive than H_2SO_4 on galvanized sheet, while *Plukenetia conophora* leaf extract displayed good inhibiting properties in both media. This inhibiting property may possibly be due to the large amount of some active constituents such as alkaloids, saponin and phenols in the leaves extract as reported by (Ekpe *et al.,* 1994; Ebenso and Ekpe, 1996; Lebrini *et al.,* 2011; Odusote and Ajayi, 2013; Ajayi *et al.*, 2014, Alaneme *et al.*, 2015 and Odusote and Ajayi, 2016). In addition, the decrease in weight loss of metal immersed in solutions with inhibitors may also be due to the adsorption of the inhibitors on the surface of the metal, which form a passive layer that inhibits the corrosion of metals (Oguzie, 2008).

Table 3: Inhibition efficiency of galvanized sheet in different concentrations of *Plukenetia conophora* leaf extract in 1.0M H_2SO_4 at different immersion time.

Time (hours)	Inhibition Efficiency of *Plukenetia conophora* Leaf extract in H_2SO_4 Solution (%)				
	Blank	**0.2g/l**	**4g/l**	**6g/l**	**8g/l**
2	-	21.70	31.70	32.10	38.10
4	-	37.10	32.90	37.10	40.00
6	-	45.60	42.10	42.10	47.40
8	-	50.00	50.00	48.00	56.80
10	-	50.00	50.00	50.00	54.80

Table 4: Inhibition efficiency of galvanized sheet in different concentrations of *Plukenetia conophora* leaf extract in 1.0M HCl at different immersion times.

Time (hours)	Inhibition Efficiency of *Plukenetia Conophora* Leaf extract in HCl solution (%)				
	Blank	**2g/l**	**4g/l**	**6g/l**	**8g/l**
2	-	53.10	40.40	46.80	35.10
4	-	8.00	38.00	38.00	22.00
6	-	25.70	20.00	25.70	14.30
8	-	30.00	26.70	26.70	20.00
10	-	33.3	25.90	29.60	29.60

3.2. Gasometric Data

The variation of inhibition efficiency against time of immersion with varying concentrations of the inhibitor is shown in Figures 5 and 6. The results showed that the volume of the hydrogen gas evolved increased with time, but decreases as the concentration of extract increases (Odusote and Ajayi, 2013). This indicates that the rate of dissolution of the galvanized steel was retarded, leading to reduction of H^+ liberation. The higher volume of hydrogen gas evolved in the control solution suggested that there was no adsorption layer to inhibit the reaction of the acid on the surface of the galvanized sheet according to Nnanna and Owate, (2014) and Omotosho *et al.*, (2012). This clearly explained the failure evaluation of zinc in 2.0M HCl solution in the presence of *Bambusa bambos* leaf extracts using gasometric method after the volume of hydrogen gas evolved was recorded. They stated that the corrosion rate had no particular trend, possibly because of the rate at which hydrogen attacked zinc, due to their relative positions in the electrochemical series (zinc being far below hydrogen).

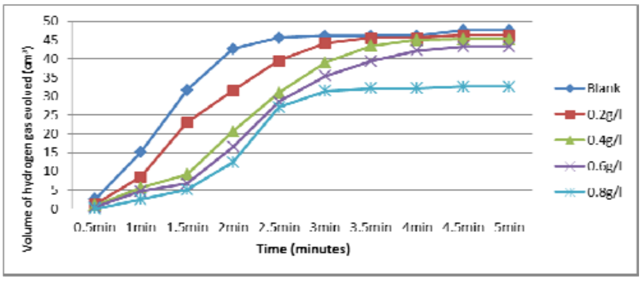

Figure 5: Variation of volume of hydrogen gas (cm^3) evolved with time (minutes) of galvanized sheet coupons for different volumes of *Plukenetia conophora* leaf extract in H_2SO_4 solution.

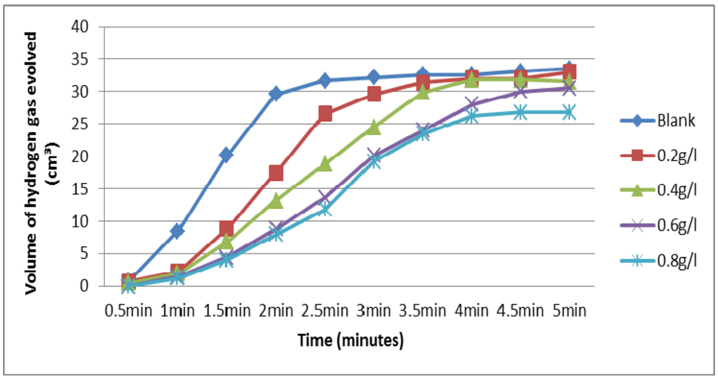

Figure 6: Variation of volume of hydrogen gas (cm3) evolved with time (minutes) of galvanized sheet coupons for different volumes of *Plukenetia conophora* leaf extract in HCl solution.

Tables 5 and 6, show that there were re-ordering of the inhibition efficiencies from highest to the least value in descending order of the inhibitor concentration i.e. (8g/l<6g/l<4g/l<2g/l) at all the exposure time. This revealed that there is an adsorption of the constituents of the leave extracts on the surface of galvanized sheet with 8g/l concentration of the inhibitor having the highest inhibition efficiency. Omotosho *et al.*, (2012) investigated the use of *Bambusa bambos* leaf extract as safe green inhibitor of zinc corrosion in 2.0M HCl solution, and found that as the concentration of the extract increased, the inhibition efficiency increased possibly as a result of the adsorption layer formed on the surface of zinc metal which inhibited the rate of corrosion. In addition, the inhibitive property of leaves extracts of Ananas sativum (Umoren *et al.,* 2010), Aloe vera (Abiola and James, 2010) and Pactin from citrus peel (Mohammad et al.,2012) were reported to retard the acid (HCl) induced corrosion on each of the materials involved, leading to increase in inhibition efficiency with increased concentration of the extract. This agrees with the observation in this study. Furthermore, it could also be drawn from their results that a strong barrier was formed on the surface of the materials, which has inhibited the penetration of the active agents present in the acid solution. The observation could also be attributed to the fact that the rate of chemical reaction increases with increasing acid concentration and/or probably due to increase in the rate of diffusion and ionization of active species in the corrosion reaction as reported by Omo-dudu and Oforka (1999).

Table 5: Inhibition efficiency of galvanized sheet in different concentrations of *Plukenetia conophora* leaf extract in 1.0M H_2SO_4 at different immersion times.

Time (hours)	Inhibition Efficiency of *Plukenetia Conophora* Leaf extract in H_2SO_4 solution (%)				
	Blank	**2g/l**	**4g/l**	**6g/l**	**8g/l**
0.5	-	53.80	65.40	80.80	100
1	-	43.70	62.90	68.90	83.40
1.5	-	27.20	70.90	78.80	83.40
2	-	26.10	51.60	57.20	70.90
2.5	-	13.10	32.20	37.50	40.60
3	-	4.12	15.40	23.20	31.90
3.5	-	1.08	5.60	14.80	30.40
4	-	1.08	2.40	8.50	30.40
4.5	-	2.70	5.00	9.20	31.50
5	-	2.70	5.00	9.20	31.50

Table 6: Inhibition efficiency of galvanized sheet in different concentrations of *Plukenetia conophora* leaf extract in 1.0M HCl at different immersion times.

Time (hours)	Inhibition Efficiency of *Plukenetia conophora* leaf extract in HCl solution (%)				
	Blank	**2g/l**	**4g/l**	**6g/l**	**8g/l**
0.5	-	12.50	37.50	100.00	100.00
1	-	73.80	77.40	83.30	85.70
1.5	-	56.20	66.20	78.10	80.10
2	-	40.90	55.40	70.30	73.30
2.5	-	16.10	40.70	57.10	63.80
3	-	8.10	23.90	37.60	40.40
3.5	-	3.60	8.30	26.40	40.40
4	-	1.80	2.50	14.10	19.60
4.5	-	1.30	3.92	9.40	19.60
5	-	1.50	5.00	8.90	20.00

4. Conclusion

Based on the results obtained, the following conclusions can be drawn:

(1) *Plukenetia conophora* leaf extract, which is non-toxic, cost effective and eco-friendly, can act as an efficient corrosion inhibitor on galvanized sheet in 1.0M HCl and H_2SO_4 solution.

(2) Hydrochloric acid environment is more aggressive than sulphuric acid environment probably because of the present of chloride ion (Cl^-) which is more active than the sulphuric ion ($SO4^{2-}$) relating to electronegativity value from the electrochemical series.

(3) The inhibition efficiency increased with increased concentration of the extract of *Plukenetia conophora* leaves but decreased with time indicating that the longer time the inhibitor spent in the environment the less reactive it becomes.

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