Nigerian Research Journal of Engineering and Environmental Sciences 4(1) 2019 pp. 71-78

Nigerian Research Journal of Engineering and Environmental Sciences
Journal homepage: www.rjees.com

Original Research Article

Evaluation of extract of *Caesalpinia pulcherrima* flower as corrosion inhibitor for Al-Si-Mg/SiC composite in 0.5M HCl Solution

Asuke, F., *Abdulwahab, M., Musa, A.A. and Haruna, I.M.
Department of Metallurgical and Materials Engineering, Ahmadu Bello University, Zaria, Nigeria.
asukef@gmail.com; *mabdulwahab@abu.edu.ng; abdulrahman@abu.edu.ng

ARTICLE INFORMATION

Article history:
Received 29 October, 2018
Revised 26 November, 2018
Accepted 27 November, 2019
Available online 30 June, 2019

Keywords:
Al-Si-Mg/SiC composite
Caesalpinia pulcherrima
Potentiodynamic polarization
Thin film
Adsorption

ABSTRACT

Gravimetric-based mass loss and potentiodynamic polarization method of corrosion measurement were employed to study the effect of extract of Caesalpinia pulcherrima flower as inhibitor in the corrosion of Al-Si-Mg/SiC composite in 0.5M HCl solution at room temperature. Different concentrations of inhibitor (0, 1.5. 3.0, 4.5, 6.0, 7.5%v/v) with time of two (2) to ten (10) hours at an interval of two hours were used for the weight loss method. The results showed a decrease in corrosion rate with increase in inhibitor concentration. The maximum inhibitor efficiency (IE) of 88.87% was obtained at a concentration of 6.0 and 7.5%v/v with exposure time of two hours followed by 81.21% inhibitor efficiency (IE) at 7.5% v/v after six hours for the weight loss method. The potentiodynamic polarization technique recorded 77.679% and 79.381% inhibitor efficiency (IE) for 6.0 and 7.5%v/v respectively. The results of the weight loss and potentiodynamic polarization technique were found to be in agreement. Microstructural observation of the samples revealed the effect of inhibitor on the test samples.

1. INTRODUCTION

Corrosion is a natural process, which converts a refined metal to a more chemically-stable form, such as its oxide, hydroxide, or sulfide. It is the gradual destruction of materials (usually metals) by chemical and/or electrochemical reaction with their environment. Corrosion engineering is the field dedicated to controlling and monitoring corrosion (Haizhi, 2003).

Due to a wide spectrum of applications of metals across industries as well as homes, researchers have designed various techniques to control metallic corrosion (Ashassi-Sorkhabi and Nabavi-Amri, 2000; Singh *et al.,* 2009; Shylesha *et al*., 2011; Loto *et al*., 2012). Rusting, the formation of iron oxides is a well-known example of electrochemical corrosion. This type of damage typically produces oxides or salt of the original metal, and results in a distinctive orange coloration. Corrosion can also occur in materials other than metals, such as ceramics or polymers, although in this context, the term "degradation" is more commonly used.

Corrosion degrades the useful properties of materials and structures including strength, appearances and permeability to liquids and gases (Abdulwahab *et al*., 2012a).

Many structural alloys corrode merely from exposure to moisture in air, but the process can be strongly affected by exposure to other corrosive gases (Zaki, 2006). Corrosion can be concentrated locally to form a pit or crack, or it can extend across a wide area more or less uniformly corroding the surface. As a result, methods to reduce the activity of the exposed surface, such as passivation and chromate conversion, can increase a material's corrosion resistance (Asuke *et al*., 2009). However, some corrosion mechanisms are less visible and less predictable (Asuke *et al.,* 2009).

Aluminium and its alloy find application in many industries due to their high strength to weight ratio, good corrosion resistance, excellent workability, high electrical and thermal conductivity (Abdulwahab *et al*., 2011). As a fundamental progressive step in this quest, it has been found that the use of corrosion inhibitors is one of the most practicable ways for providing protection for metals in aggressive media (Sachin *et al*., 2009). Although synthetic inhibitors when used for corrosion control, have been reported to indicate an excellent performance. But majority of these inhibitors are not eco-friendly and are expensive (Popoola *et al*.,2012). Therefore, effort towards identifying potential eco-friendly and less expensive corrosion inhibitors remain relevant and important. The growing interest among researchers for green inhibitors remained a top research focus. In this direction, plant extracts and oils have gained acceptance as corrosion inhibitors that are considered safe, eco -friendly, available and less expensive for most metals and alloys (Peter *et al*., 2010; Adams *et al*., 2016; and Abdulwahab *et al*., 2012b) Accordingly, the present study involves the use of extract of *Caesalpinia pulcherrima* flower as corrosion inhibitor for Al-Si-Mg/SiC composite in 0.5M HCl acid solution.

2. MATERIALS AND METHOD

2.1. Materials

The materials used in this research include; an alloy of aluminium-silicon-magnesium, magnesium, silicon carbide powder, hydrochloric acid, methanol, extract of *Caesalpinia pulcherrima,* grit papers (400 and 600), polishing powder (Alumina) and distilled water.

2.2. Equipment

The equipment used in this work include; digital weighing balance, measuring cylinder, beakers, stopwatch, grinding machine, brush, sponge, masking tape, thread, syringe, stirrer, crucible, funnel, sauchill concentrator, spatula, Autolab potentiostat, graphite rod, and a glass of saturated Ag/AgCl.

2.3. Method of Production of the Composite

The aluminum metal-matrix composite that was used in this study was produced using the chill casting method (Hiremath and Hemanth, 2017) at the foundry shop of the Department of Metallurgical and Materials Engineering, Ahmadu Bello University, Zaria, Nigeria. Aluminium-silicon-magnesium alloy were placed in crucible then heated to melting in the furnace. Silicon carbide particles equivalent to 10% of the total weight of the molten metal were preheated. Magnesium equivalent to 0.5% of the total weight of the molten metal was added to the melt, immediately followed by the preheated silicon carbide reinforcement. The melt was thoroughly stirred to obtain a homogeneous mixture, and then poured into the cylindrical metal mould of 30cm length and 1.5 cm diameter which was initially prepared. The produced composite was allowed to solidify in the mould after which it was machined to the dimension of the coupon used (12×10 mm), cleaned properly and stored in a desiccator for further use.

2.4. Extraction of the juice of *Caesalpinia pulcherrima* Flower

The petals of the flower were collected and dried under shade, then completely soaked inside methanol for three days to extract the juice, the methanol was removed using sauchill concentrator in order to concentrate the extract, then dried under shade for one week to achieve maximum evaporation of the methanol from the extract (Nainwal *et al*., 2014).

2.5. Phytochemical Screening of Extract

The phytochemical screening of the *Caesalpinia pulcherrima* extract was carried out in the Faculty of Pharmaceutical sciences laboratory, Ahmadu Bello University, Zaria.

2.6. Gravimetric Corrosion Measurement

Samples of the composite (in form of coupons) were inserted into a beaker containing 0.5M HCl and 100 ml of distilled water for the test. The first corrosion test was conducted at room temperature for two (2) to ten (10) hours. (i.e. after every two hours a sample was removed and weighed and the difference in weight was noted and recorded). The above procedure was repeated with varying inhibitor concentration of 1.5, 3.0, 4.5, 6.0, and 7.5v/v at room temperature. The weight losses, the corrosion rate and inhibitor efficiency were then calculated using the appropriate formulas shown in Equation 1 to 3 respectively. The weight loss was determined by finding the difference between initial weight of the samples and the final weight of the sample after each exposure time using Equation 1.

$$W = Wo - Wf \tag{1}$$

Where:

W = weight loss (g)
Wo = initial weight (g)
Wf = final weight (g)

Corrosion rate (mile per year) was calculated using Equation 2.

$$\text{Corrosion rate } (mpy) = \frac{534w}{DAT} \tag{2}$$

Where:

mpy = miles per year
w = weight loss (mg)
D = density of the materials (g/cm^3)
T = time of exposure (hours)
A = surface area (in^2)

The inhibitor efficiencies for the weight loss and potentiodynamic polarization corrosion measurements were computed using Equation 3.

$$Inhibition\ efficiency = \frac{Wo-W}{Wo} \tag{3}$$

Where W and Wo are the corrosion rates with and without inhibitors respectively.

2.7. Potentiodynamic Polarization Corrosion Measurement

Samples of the composite were attached to a wire and then mounted in an epoxy resin, and allowed to dry for 12 hours. The epoxy resin was ground from the surface of the composite, the working electrode samples were positioned at the glass corrosion cell. The test was carried out using electrode glass cells with an AUTOLAB potentiostat (PGSTAT204) which was connected to a computer. A graphite rod, saturated Ag/AgCl and the composite specimen were used as counter, reference, and working electrode respectively inside a beaker. The working electrode samples were positioned at the glass corrosion cell, leaving 534 mm^2 in contact with the solution. The polarization test was carried out in 0.5M HCl solution at room temperature. The same procedure was repeated for each of the six (6) samples with varying inhibitor concentration of 1.5, 3.0, 4.5, 6.0, and 7.5%v/v. The polarization experiment data were analyzed by GPES software version 1.10.19.

2.8. Microstructural Analysis

The as-cast composite was ground using series of SiC grades impregnated emery papers (400-800 grits), using water as coolant. Polishing was done roughly using 1.0-micron size alumina polishing powder suspended in distilled then followed by 0.5-micron size alumina on a rotating disc of propriety nap cloth. The samples were etched using hydrofluoric and nitric acid after which the microstructure of the samples was captured by metallurgical microscope. The microstructure of the other samples (uninhibited and inhibited) was taken without any surface preparation.

3. RESULTS AND DISCUSSION

3.1. Phytochemical Screening of the Extract

The phytochemical screening of the extract, shown in Table 1 was carried out to identify the presence of the chemical constituents in the plant that enable the plant to have the inhibitive property. It was found that tannins and flavonoid were present in the extract while alkaloids, steroids and anthraquinones were absent. The presence of flavonoids and tannins contribute to the inhibition efficiency of the plant possibly because they are cyclic compounds containing O atoms attached to it (Helen *et al*., 2014). These chemical constituents might have been adsorbed on the surface of the sample thereby blocking the active corrosion site (Idawu *et al*., 2013).

Table 1: Phytochemical test result of *Caesalpinia pulcherrima* flower

Phytochemicals	Result
Alkaloids	-
Anthraquinones	-
Flavonoids	+
Tannins	+
Saponin	-

3.2. Corrosion Measurement using Weight Loss Method

The results for the weight loss corrosion test are presented in Figure 1. Figure 1 shows the variation of corrosion rates with exposure time of (2-10 hours) at room temperature using extract of *Caesalpinia pulcherrima* flower as corrosion inhibitor in 0.5M HCl solution. From the Figure, the highest corrosion rate was observed in the control solution at exposure time of 2 hours and the least value of corrosion rate was observed at 7.5%v/v of *Caesalpinia pulcherrima* and exposure time of 8 hours. This shows that the rate of corrosion in the presence of inhibitor studied depends on both the inhibitor concentration and exposure time.

There was a decrease in corrosion rate with increase in inhibitor concentration. This behaviour could be attributed to the strong interaction of the compound with metal and resulted in the adsorption of the inhibitor molecules on metal surface (Hari *et al*., 2016). It may also be due to the presence of lone pair of electrons on nitrogen atom which it can donate to metal surface to increase adsorption thereby enhancing inhibition. Adams *et al.* (2016) also reported that increasing inhibitor concentration increases the formation of passive films which protect the material against further corrosion attack. The anomalous behavior observed in the plot is generally attributed to the roughness and surface inhomogeneity of the samples (Lebrini *et al.,* 2007).

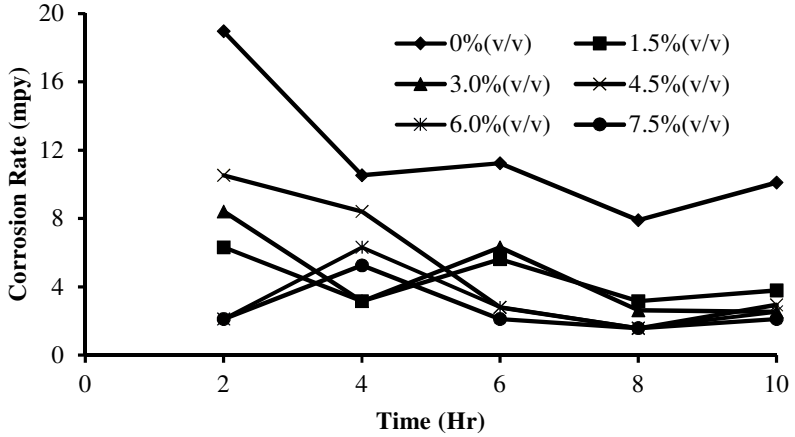

Figure 1: Variation of corrosion rates (mpy) with time for Al-Si-Mg/SiC composite in 0.5M HCl solution

3.3. Inhibitor Efficiency (IE%) Measurement

The results for the inhibition efficiency were presented in Figure 2. Figure 2 shows the variation of inhibitor efficiency with time for the various inhibitor concentrations. The inhibition efficiency increased with increase in the concentration of the extract of *Caesalpinia pulcherrima* flower. Maximum inhibition efficiency of 88.87% was observed at 6.0%v/v and 7.5%v/v inhibitor concentration and exposure time of two (2) hours, while the next level of 81.21% inhibitor efficiency was obtained at 7.5%v/v after 6 hours. Inhibitor efficiency of 80.00% was also observed at the same inhibitor concentration of 6.0%v/v and 7.5%v/v and time interval of 8 hours. Minimum inhibition efficiency of 20.04% was observed at 4.5%v/v inhibitor concentration and exposure time of four (4) hours. Therefore, it can be concluded that, inhibition efficiency increased with increase in concentration of inhibitor.

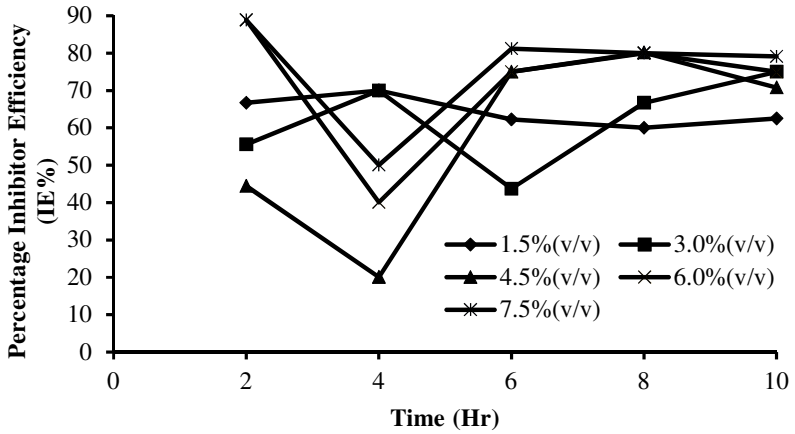

Figure 2: Variation of percentage inhibitor efficiency (IE%) with time for Al-Si-Mg/SiC composite in 0.5M HCl solution

3.4. Potentiodynamic Polarization Corrosion Measurement

The results for the potentiodynamic polarization corrosion measurement are presented in Table 2. The results of potentiodynamic polarization corrosion measurements presented in Table 2 shows that the current density (J_{corr}) decreased with increase in inhibitor concentration without causing significant change in corrosion potential (E_{corr}). This suggests that, the extract is a mixed types inhibitor within the experimental conditions under investigation (Adams *et al.*, 2016). Also, it can be observed from Table 2 that current density (J_{corr}) decreased with increase in inhibitor concentration causing significant change in corrosion potential (E_{corr}). The corrosion potential of the composite in control solution was -1.5076 V, but at various concentration of the inhibitor, the corrosion potential values shifted towards the anodic side (-1.5076 to -0.53344) indicating that the extract affects the anodic reaction predominantly.

Table 2: Potentiodynamic polarization corrosion measurements

Inhibitor conc. (v/v)	E_{corr}, Calculated (V)	E_{corr}, Observed (V)	J_{corr}, (A/cm^2)	I_{corr} (A)	Polarization resistance (Ω)	Corrosion rate (mm/yr)	Inhibitor Efficiency (%)
0.0	-1.5076	-1.4069	0.0015043	0.0015043	23.911	16.4	0
1.5	-1.3637	-1.2928	0.001294	0.001294	41.129	14.103	14.006
3.0	-1.3546	-1.2815	0.000902	0.000902	53.846	9.8313	40.053
4.5	-1.2253	-1.0942	0.000412	0.000412	68.13	4.4894	72.626
6.0	-0.92245	-0.8986	0.000336	0.000336	66.751	3.6605	77.679
7.5	-0.53344	-0.5212	0.00031	0.00031	100.16	3.3815	79.381

The linear polarization results show that addition of extract of *Caesalpinia pulcherrima* flower resulted in an increase in polarization resistance value (Rp) of the composite from 23.911 Ω (uninhibited) to 100.16 Ω (inhibited) condition with an inhibitor efficiency of 79.381%. It shows that the control solution have the highest corrosion rate (16.4 mm/yr) and least corrosion resistance (23.911 Ω) on the Al-Si-Mg/SiC composite while the solution with the highest inhibitor concentration (7.5%v/v) has the least corrosion rate (3.3815 mm/yr) and highest corrosion resistance (100.16 Ω), Therefore from Table 2, it can be seen that the corrosion rate decreases with increase in inhibitor concentration while the corrosion resistance increases with increase in inhibitor concentration.

3.5. Microstructural Analysis

Plate 1 shows the microstructure of the as-cast composite while plate 2 shows the micrograph of the corroded composites. Plate 1 shows the microstructure of the as-cast Al-Si-Mg/SiC composite. From Plate 1, there are two distinct areas; the bright region and the dark region which indicates the matrix (Al-Si-Mg) and the reinforcement (SiC) respectively. It can also be observed that the reinforcement distributed uniformly within the grain boundaries of the aluminium matrix.

Plate 2 (a) shows the surface morphology of the most corroded (i.e. uninhibited) composite with severe corrosion product covering the entire surface of the material while Plate 2(b) shows the surface morphology of the least corroded composite, due to the presence of 7.5%v/v inhibitor. The matrix is still visible with minimal corrosion product compared to the uninhibited sample (a). This confirms that the presence of the inhibitor reduces the corrosion rate.

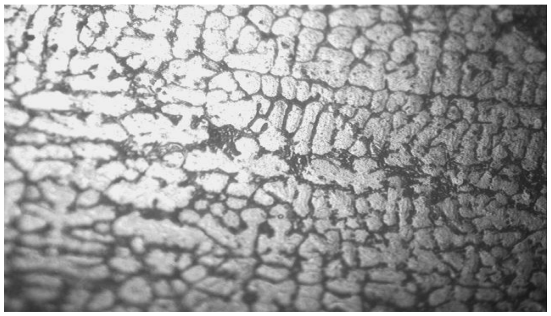

Plate 1: Micrograph of as-cast Al-Si-Mg/SiC composite (×100)

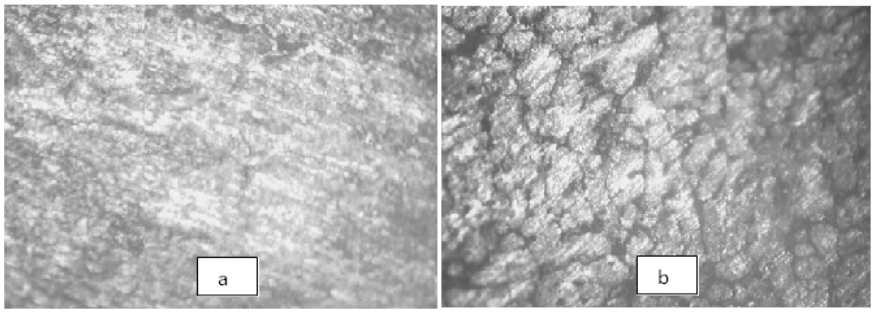

Plate 2: Micrograph of the corroded composite (Al-Si-Mg/SiC) after exposure time of two hours in 0.5M HCl solution (a) without inhibitor and (b) with 7.5%v/v inhibitor concentration (×100)

4. CONCLUSIONS

In this research, the corrosion inhibition of Al-Si-Mg/SiC composite in 0.5M HCl solution using extract of *Caesalpinia pulcherrima* flower at room temperature was studied. Based on the results obtained, the following conclusions were drawn:

i. The extract of *Caesalpinia pulcherrima* flower can be used as inhibitor for Al-Si-Mg/SiC composite.
ii. The result of gravimetric based-mass loss and potentiodynamic polarization technique indicates an improvement on corrosion resistance up to a concentration of 7.5%v/v.
iii. The results of the weight loss and potentiodynamic polarization measurement are in a good in agreement.

iv. For duration of two and eight hours, 6.0%v/v of inhibitor is suitable for use, while 7.5%v/v is suitable for use for duration of 6 hours.

REFERENCES

Abdulwahab, M., Kasim, A., Fayomi, O.S., Asuke, F. and Popoola, A.P.I. (2012a). Inhibitive effect of Arachis hypogeae on the corrosion of mild steel in sulphuric acid solution. *Journal of Materials and Environmental Science*, 3(6), pp. 1177-1182.

Abdulwahab, M., Popoola, A.P.I. and Fayomi, O.S.I. (2012b) Inhibitive Effect by *Ricinus communis* on the HCl/H_3PO_4 Acid Corrosion of Aluminium Alloy. *International Journal Electrochemical Science*, 7, pp. 11706-11717

Abdulwahab, M., Yaro, S.A., Hassan, S.B. and Popoola A.P.I. (2011). Inhibitive Action of Ferrous Gluconate on Aluminum Alloy in Saline Environment. *Materials and design,* 32(3), pp. 1159- 1166

Adams, S. M., Yaro, S. A., Abdulwahab, M. and Oguzie, E. E. (2016). Extract of *Combretum micranthum* as corrosion inhibitor for Al-Si-Mg alloy in simulated sea water environment. *Nigerian Journal of Technology,* 35(4), pp. 888 – 894

Ashassi-Sorkhabi, H. and Nabavi-Amri, S.A. (2000). Corrosion inhibition of carbon steel in petroleum/water mixtures by N-containing compounds. *Acta Chimica Slovenical journal.*, 47, pp. 507-717.

Asuke, F., Yaro, S.A., Oloche, O.B and Aigbodion, V.S (2009). Potential of palm exudate (Palmwine) as corrosion inhibitor for Al-Si alloy in caustic soda solution. *Journal of Metallurgy and Materials Engineering*, 2, pp. 48-54.

Haizhi, Y. (2003). An overview of the Development of Al-Si alloy-based material for Engine Applications. *Journal of Materials Engineering and performance*, 12(3), pp. 288-297.

Hari, O., Anjoo, B., Harish, K. V. and Pradeep K. (2016). Corrosion Inhibition of carbon steel by an Isatin Schiff base in acidic medium. *Der Pharma Chemica,* 8(12), pp. 149-156

Helen, L.Y.S, Rahim, A.A., Saad, B., Saleh, M.I. and Bothi, R.P. (2014). *Aquilaria crassna* leave extracts as a green corrosion inhibitor for mild steel in 1M HCl medium. *International Journal of Electrochemical Science,* 9, pp. 830-846.

Hiremath, A. and Hemanth, J. (2017). Experimental Evaluation of the Chill Casting Method for the Fabrication of LM-25 Aluminum Alloy-Borosilicate Glass (p) Composites. In: *Key Engineering Materials* (Vol. 748, pp. 69-73). Trans Tech Publications.

Idawu, Y.S. (2013). Evaluation of plant extracts as corrosion inhibitors for mild steel in acidic environment. Ph.D Thesis, Department of Metallurgical and Materials Engineering, Ahmadu Belllo University, Zaria.

Lebrini, M., Lagrenee, M., Vezin, H., Traisnal, M. and Bentiss, F. (2007). Experimental and theoretical study for corrosion inhibition of mild steel in normal hydrochloric acid solution by some new macrocyclic polyether compounds. *Journal of Corrosion Science*, 49, p. 2254.

Loto, R.T., Loto, C. A. and Popoola, A. P. I. (2012). Effect of amino benzene concentrations on the corrosion inhibition of mild steel in sulphuric acid. *International Journal of Electrochemical Science*, 7, pp. 7016-7032.

Nainwal, P., Nanda, D., Ranveer, B. and Pankaj, N. (2014). A review on Phytochemical and Pharmacological aspects of *Caesalpinia pulcherrima. International Journal of Research in Ayurveda and Pharmacy,* 2(2):416-421

Peter, C., Eno, E. and Udofot, J. (2010). Azadirachta indica Extract as Corrosion Inhibitor for Mild steel in Acid Medium. *International Journal of Electrochemical Science,* 5, pp. 978-993.

Popoola, A.P.I., Fayomi, O.S.I. and Abdulwahab, M. (2012). Degradation Behaviour of Aluminum in 2M HCl/HNO_3 in the presence of *Arachis hypogeae* Natural oil. *International Journal of Electrochemical Science*, 7, pp. 5817-5827

Sachin, H.P., Moinuddin, K.M.H. and Bhujangaiah, N. S. (2009). Surface modification of mild steel by orthophenylenediamine and its corrosion study. *International Journal of Electrochemical Science*, 4, pp. 134-143.

Shylesha, B.S., Venkatesha, T.V. and Praveen, B. M. (2011). Corrosion inhibition studies of mild steel by new inhibitor in different corrosive medium. Research *Journal of Chemical Sciences*, 1(7), pp. 46-50.

Singh, M., Bhrara, K. and Singh, G. (2009). The inhibitory effect of di-ethanolamine on corrosion of mild steel in 0.5M sulphuric acid medium. *Portugaliae Electrochimica Acta*, 26, pp. 479-492.

Zaki, A. (2006). *Principles of corrosion engineering and corrosion control.* 1 ed. Elsevier science and Technology.