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AN INTERESTING AND EFFICIENT GREEN CORROSION INHIBITOR FOR ALUMINIUM FROM EXTRACTS OF *Moringa oleifera* IN ACIDIC SOLUTION

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ABSTRACT
The leaf extracts of Moringa oleifera has been studied as a possible source of green inhibitor for corrosion of aluminium alloy in 0.5M HCl using gravimetric and thermometric techniques at 30 and 60 °C. Results obtained showed that Moringa oleifera functioned as an excellent corrosion inhibitor for aluminium in the acidic environment. Inhibition efficiency increased with extract concentration but decreased with temperature. The adsorption of Moringa oleifera on Al surface is in accord with Langmuir adsorption isotherm. Both kinetic and thermodynamic parameters governing the adsorption process were calculated and discussed. Results show that Moringa oleifera which is biodegradable, environmentally benign, and are obtained from a renewable resource with minimal health and safety concerns has the potential to be a cost effective alternative to synthetic corrosion inhibitors. This present study provides new information on the inhibiting characteristics of Moringa oleifera extract under specified conditions. The environmentally friendly inhibitor could find possible applications in metal surface anodizing and surface coating in industries.

Keywords: *Moringa oleifera*, Aluminium, Hydrochloric acid, Langmuir isotherm, Plant extracts, Corrosion inhibition.

INTRODUCTION
Corrosion inhibitors are compounds that are added in small quantities to an environment to prevent corrosion of metals (Sharma et al., 2008). Most of the efficient acid inhibitors are organic compounds containing nitrogen, sulphur and/or oxygen atoms in their molecule (Obot and Obi-Egbedi, 2008; Obot et al., 2009). The known hazardous effects of synthetic organic inhibitors, which have been in use (Popova et al., 2007; Li, et al., 2009) and the need to develop cheap, non-toxic and ecofriendly processes have now made researchers to focus on the use of natural product (Umoren et al., 2008d; Umoren & Ebenso, 2008; El-Etre, 2008).

Recent literatures have shown that naturally occurring materials such as Raphia hookeri (Umoren et al., 2009), Ipomoea invulcrata (Obot & Obi-Egbedi, 2009a), Vigna unguiculata (Umoren et al.,2008a), Pachylobus edulis (Umoren et al., 2008c), Ginseng root (Obot and Obi-Egbedi, 2009b), Dacroydes edulis (Umoren et al., 2008b), Zenthoxylum alatum (Chauhan and Gunasekaran, 2007), Hibiscus sabdariffa (Noor, 2009), limonene (Chaieb et al.,2009), bamboo leaf (Li, et al., 2012), Neolamarckia cadamba (Raja, et al., 2013) to mention but a few have been found to be very efficient corrosion inhibitors for metal in acidic/alkaline media. Moreover, extensive work has been done in both acidic and alkaline media using *Euphorbia hirta* and *Dialum guinnense* leave extracts as inhibitors (Nnanna et al., 2011; Nnanna et al., 2012, Mejeha, et al., 2012). The inhibitive performance could be attributed to adsorption of flavonoids on aluminium surface. A detailed review of natural products as corrosion inhibitors can be found in (Raja and Sethuraman, 2008). *Moringa oleifera* is also known as drumstick tree. Although *M. oleifera* is native to the sub-Himalayan tracts of India, Pakistan, Bangladesh and Afghanistan where it is used in folk medicine, it is now widely distributed all over the world (Lim, 2012). All parts of the tree-leaves, tender young capsules (pods), immature seeds, flowers, fruits and young roots are edible (Lim, 2012). The leaves can be eaten fresh, cooked, or stored as dried powder for many months without refrigeration, and reportedly without loss of nutritional value. The leaves are considered to offer great potential for those who are nutritionally at risk and may be regarded as a protein and calcium supplement. It is a general belief that the stage of maturity of plant affects the concentration of nutrients of leaves (Bamishaye, et al., 2011). Phytochemical screening reports have shown that the leaves contain phenolics, tannins, alkaloids, saponins, flavonoids and steroids (Kasolo, 2010; Bamishaye, et al., 2011) which are very important in inhibition of corrosion of metals.

The present work therefore, has been designed to evaluate the effect of the leaf extracts of *Moringa oleifera* on the corrosion inhibition of aluminium in 0.5M HCl solution with a view to contributing to the search for further beneficial uses of plant extract. Gravimetric technique was used at 30^{o}C for the investigation. Activation and thermodynamic parameters were effectively used to characterize the inhibition mechanism of the adsorption process.

MATERIALS AND METHODS

Materials preparation

The sheets of aluminium alloy AA8011 used for this study were obtained from First Aluminium Plc, Port Harcourt, Nigeria. Each sheet was 1.32 mm in thickness and were mechanically pressed cut into 2 cm $\times$ 2 cm coupons. These coupons were used as cut without further polishing. However, they were degreased in ethanol, dried in acetone and stored in moisture free desiccators before their use in corrosion studies. The weight percentage composition of the aluminium alloy is: Si-0.240%, Fe-0.241%, Cu-0.035%, Mn-0.102%, Ti-0.019%, Pb-0.014%, Zn-0.043% and the remainder being Al were used.

Preparation of the leaf extracts of *Moringa oleifera*

The procedure for the preparation of the leaf extracts is similar to that reported recently by Okafor et al (2008). *Moringa oleifera* leaves were collected from Abiriba, Abia State, Nigeria. They were dried in an N53C-Genlab Laboratory oven at 50^{o}C, and ground to powder form. Ten gram of the powder was digested in 1L of 0.5M HCl solution. The resultant solution was kept for 24hr, filtered and stored. From the stock solution, the leaf extracts test solutions were prepared at concentration range of 0.1 – 0.4g/L using excess acid as solvent at room temperature.

Scientia Africana, Vol. 13 (No.1), June 2014. Pp 52-63
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Gravimetric experiment

The cleaned and dried specimens were weighed before immersion into the respective test solutions of 0.5M HCl using JA1003A electronic weighing balance with the accuracy of ± 0.005. Tests were conducted with different concentrations of inhibitor. At the end of the tests, the specimens were carefully washed in absolute ethanol having used nitric acid to quench further corrosion from taking place, and then reweighed. Triplicate experiments were performed in each case and the mean values reported.

RESULTS AND DISCUSSION

Fourier Transform Infrared Spectroscopy (FTIR)

The Fourier Transform Infrared Spectroscopy (FTIR), was employed to determine the functional groups present in the organic compound of the inhibitor, *Moringa oleifera*. MO was subjected to IR radiation, the energy absorbed results in molecular vibrations in bonds between atoms in the molecule. The bond between two atoms are likened to springs that have stretching and bending properties, while the atoms are regarded as tiny masses attached to both ends of the spring. It is also important to note that for IR absorption to take place, the bond vibration must result in a change in the dipole moment of the bond. If the stretching or bending vibration of a bond results in a large change in its dipole moment, the bond will show strong absorption bands in the IR spectrum. If the change in dipole moment is not significant, a weak absorption band will result.

The absorption of IR radiation results in the elevation of the molecule to a higher vibrational energy level. 3405 cm^{-1} signifies H - C - H asymmetric and symmetric stretch which belong to the functional group alkenes (alcohol and phenols) while 2897 cm^{-1} is the C – H stretch of alkanes. The peak value of 1729 cm^{-1} is the C = O stretch which belongs to the functional group of aldehydes and saturated aliphatic and the vibration is in conjugation with oxygen in the R portion shifts the absorption to higher frequencies. 1250 cm^{-1} is the C – N stretch of aliphatic amines. From the interpretation of the IR result, the absorption rate of *Moringa oleifera* as an inhibitor is quite high with the presence of the C = O functional group.

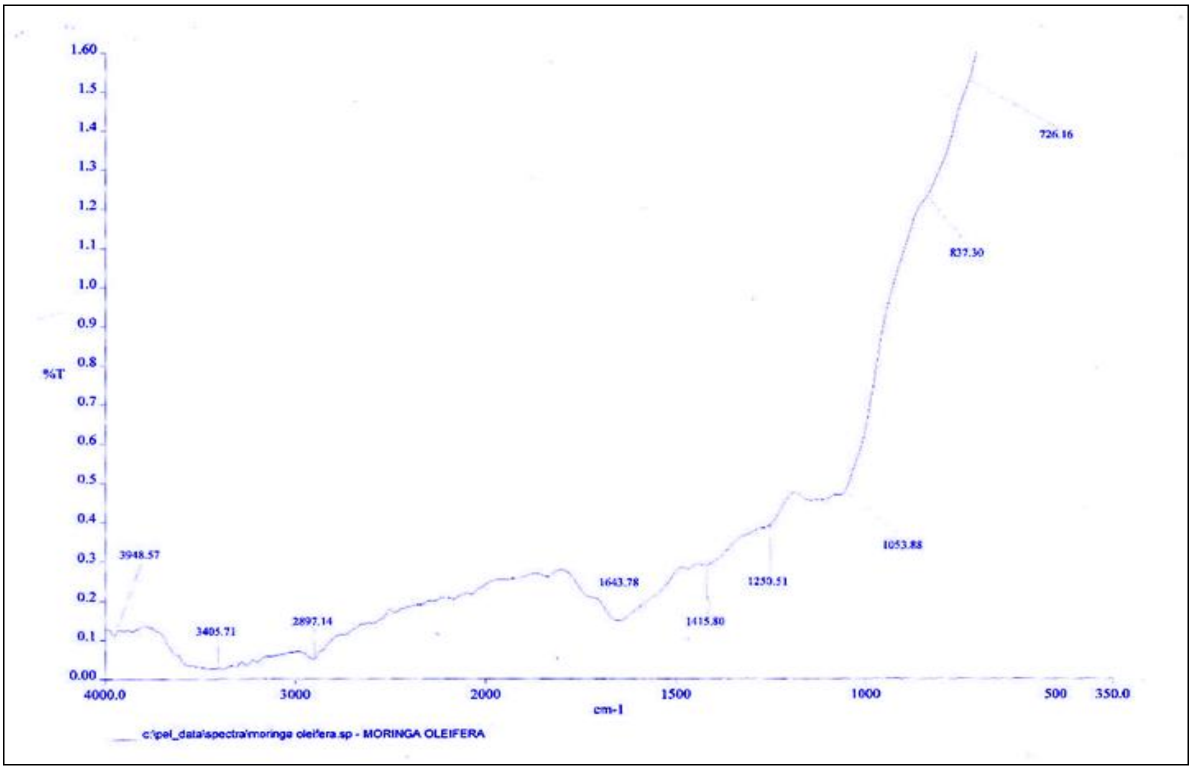

Figure 1. FTIR data of *Moringa oleifera*leaves.

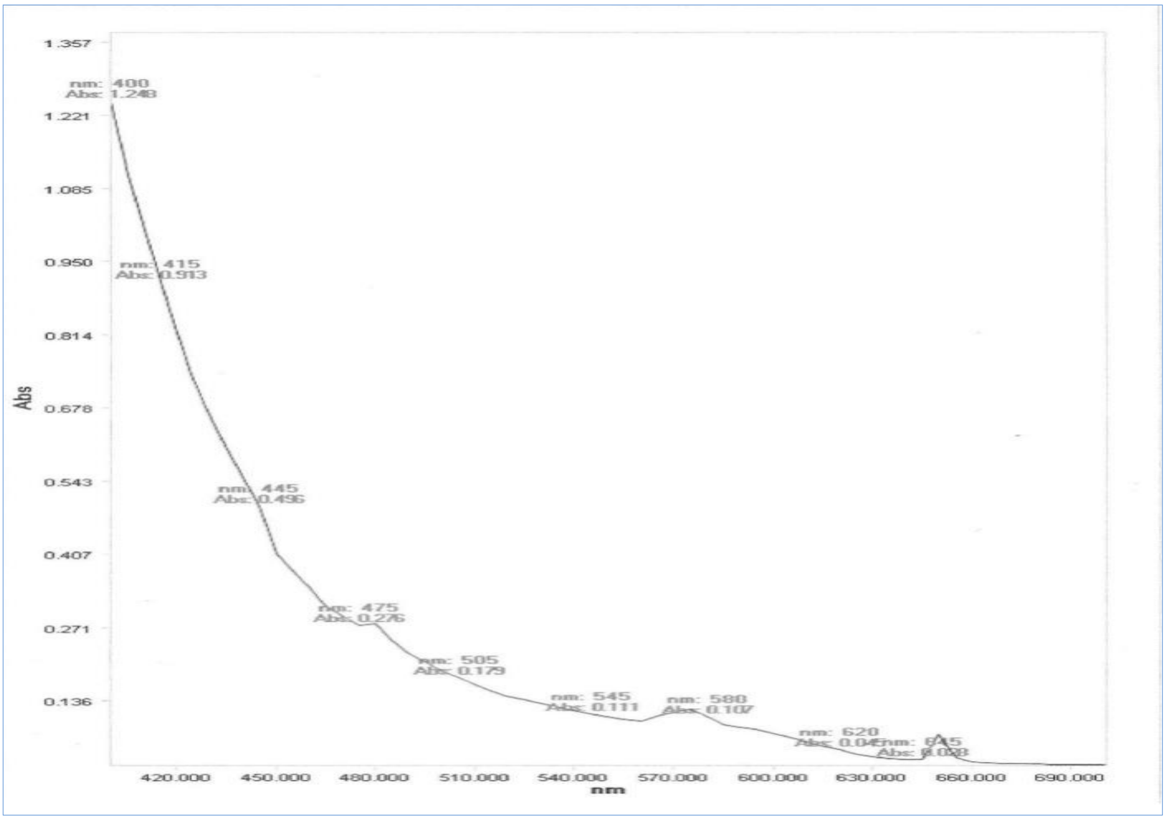

Figure 2. Ultra-violet Spectroscopy of *Moringa oleifera* leaves.

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Ultraviolet (UV) Spectroscopy

Ultraviolet and visible regions of electromagnetic spectrum involve light radiation of wavelength between 200 and 800 nm, with energy ranging from about 159-419 $kJmol^{-1}$ capable of causing the excitation of electrons from their ground state in a molecular orbital to an excited state. Absorption of radiation in the UV region is made possible by the presence of loosely bound electrons such as are found in non-bonding and π-molecular orbitals. Excitation of an electron on absorption of UV radiation occurs between the highest occupied molecular orbital (HOMO) of the ground state and the lowest unoccupied molecular orbital (LUMO) of the excited state. The analysis of the UV characteristic of *Moringa oleifera* shows that there was a promotion of an electron from a non-bonding (n) or a π-bonding molecular orbital to a much lower unoccupied π^* anti-bonding molecular orbital.

Gravimetric technique and corrosion rates

The corrosion rates of the aluminium alloy AA8011 in 0.5M HCl solutions in the absence and presence of *Moringa oleifera* leaf extract were determined at room temperature. Figure 1 shows the variation of the corrosion rates of the aluminium in 0.5M HCl with inhibitor concentration after 7 hours. It can be seen from Figure 1 that the corrosion rate decreases with increase in the concentration of the inhibitor. That clearly shows that the leaf extract retards the corrosion rate of the aluminium in the test solution. The equation for corrosion rate is given by

$$C = \frac{K\Delta W}{\rho A t} \quad (1)$$

where C is the corrosion rate, ΔW is the weight loss in mg ; ρ is the density of the Al (g/cm^3); A is the exposed area of the coupon (in^2; 1 in^2 = 6.5146 cm^2); t is the immersion time (h); K is the rate constant (534 mpy; mils per year; 1 mil = 10^{-3} in).

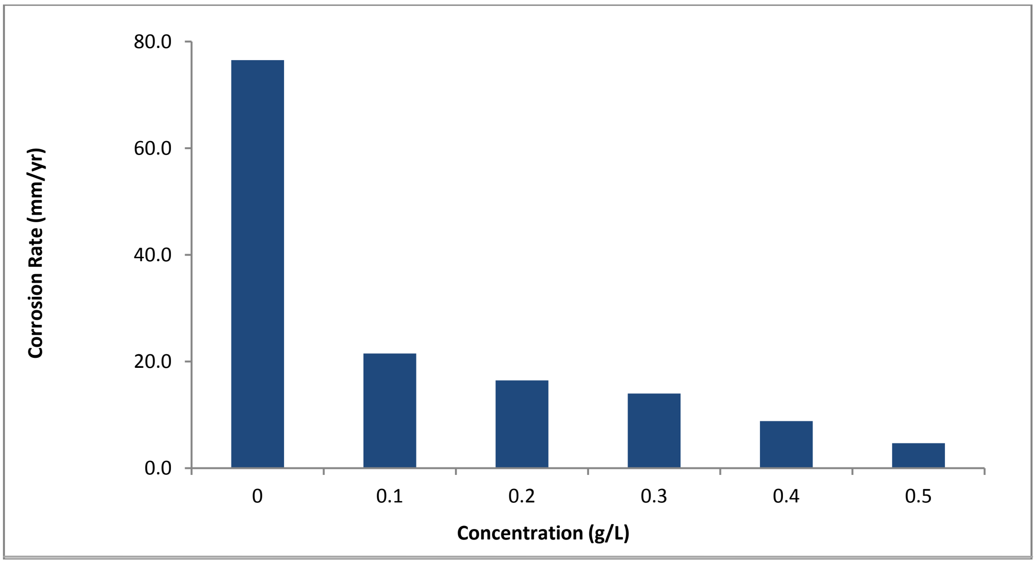

Figure 3. Variation of corrosion rate of aluminium in 0.5M HCl with different concentrations of *Moringa oleifera* extract.

Figure 2 illustrates the variation of the inhibition efficiencies of the inhibitor in 0.5M HCl on aluminium for an exposure time of 7hours. The inhibition efficiency of the *Moringa oleifera* leaf extract on the corrosion of the aluminium in and 0.5M HCl containing different concentrations of the leaf extract was computed by using the relation (Oguzie et al, 2008).

$$I\% = \left(-{}^{\rho_{inh}}/_{\rho_{blank}}\right) \qquad (2)$$

Where I% represents the inhibition efficiency expressed in percentage, ρ_{inh} is the corrosion rate in the presence of the inhibitor while ρ_{blank} is the corrosion rate in the absence of inhibitor. Optimum value of about 94% at 0.5g/L 0.5M HCl for concentration of *Moringa oleifera* extract was obtained as shown in Figure 2 below. The results show that *Moringa oleifera* leaf extract is a good inhibitor.

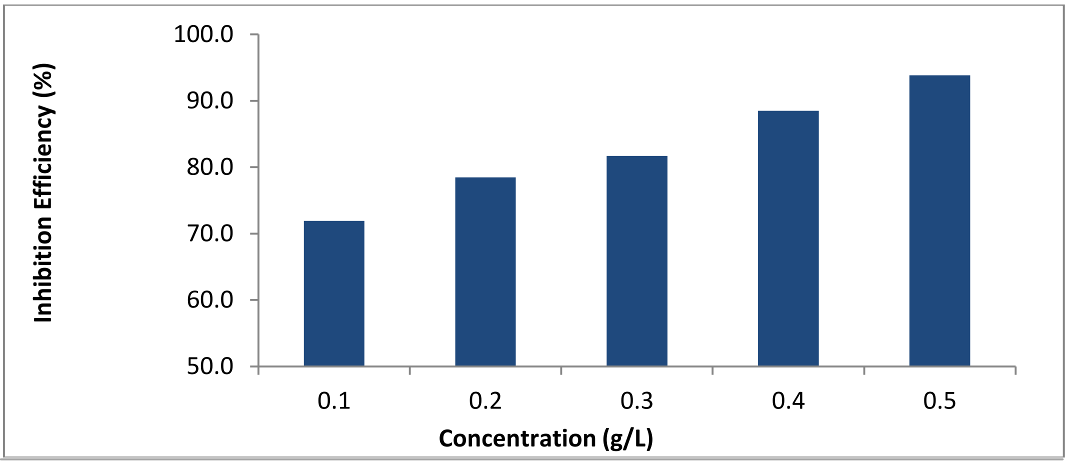

Figure4. Variation of inhibition efficiency with concentration of *Moringa oleifera* leaf extract for aluminium in 0.5M HCl

Inhibition efficiency

Clearly, the inhibition efficiency of *Moringa oleifera* leaf extract increases with exposure time for the inhibitor concentration considered. This result is consistent with the findings of (Nnanna et al., 2011) who showed that the inhibition efficiency of *Euphorbia hirta* in the corrosion of the aluminium alloys AA8011 in 0.5M HCl increases with exposure time. As shown in Figure 2, the inhibition efficiency increases with increase in inhibitor concentration, tending to saturate at higher values of inhibitor concentration. This indicates that the effectiveness of the *Moringa oleifera* leaf extract in retarding the corrosion rate of aluminium in the test solution improves significantly with increase in inhibitor concentration and hour of exposure.

The result that the inhibition efficiency of *Moringa oleifera* leaf extracts increases with increase in inhibitor concentration suggests that some of the molecules of the inhibitor are adsorbed on the metal surface thereby protecting the "covered" surface from further corrodent attack. Increasing the inhibitor concentration increases the degree of surface coverage, θ, of the metal surface defined as:

$$\theta = 1 - \frac{\rho_{inh}}{\rho_{blank}} \qquad (3)$$

The *Moringa oleifera* leaf extract consists of a mixture of complex organic components

Scientia Africana, Vol. 13 (No.1), June 2014. Pp 52-63
 ISSN 1118 - 1931

including flavonoids, several phenolic compounds (β-carotene-linoleic acids) and some other organic compounds. The plant extract also contains some nutritional minerals including potassium, magnesium, copper and zinc (Popova et al, 2007).

Plant/leaf extracts that have proven corrosion inhibiting abilities in corrosive media are known to contain one or more of the following organic substances, namely tannins, triterpenoids, flavonoids, amino acids, alkaloids, saponins, phenols, glycosides, essential oils, carotenoids, β-carotene, ascorbic acid, crude proteins among others (Noor, 2009; Popova et al, 2007). Moreover, the presence of some metallic ions particularly Mg^{2+} may enhance the corrosion inhibiting action of the extract. This is because it has been observed that some inorganic ions particularly Ca^{2+}, Mg^{2+} and Zn^{2+} ions synergistically increase the inhibition efficiency of organic substances (Sharma et al, 2008). Nevertheless, it is not possible at this point to identify the particular constituent or group of constituents of the plant extract that are adsorbed onto the metal surface. In any case, the adsorbate molecules on the metal surface constitute a barrier to charge and mass transfer between the metal and the corrodent, thereby protecting the metal surface from corrodent attack. The larger the degree of surface coverage resulting from enhanced adsorption of molecules of the plant extract, the greater the protection to corrosion offered by the inhibitor (Oguzie et al, 2007).

Table 1: Major constituents of *Moringa oleifera*

Phytochemical	Ether extract	Ethanol extract	Water extract
Gallic tannins	+	+	++
Catechol tennins	+	-	++
Coumarins	-	-	-
Steroids and triterpenoids	+++	++	++
Flavonoids	++	++	++
Saponins	+	+	++
Anthraquinones	+	++	+++
Alkaloids	+	-	++
Reducing sugars	-	++	++

Kasolo et al., 2010

Adsorption considerations and Adsorption Isotherms

In the situation where it is suspected that the inhibition of metal corrosion occurred as a result of the adsorption of molecules of plant extracts onto the metal surface, it is instructive to investigate the possible adsorption mode by testing the experimental data obtained with several adsorption isotherms. Such an exercise will greatly elucidate one's understanding of the corrosion inhibition mechanism. The generalized expression for several adsorption isotherms usually tested is of the form (Okafor, 2008).

$$f(\theta, x)\exp(-\alpha\theta) = kC \quad (4)$$

where f (θ, x) is the configuration factor whose functional form depends on the physical model adopted and assumptions made in deriving the isotherm, θ is the degree of surface coverage, x is known as the size ratio which gives the number of water molecule replaced by the inhibitor molecule, α is a molecular interaction parameter whose value depends on the type of molecular interactions in the adsorption layer and the degree of homogeneity of the surface, C is the inhibitor concentration while k is the adsorption equilibrium constant which is temperature dependent according to the relation (Okafor, 2008; Umoren and Ebenso, 2008).

$$k = \frac{1}{55.5}\exp\left(-\frac{\Delta G_{ads}}{RT}\right) \quad (5)$$

where ΔG^o_{ads} is the standard free energy of adsorption, R, is the molar gas constant and T is absolute temperature. Several adsorption isotherms were tested for fit with the experimental data. These include the Langmuir, Frumkin, Temkin, Freundlich and the Flory-Huggins isotherms. Incidentally, the Langmuir isotherm gave the best fit with the experimental data. The Langmuir isotherm equation is of the form (Oguzie et al, 2007; Sharma et al, 2008):

$$C/\theta = 1/k + C \quad (6)$$

From a plot of $\frac{C}{\theta}$ against C, a straight line graph was obtained with a slope of 0.984 which is just about unity and an intercept of 0.053 was obtained. The coefficient of correlation R^2, gave the degree of fit between the experimental data and the isotherm equation. The value obtained was found to be 0.992 which indicates a very good fit between the Langmuir isotherm and the experimental data. Figure 4 shows the Langmuir isotherm plot for the inhibition of the corrosion of aluminium in 0.5M HCl by *Moringa oleifera*leaf extract. Using the obtained value of the intercept fromthe graph and equations 4 and 5, a value of $1.3kJmol^{-1}$ was obtained for ΔG^o_{ads} in 0.5M HCl. The very good fit of the experimental data with the Langmuir adsorption isothermsuggests that the Langmuir adsorption model isapplicable in the corrosion inhibition mechanism. In the derivation of the Langmuir isotherm, it was assumed that the adsorption sites on the metal surface are uniformly distributed and energetically identical and that the maximum number of adsorbed molecules per site is one, implying a case of monolayer adsorption. Additionally, it was assumed that adsorbate molecules do not interact with one another. The negative values of ΔG^o_{ads}, the Gibb's free energy of adsorption, obtained means that the adsorption process was spontaneous.

Table 2: Values of adsorption parameters for aluminium alloy

Isotherm	Intercept	Slope	k (x 10^{-4})	R^2	ΔG^o ($kJmol^{-1}$)
Langmuir	0.053	0.984	18.87	0.992	1.73
Temkin	1.004	0.130	2259.90	0.936	2.93
Freundlich	0.022	0.159	1.67	0.954	1.13

The values of ΔG^o_{ads} obtained in this study is low enough for one to attribute the adsorption process as due to an electrostatic interaction between the atoms/ions on the metal surface and the adsorbate molecules, a mechanism which is consistent with physical adsorption (Umoren et al, 2008d). The value of the free energy of adsorption is still within the range characterising the physisorption model of adsorption. It has

Scientia Africana, Vol. 13 (No.1), June 2014. Pp 52-63
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been pointed out (Umoren et al, 2008a; Umoren et al, 2008c; Umoren et al, 2009) that generally, values of ΔG^o_{ads} up to -20 $kJmol^{-1}$ are consistent with physical adsorption. The interactions involved in this mechanism are more or less weak lectrostatic interactions between metal atoms and adsorbate species. In fact, the adsorption energies involved have the same range of energy values as the van der Waals bond energies (Umoren et al, 2008a).

However, values of ΔG^o_{ads} which are more negative than - 40 $kJmol^{-1}$ are associated with chemical adsorption, also called chemisorption. The mechanism involves charge sharing or charge transfer between the atoms of the metal and the adsorbate molecules. The associated bonds are strong and the corresponding bond energies could be as large as those characteristics of primary bonds in solids (Umoren et al, 2008a).

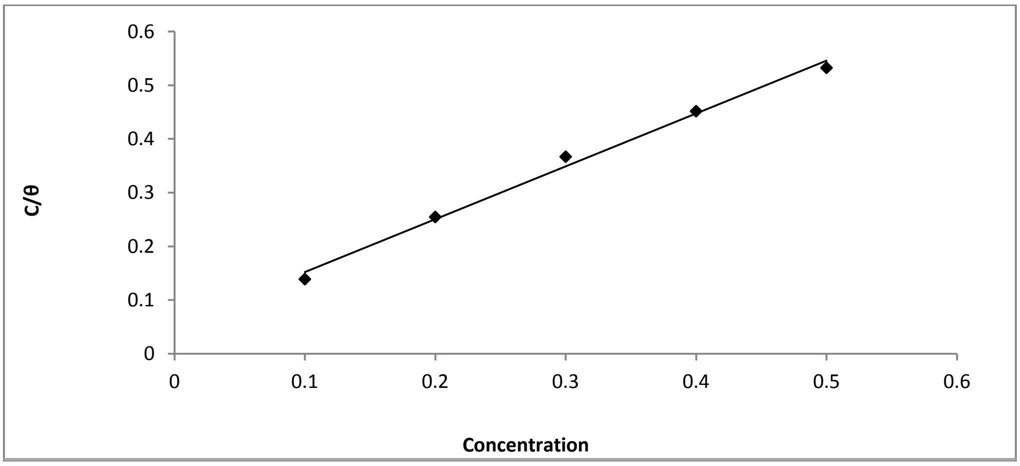

Figure 5. Langmuir adsorption isotherm for inhibition of aluminium in 0.5M HCl by *Moringa oleifera* leaf extract.

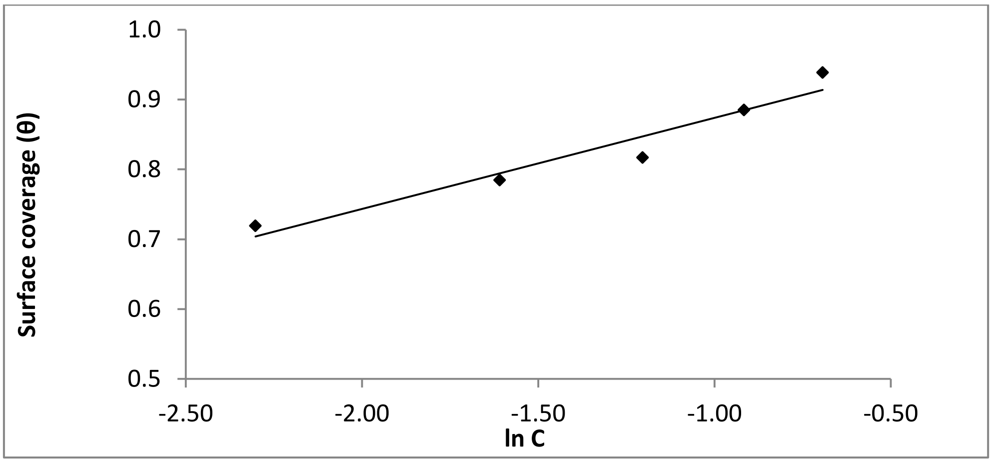

Figure 6. Temkin adsorption isotherm for inhibition of aluminium in 0.5M HCl by *Moringa oleifera* leaf extract.

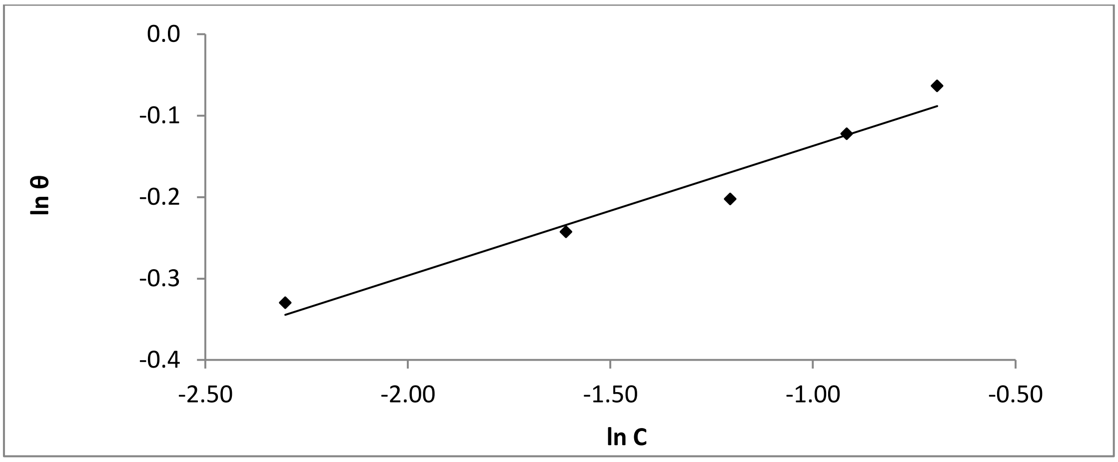

Figure 7. Freundlich adsorption isotherm for inhibition of aluminium in 0.5M HCl by *Moringa oleifera* leaf extract.

CONCLUSION

It has been shown in this study that the addition of *Moringa oleifera* leaf extract to HCl reduces the corrosion rate of aluminium in 0.5M Hydrochloric acid. The inhibition efficiency of the plant extracts increases with increase in concentration. The experimental data obtained are best described by the Langmuir adsorption isotherm, signifying the formation of a single layer of inhibitor molecules onto aluminium alloy surface. Moreover, values of the Gibb's free energy of adsorption obtained suggests that the spontaneous physical adsorption of the plant extract molecules is the most likely inhibitory mechanism responsible for the reduction of the corrosion rate of the aluminium in 0.5M HCl containing *Moringa oleifera* leaf extract.

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