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Investigating the influence of immersion time and inhibitor concentration on the inhibiting potential of imperrata cylindrica as Corrosion Inhibitor of mild steel

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Abstract

In this research, the influence of immersion time and inhibitor concentration on the inhibiting potential of imperrata cylindrical (spear grass) as Corrosion Inhibitor of mild steel in sulphuric acid solution was investigated using weight loss technique at room temperature. The research was achieved by using 1M concentration of the sulphuric acid solution and varied inhibitor (spear grass) concentration of 1.0g, 1.5g, 2.0g, 2.5g and 3.0g respectively; readings were obtained every 24 hours (1 day) for a period of 120 hours (5 days). Weight loss, corrosion penetration rate, inhibition efficiency and degree of surface coverage were calculated. The electrode potential between the corroding mild steel and the corrosive environment was measured using zinc electrode as the reference electrode. Results obtained revealed that both the immersion time and inhibitor concentration has marked influence on the potential of imperrata cylindrical (spear grass) as corrosion inhibitor for mild steel sulphuric acid solutions. The results show that both the inhibition efficiency and degree of surface coverage increased with an increase in the concentration of the imperrata cylindrical (spear grass) causing a corresponding decrease in corrosion penetration rate and weight loss with increased imperrata cylindrical (spear grass) concentration. Also, the analysis of the results show that inhibition efficiency and surface coverage decreased with increased immersion time, causing a gradual and significant increase in both the weight loss and corrosion penetration rate with time.

Keywords: Imperrata cylindrica, Mild steel Inhibition efficiency, Degree of Surface coverage, Sulphuric Acid Solution

1. Introduction

Metallic corrosion is the passage of the metal into the chemically combined state [1-3]. Mineral acids find applications in many industries: chemical, fertilizer, steel, mineral, water, oil, food, etc. [4]. The acidic properties of their ionized, aqueous solutions are due to the presence of hydrogen ion, H^+ and its complexed form, hydroxonium ion, H_3O^+. The dissolution of metals in solutions of acids is a heterogeneous electrochemical reaction. Initially, two phases co-exist - a solid and a liquid, but as the reaction starts, a third phase appears – hydrogen gas, evolving from the metal surface. The kinetics of the dissolution process depend on the collision between the solvated protons and the ‘‘active sites’’ on the metallic crystal surface; promoting anodic dissolution [5-7]. Hence, metal surfaces exposed to aqueous acidic environments are observed to undergo corrosion attack. The corrosion of mild steel in any given environment may proceed via different mechanisms and manifest in various forms. In a bid to reducing corrosion, some organic compounds called inhibitors are added to the aggressive solution in contact with the metal surface thereby inhibiting corrosion and reducing the corrosion rate of the metal [8]. Tetraoxosulphate (VI) acid (Sulphuric acid) is the corrosive media used in this work. It is produced more than any other chemical in the world. It has large scale uses covering nearly all industries, such as fertilizer industries, petroleum refinery, paint industry, steel pickling, extraction of non-metals, and manufacture of explosives.

Mild steel is a ferrous alloy with less than 0.25% carbon content. It is unique among other metallic materials because it is readily available and has very wide range of application for domestic, services, construction, marine, industrial and engineering purposes. This grade of steel is very reactive and will readily revert to iron oxide (rust) in the presence of water (H_2O), oxygen (O_2) and ions such as chloride ions (Cl^-) [9]. All of these corrosion agents exist freely in the atmosphere and other mild steel service environment [10 – 11]. The readiness of mild steel to oxidize when its surface is exposed means that it must be adequately protected from the elements in order to meet and exceed its design life.

The mitigation of metallic corrosion using chemical inhibitors

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is a very active field of research. Chemical inhibitors are organic and inorganic chemical compounds that are adsorbed in the metal surfaces to control, prevent and/or minimize the destructive corrosion reactions [12 – 13]. Corrosion inhibiting chemical compounds are extensively applied industrially to minimize the deterioration rate of metals and alloys used in corrosive environments. However, most corrosion inhibitors are costly, toxic and environmentally unfriendly [13 – 15]. Most common inhibiting organic compounds evaluated as effective corrosion inhibitors for ferrous alloys are heterocyclic compounds with oxygen (O), nitrogen (N) and/or sulphur (S) as hetero atoms [16 – 20].

Over the years, concerted efforts have been deployed to finding suitable or green (safe and friendly) corrosion inhibitors of organic origin in various corrosive media [21]. Several factors including cost and amount, easy availability and most important safety to environment and its species need to be considered when choosing an inhibitor [21].

Plant extracts have attracted the attention of researchers as eco-friendly corrosion inhibitors for many decades. Plant extracts are an incredibly rich source of natural chemical compounds that can be extracted by simple procedures with low cost and are biodegradable in nature. Their abundant availability is also an advantage of the naturally occurring organic compounds. The actual inhibitors in the plants extracts are usually alkaloids and other organic nitrogen bases, as well as carbohydrates, proteins, and their acid hydrolysis products. Alkaloids have an ability to coordinate the transition metals or their alloys via the d orbitals of metal and empty p-orbitals of the heteroatoms in the inhibitor molecules. A number of natural compounds have been used as corrosion inhibitors for metals [21-25]

Thus, this research focuses on investigating the influence of immersion time and inhibitor concentration on the inhibiting potential of imperrata cylindrica as Corrosion Inhibitor of mild steel

2. Materials and Methods

2.1 Materials

Mild steel rod of 12mm in diameter was used in this research work. The chemical analysis of the steel is shown in Table 1. Imperrate cylindrica leaves is the ‘‘green’’ inhibitor used while tetraoxosulphate VI acid is the corrosive agent used in this research work. The phytochemical composition of the imperrata cylindrica is shown in Table 2

Table 1: Chemical Composition of Investigated Mild Steel

Element	Weight %	Element	Weight %
C	0.288	Nb	0.0001
Si	0.192	Al	0.021
Mn	0.624	B	0.0001
S	0.052	W	0.0001
P	0.051	Mo	0.0001
Cr	0.182	V	0.0001
Ni	0.090	Ti	0.008
Cu	0.268	Fe	98.224

Table 2: Phytochemical Composition of Imperrata cylindrical

Phytoconstituents	Dichloromethane	Chloroform	Acetone	Methanol
Alkaloids	+	+	-	+
Carbonhydrates	-	+	+	+
Flavanoid	-	-	+	+
Glycosides	-	+	+	+
Proteins	-	-	-	+
Saponins	-	-	+	+
Steroids	+	+	+	+
Tannins	-	-	+	+

Source: [26]

2.2 Experimental Procedure

The mild steel rod was prepared to a dimension of 3mm x 12mm. The samples totaled 30. The samples were degreased using acetone by immersing them for a period of about 5minutes, and then washed (rinsed) with double distilled water and dried. The samples were shared into thirty different containers of the ‘solutions’ with varied concentration of the inhibitor. The solution in the containers into which the different samples were immersed is described below.

A = Blank $1M_{acid}$.
B = $1M_{acid} + 1.0g_{inhibitor}$
C = $1M_{acid} + 1.5g_{inhibitor}$
D = $1M_{acid} + 2.0g_{inhibitor}$
E = $1M_{acid} + 2.5g_{inhibitor}$
F = $1M_{acid} + 3.0g_{inhibitor}$

That is, in samples A – F the concentration of the acid is the same (1M) while that of the inhibitor’s concentration was varied. In all cases, a sample was obtained (taken) from each container at 24 hours interval, rinsed and dried for about five minutes, and then their corrosion rate determined by weight loss. Equations (1-4) were used to obtain the results plotted in Figures 1-8.

$$CPR = \frac{W}{AT} \qquad (1)$$

$$SML = \frac{W}{A} \qquad (2)$$

$$IE = \frac{CRo - CRi}{CRo} \text{ x } 100 \qquad (3)$$

$$\Theta = \frac{Wto - Wti}{Wto} \qquad (4)$$

Where W = weight loss, A = area, T = immersion time, CRo = corrosion penetration rate in the absence of inhibitor, CRi = corrosion penetration rate in the presence of inhibitor, Wto = weight loss in the absence of inhibitor, and Wti = weight loss in the presence of inhibitor.

3. Results and Discussion

3.1 Results

The results of the weight loss, corrosion penetration rate, and inhibition efficiency, degree of surface coverage and electrode potential in 1.0M sulphuric acid solution and varied concentration of imperrata cylindrica as corrosion inhibitor, during the corrosion of investigated mild steel are given in Figures 1-8.

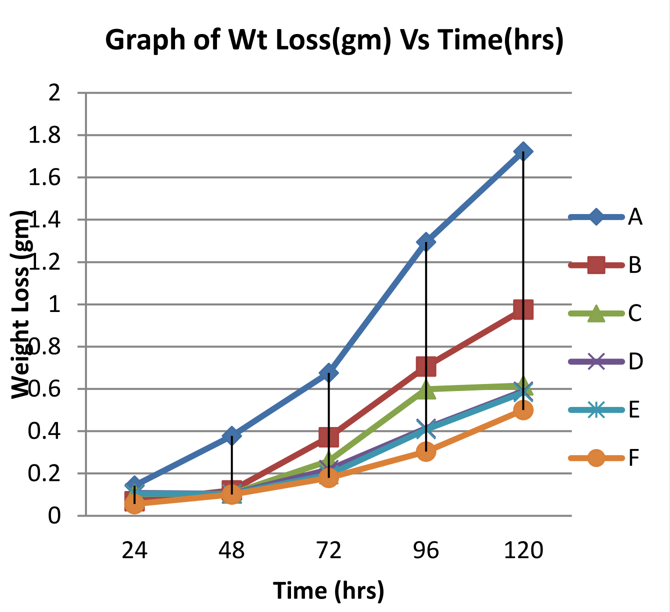

Figure 1. Graph of weight loss (mg/cm^2) Vs Time (hrs)

The Figure 1 above shows the variation of the weight loss data (mg/cm^2) with the immersion time (hrs) recorded for the mild steel in 1.0M H_2SO_4 solutions without and with various concentrations of imperrata cylindrica. (A) Blank; (B) 1.0g; (C) 1.5g; (D) 2.0g; (E) 2.5g; (F) 3.0g.

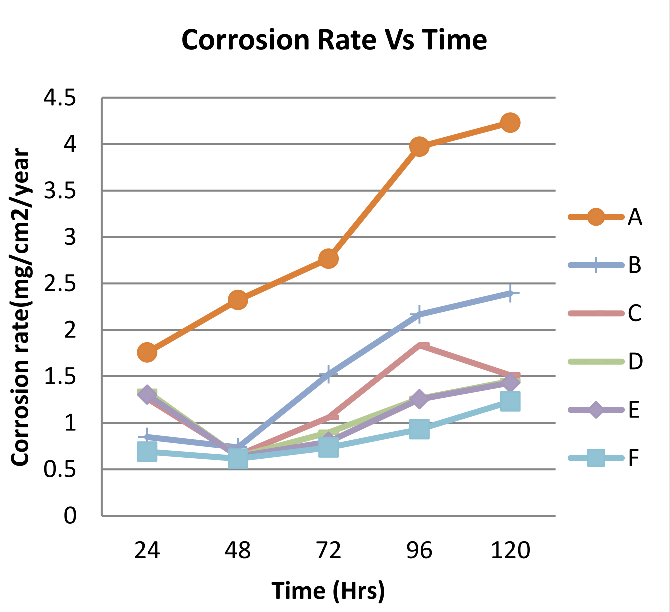

Figure 2.Graph of Corr.Rate(mm/yr) Vs (Time hrs)

The Figure 2 above shows the variation of the Corrosion penetration rate (mg/cm^2/yr) with the immersion time (hrs) recorded for the mild steel in 1.0M H_2SO_4 solutions without and with various concentrations of imperrata cylindrica. (A) Blank; (B) 1.0g; (C) 1.5g; (D) 2.0g; (E) 2.5g; (F) 3.0g.

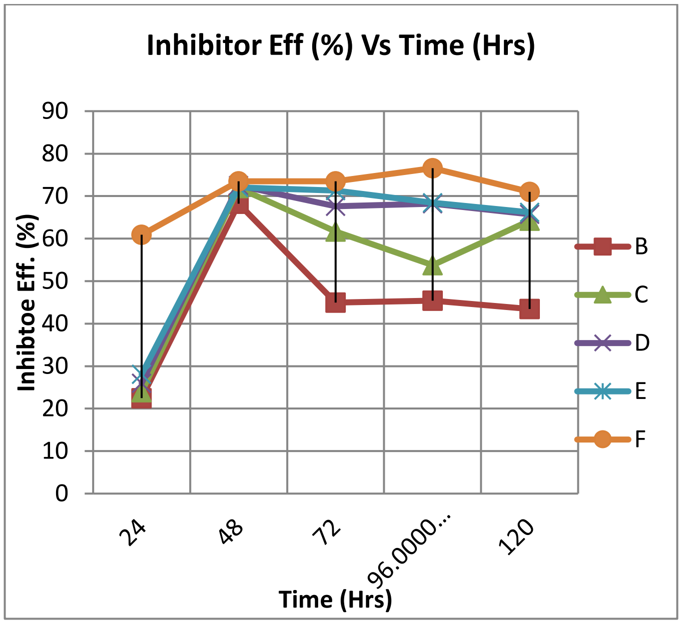

Figure 3.Inhibitor Efficiency (%) Vs (Time hrs)

The Figure 3 above shows the variation of the inhibitor efficiency with the immersion time (hrs) recorded for the mild steel in 1.0M H_2SO_4 solution with various concentrations of imperrata cylindrica as corrosion inhibitor (B) 1.0g; (C) 1.5g; (D) 2.0g; (E) 2.5g; (F) 3.0g.

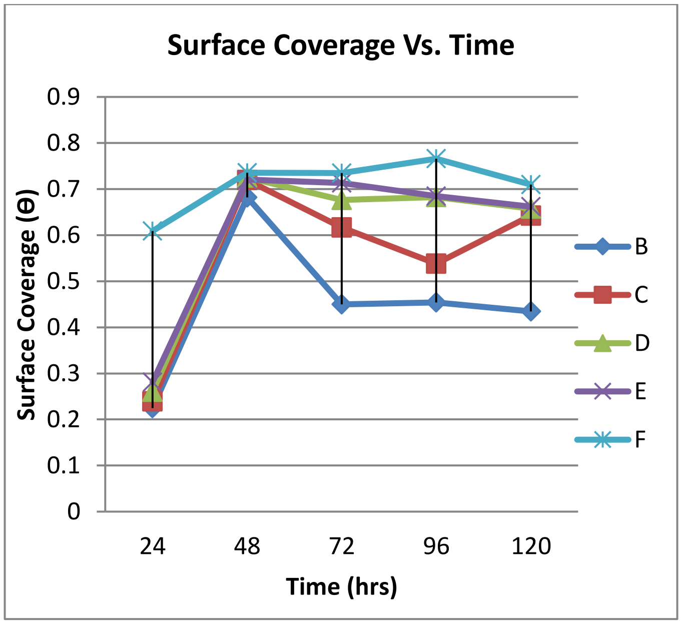

Figure 4.Degree of Surface Coverage (%) Vs (Time hrs)

The Figure 4 above shows the variation of the degree of surface coverage with the immersion time (hrs) recorded for the mild steel in 1.0M H_2SO_4 solution with various concentrations of imperrata cylindrica as corrosion inhibitor (B) 1.0g; (C) 1.5g; (D) 2.0g; (E) 2.5g; (F) 3.0g.

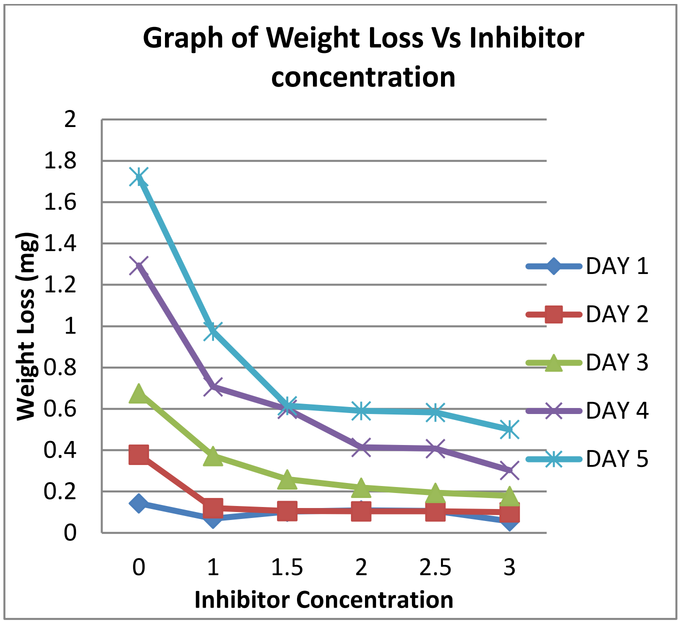

Figure 5. Graph of Weight loss (mg/cm^2) Vs Inhibitor conc. (g)

The Figure 5 above shows the variation of the weight loss data (mg/cm^2) with the inhibitor concentration (g) recorded for the mild steel in 1.0M H_2SO_4 solutions without and with various concentrations of Imperrata cylindrica at 1 day (24hrs) interval for 5days.

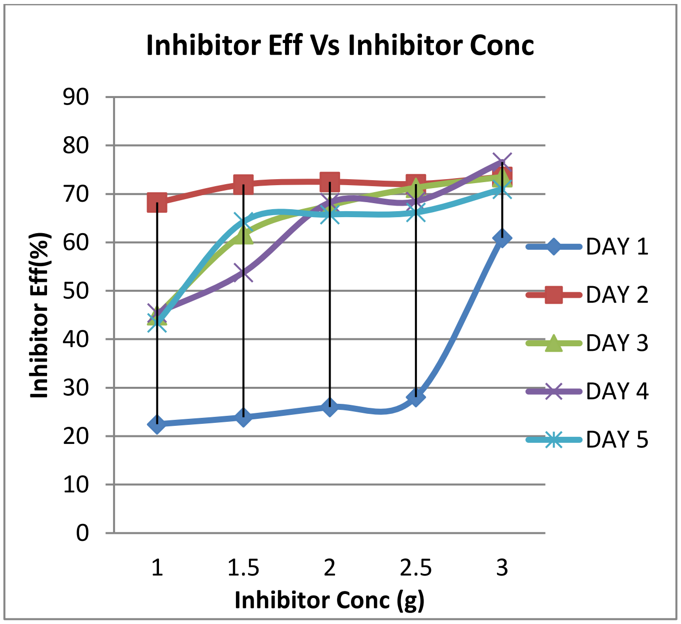

Figure 6. Graph of Inhibitor Efficiency (%) Vs Inhibitor conc. (g)

The Figure 6 above shows the variation of the inhibitor efficiency (%) with the inhibitor concentration (g) recorded for the mild steel in 1.0M H_2SO_4 solutions with various concentrations of Imperrata cylindrica at 1 day (24hrs) interval for 5days (120hrs).

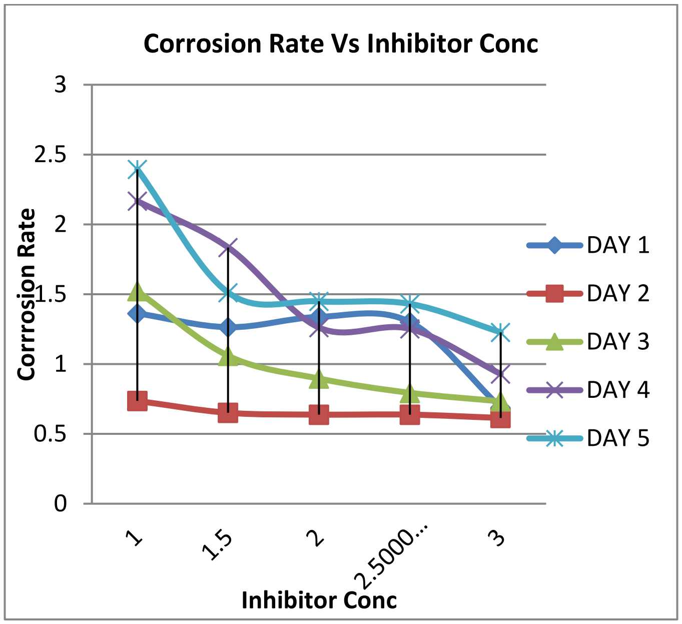

Figure 7. Graph of Corr. rate (mg/cm$^{2/}$yr) Vs Inhibitor conc. (g).

The Figure 7 above shows the variation of the Corrosion penetration rate (mg/cm$^{2/}$yr) with the inhibitor concentration (g) recorded for the mild steel in 1.0M H_2SO_4 solutions with various concentrations of *Imperrata cylindrica* at 1 day (24hrs) interval for 5days (120hrs).

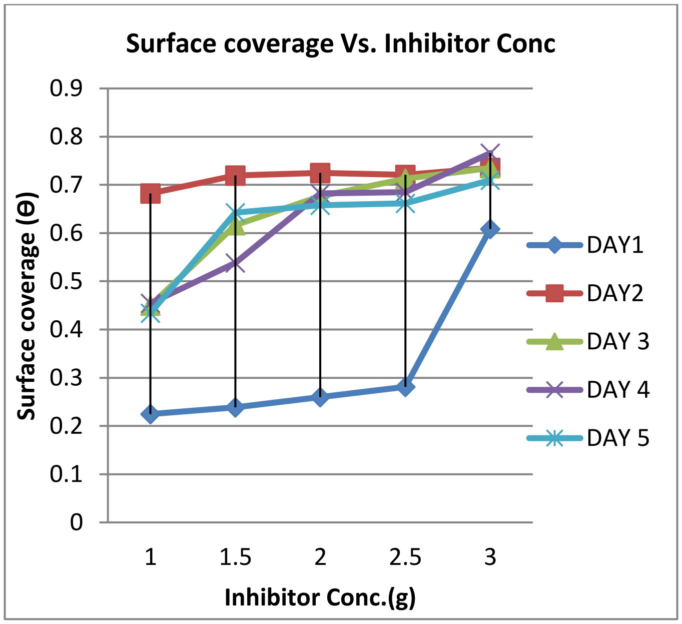

Figure 8. Graph of Surface Coverage Vs Inhibitor conc. (g)

The Figure 8 above shows the variation of the degree of surface coverage with the inhibitor concentration (g) recorded for the mild steel in 1.0M H_2SO_4 solutions with various concentrations of Imperrata cylindrica at 1 day (24hrs) interval for 5days (120hrs).

3.2 Discussion

The plots of Figures 1 – 4 show the influence of the immersion time on the inhibiting action of the imperrata cylindrica on the mild steel samples. The results obtained show that the immersion time has a significant influence on the weight loss, corrosion penetration rate, inhibition efficiency and the degree of surface coverage. The results (Figures 1 – 2) showed that the weight loss and the corrosion penetration rate increase with immersion time for all the specimens (That is, both the uninhibited samples and the inhibited specimens). But on comparing the amount of weight lost by the various samples, the uninhibited specimens recorded the highest weight loss and corrosion penetration rate more than any other specimen, while the amount of weight loss and corrosion penetration recorded by the inhibited samples generally decreased with increasing in inhibitor's concentration. Thus, the increase in weight loss and corrosion penetration rate noticed can be attributed to the increased time of immersion in the aggressive/unfavourable environment, making the steel to be ''wet'' for a prolonged time. This finding is in accordance with the works of Sayed et al. He observed a gradual increase in weight loss with increase in immersion time of low carbon steel in aerated 4.0M H_2S0_4 sulphuric acid solutions, and that the weight loss decreased with increase in inhibitor's concentration [27]

This result is also in accordance with the investigation of Habibat et al. He exposed the specimens for 5 days (120hrs) while taking reading every 20hrs and observed an increase in weight loss and hence increased corrosion rate with immersion time. From the analysis of his results the uninhibited samples recorded the highest weight loss [28].

Furthermore, Figure 3 shows the plot of the inhibition efficiency (%) against time (hrs) while Figure 4 shows the plot of the degree of surface coverage with time (hrs); from these results obtained it can be seen that both the inhibition efficiency and the degree of surface coverage both increased as time is appreciably increased for all levels of inhibitor concentration. And then, a decrease in value was observed after some days depending on the concentration of the inhibitor. This behaviour could possibly be due to the fact that the extract of the imperrata cylindrica was not extracted before dosing it in the aggressive solution!

Also, the Figures 5-8 shows the effect of the Imperrata cylindrica concentration on the measured parameters. Figure 5 shows the variation of weight loss (mg/cm^2) with and without various concentrations of imperrata cylindrica as an inhibitor, while, and Figure 7 shows the variation of the corrosion rate (mg/cm^2/yr) against the concentration of the imperrata cylindrica respectively. The slope of each line (weight loss per unit time; mg/cm^{2}h) of Figure 5 represents the corrosion rate of the mild steel at the specified conditions. From these data, the weight loss decreased with increasing concentration of the imperrata cylindrica. That is, the corrosion rate of the mild steel in 1.0 M solution of sulphuric acid was retarded by increasing concentration of the imperrata cylindrica. This is clearly seen from the decrease in weight loss, corresponding to a noticeable decrease in the slope of each line (mg/cm^2/hr), and in the corrosion penetration rate with an increase in imperrata cylindrica concentration. This finding is in agreement with Sayed et al. [28]. He exposed the specimens to the respective environments for a period of 12hrs and readings obtained at intervals of 2 hrs and discovered that the corrosion of low carbon steel in aerated 4.0 M sulphuric acid solution was inhibited by increasing concentration of the inhibitor. According to Chikkur and Kikkeri [21], "The corrosion rate and hence weight loss decreased on increasing the inhibitor's concentration. This behaviour could be attributed to the increases in adsorption of the inhibitor at the metal/solution interface and the reduction in the surface area that is available for direct attack on the metal surface. When compared, the decrease in weight loss and corrosion penetration observed decreases with increase in inhibitor (imperrata cylindrica) concentration. Similarly, Figure 6 shows the plot of inhibitor efficiency against the concentration of the imperrata cylindrica as an inhibitor while Figure 8 shows the plot of the degree of surface coverage against the imperrata cylindrica concentration. The figures reveal that both the inhibition efficiency of imperrata cylindrica and the degree of surface coverage increased as the concentration of the inhibitor (imperrata cylindrica) is increased reaching a maximum of 76.58% and 0.7658 at 3.0g of imperrata cylindrica after 4 days, and decreased with decreasing concentration of the inhibitor (imperrata cylindrica).

Thus, a higher coverage of the inhibitor on the mild steel surface was obtained in solutions with higher inhibitor (imperrata cylindrica) concentration. The formation of surface inhibitor film on the mild steel surface provides consideration protection to the mild steel against corrosion. This film reduces the active surface area exposed to the corrosive medium and delays the hydrogen evolution and iron dissolution. The observed phenomenon is generally described as the corrosion inhibition of the metal with the formation of a protective layer of adsorbed species at the metal surface.

This trend may result from the fact that adsorption and surface coverage increases with the increase in Adenine concentration. Thus, the surface is separated from the media [21]. ''The increase in inhibition efficiency with increase in concentration of the inhibitor may be attributed to the increase in surface coverage (Θ) by the adsorption of the inhibitor on to the steel surface'' [31]. It is pertinent and worth noting from Figures 6 and 8 that an optimum inhibition efficiency and degree of surface coverage of 76.58% and 0.7658 respectively were reached after 4 days at 3.0g imperrata cylindrica concentration beyond which there is a decrease in inhibition efficiency and degree of surface coverage within the range of the inhibitor's concentration investigated in this present research.. This observation may possibly be due to the inhibition efficiency reaching a saturation point after 3.0g concentration of the inhibitor [31]. It could also be attributed to the competitive adsorption effect between inhibitor molecules and the metal surface which is already covered with initial layers of the inhibitors molecules [30] leading to possible desorption.

When compared, the increase in inhibition efficiency and degree of surface coverage observed increased with increase in inhibitor (imperrata cylindrica) concentration.

4. Conclusion

From the foregoing, it can be concluded that:
Imperrata cylindrica is safe and eco-friendly inhibitor since it poses no threat to life and the environment. It is readily available, readily accessible and sufficiently available. Imperrata cylindrica is a suitable corrosion inhibitor of mild steel in the stated aggressive solution especially during the processes of pickling, acid descaling, industrial acid cleaning and oil well acidizing. Also, both the immersion time and the inhibitor concentration influenced the inhibiting potential of imperrata cylindrica.

References

[1] Onyekpe, B.O. (2002). Corrosion in oil and Gas Production Ambik Press, Benin City, Nigeria.
[2] Ijeoma, M.N.C. (1991). Elements of Corrosion and Protection Theory. Auto Century Publication Co. Ltd, Enugu, Nigeria.
[3] Wranglen, G. (1972) ‘‘An Introduction to Corrosion and Protection of Metals’’, Halsted Press. A Division of John Wiley & Sons Inc. New York.
[4] Dillon, C.P. (1994). Corrosion Control in the Chemical process Industries (2nd Ed.), MTI
[5] Schorr, M., Valdez, B., Stoytcheva, M., and Zlatev, R. (2008). Kinetics and Structure aspects of the dissolution of metals in acid. ECS Tran., 13 (27), 143-150.
[6] Sherif, E.M., and Park, S.M. (2006). Inhibition of copper corrosion in acidic pickling solutions by N-phenyl-1,4 phenylenediamine. Electrochem. Acta, 51, 4665-4673.
[7] Mattson, E., and Bockris, J.O.M. (1959). Galvanostatic studies of the kinetic of deposition and dissolution in the Copper + Copper sulphate system. Trans. Faradey Soc., 55, 1586-1601.
[8] Sastri, V.S. (1998). Corrosion Inhibitors: Principles and Application. John Wiley and Sons Ltd. New York, NY.
[9] Abdulrahman, A.S., Kobe, I.H., Aigbodion, V.S., Ganiya, K.A.,and Awe, I.C. (2014). Thermodynamic Behaviour of Mild Steel Corrosion in an Environment of Bilberry Cactus Plant Extracts in Hydrochloric Acid. Journal of Metallurgy and Materials Engineering, Vol.9(2): 1-14.
[10] Osarolube, E., Owate, I.O., and Oforka, N.C. (2008). Corrosion Behaviour of Mild and High Carbon Steels in Various Acidic Media. Scientific Research and Essays, Vol.3(6):224-228.
[11] Finsgar, M., and Jackson, J. (2014). Application of Corrosion Inhibitors for Steels in Acidic Media for the Oil and Gas Industry: A Review. Corrosion Science, vol.86:17-41.
[12] Loto, C.A., and Loto, R.T. (2016). Corrosion Inhibition Effect of Allium Cepa Exytracts on Mild Steel in H_2SO_4. Der Pharma Chemica, vol.8(20): 63-73.
[13] Loto, C.A., Loto, R.T., and Joseph, O.O. (2017). Effect of Benzamide on the Corrosion Inhibition of Mild Steel in Sulphuric Acid. South African Journal of Chemistry, Vol.70: 38-43.
[14] Loto, C.A., Loto, R.T.,Osugbunu, O., and Popoola, A.P. (2016). Corrosion Inhibition Effect of Allium Cepa Extracts on Mild Steel in HCl. Der Pharma Chemica, 2016, vol.8(20): 20-30.
[15] Fang, J. And Li, J.(2002). Quantum Chemistry Study on the Relationship between Molecular Structure and Corrosion Inhibition Efficiency of Amides. Journal of Molecular: THEOCHEM, 593: 179-185.
[16] Murthy, Z.V.P, and Vijayaragavan, K. (2014). Mild Steel Corrosion Inhibition by Acid Extracts of Leaves of Hibiscus Sabdariffa as a Green Corrosion Inhibitor and Sorption Behaviour. Green Chemistry Letters and Reviews, vol.7(3): 209-219.
[17] Loto, R.T. (2013). Corrosion Inhibition of Mild Steel in Acidic Media by Butyl Alcohol. Research on Chemical Intermediates.
[18] Subir, P. And Ishita, K. (2016). Corrosion Inhibition of Carbon Steel in Acidic Environment by Papaya Seed as Green Inhibitor. J. Bio Tribo Corros, vol.2: 6.
[19] Loto, R.T., and Loto, C.A. (2016). Corrosion Inhibition Properties of the Combined Admixture of Thiocarbanilide and Hexadecyltrimethylammoniumbromide on Mild Steel in Dilute Acid Solutions. Cogent Chemistry, vol.2: 1268377.
[20] Amitha Rani, B.E., and Bai J. Basu, (2012) ‘‘Green Inhibitors for Corrosion Protection of Metals and Alloys: An Overview’’, International Journal of Corrosion, Vol. 2012, Article 10 380217.
[21] Chikkur B. Pradeep Kumar and Kikkeri N. Mohana, (2013) ‘‘The Effect of Achyranthes aspera Extracts on Mild Steel Corrosion in Industrial Water Medium,’’ ISRN Corrosion, vol. 2013.
[22] Pradeep Kumar and Mohana. Adsorption and Thermodynamic Characteristics of Plumeria rubra Plant Extracts on Mild Steel Corrosion in Industrial Water Medium.(2013) International Research Journal of Pure and Applied Chemistry. 3(4): 330-346
[23] Neha Patni, Shruti, and Pallav Shah (2013) Greener Approach towards Corrosion Inhibition. Chinese Journal of Engineering.
[24] Soltani N, Tavakkoli N, Khayatkashani M, Jalali MR, Mosavizade A 2012. Green Approach to Corrosion inhibition of 304 stainless steel in hydrochloric acid solution by the extract of Salvia officinalis leaves. Corrs Sci. 2012;62(1): 122-135.
[25] Fredy and Karika (2015).Electrochemical and optical microscopy study of red pepper seed oil corrosion inhibition by self-assembled monolayers (SAM) on 304 SS
[26] Parvathy N.G, Padma.R, Renjith.V, Kalpana. P. Rahate, Saranya T.S. (2012). Phytochemical Screening and Anthelmintic Activity Of Methanolic Extract Of Imperata Cylindrica. International Journal of Pharmacy and Pharmaceutical Sciences. Vol 4, Suppl 1.
[27] Sayed S. Abdel Rehim, Omar A. Hazzaxi, Mohammed A. Amin, Khaled F. Khaled (2008) ‘‘On the Corrosion Inhibition of Low Carbon Steel in Concentrated Sulphuric Acid Solution. Part 1: of Electrochemical Science, 6 (2011) 4593-4613
[28] Habibat Momoh-Yahaya, Nnabuk O.Eddy, Johnson F. Iyun, Casmir E. Gimba and Emeka E. Oguzie (2013) ‘‘Experimental Investigation of the Inhibiting Action of Adenine on the Corrosion of Mild Steel in Acidic Environments’’, Journal of materials Science Research; Vol.2, No. 1; 2013. ISSN 1927-0585. Canadian Centre of Science and Education.
[29] Chitra S., Parameswari K., Vidhya M., Kalishwari M and Selvaraj A., (2011) ‘‘Evaluaton of Quinoxalines as Corrosion Inhibitors for Mild Steel in Acid Environment’’, International Journal.