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Effect of *Azadirachta Indica* (Neem) Leaf Extract on the Corrosion of Medium Carbon Steel in Sulphuric Acid

Alexander Nwachukwu Okpala, Ezenwa Alfred Ogbonnaya, Yusuf Bamidele Waidi

Department of Mechanical Engineering, Niger Delta University, Amasoma, Nigeria

Email address:
okpalana@gmail.com (A. N. Okpala), ezenwaogbonnaya@yahoo.com (E. A. Ogbonnaya), yusufwaidi@yahoo.com (Y. B. Waidi)

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Abstract: Using gravimetric technique, the inhibitory properties of Azadiracta Indica leaf extract for medium carbon steel corrosion were investigated, and inhibition efficiency was calculated. The leaves were gathered and dried before being pulverized to micron size for gravimetric analysis. The leaves were extracted using the reflux ethanol extraction method, and the weight of the leaf removed was calculated. The quantities for various concentrations of the extract were also calculated and applied to the acidic medium, where the medium carbon steel coupons were dipped at four hours intervals for a total of 20 hours. The inhibition efficiency was calculated using the weight loss values for each metal coupon. Weight loss (from 0.1395g to 0.0135g) and corrosion rate (from 0.9700 mm/yr. to 0.1878 mm/yr.) both decreased as the content of the plant extract increased (from 0.0g/L to 0.5g/L). The gravimetric method revealed that Azadiracta Indica leaves extract is an excellent medium carbon steel corrosion inhibitor, with inhibition efficiency increasing from 41 percent to 86 percent as the inhibitor concentration increased. Plant extracts are an excellent corrosion inhibitor due to their availability, biodegradability, low cost, and lack of toxicity to humans and the environment. This study found that Azadiracta Indica leaf extracts, when used in the proper concentration, can extend the service life of medium `carbon steel.

Keywords: Corrosion, Passivation, Reflux, Gravimetric, Inhibition

1. Introduction

The American Society of Testing and Materials (ASTM) International defines corrosion as a chemical or electrochemical reaction between a material, usually a metal, and its environment that causes the material and its qualities to deteriorate [2]. The International Organization for Standardization (ISO) also defines corrosion as the physiochemical interaction between a metal and its environment that causes changes in the metal's characteristics and may result in considerable deterioration of the metal's, environments, or technical system's function [8]. Metal atoms can be found in chemical compounds in nature, and corrosion can occur owing to electrolyte variations. Soil resistivity, oxygen concentrations, moisture content, and different ion concentrations are examples of such variances [15].

Microbial corrosion is thought to be responsible for 40% of all internal pipeline corrosion in the gas and oil industry. The application of a protective surface coating is one of the strategies for protecting pipelines conveying fluids (petroleum products and water) against corrosion. This is a costly technique, and coating a pipeline that is already rusted and full of petroleum product is impossible. Application of corrosion inhibitors is one of the most efficient and cost-effective ways to deal with this problem. As a result, finding an effective corrosion inhibitor is a pressing and immediate need [13].

Corrosion control of metals has been of great importance, especially in the production industry [16]. Corrosion inhibitors have historically been regarded the primary line of defense against internal corrosion in the oil and gas production and processing industries. Corrosion can cause plant infrastructure and equipment breakdowns, which are usually expensive to fix, both financially and in terms of lost or polluted products, environmental harm, and perhaps human safety. A precise assessment of the conditions impacting a structure's corrosion and rate of degradation is required to make decisions about the structure's or its components' future integrity. With this information, you may make an informed judgment on the nature, cost, and urgency of prospective remedial measures [5].

The use of corrosion inhibitors is the most practical strategy utilized in industry and academic studies to protect metals and their alloys against strong environmental attacks. The majority of commercial corrosion inhibitors used in industries like oil and gas are multi-component inhibitor systems with nitrogen and sulphur functions. These inhibitors are stable and effective in corrosive conditions, but they can be costly to make and, because of their toxicity, they can constitute a threat to public health and the environment. Multi-component inhibitor systems are also persistent and need costly techniques to remove because they are accompanied by additional heavy metals, chromates, nitrites, and phosphates [1].

Inorganic inhibitors like sodium arsenite ($NaAsO_2$) and sodium ferrocyanide ($Na_4Fe(CN)_6$) were used in the beginning to prevent carbon dioxide (CO_2) corrosion in oil wells, but because of the frequency and effectiveness of the treatments, many organic chemical formulations with film-forming amines and their salts were developed [6]. These corrosion inhibitors were created due to their efficiency across a wide temperature range, compatibility with protected materials, good water solubility, low prices, and low toxicity [2]. Organic corrosion inhibitors adsorb on the surface, forming a protective coating that repels water and prevents corrosion.

In the chemical, automobile, and transportation industries metallic degradation is one of the main factors influencing the reliability of the systems. For instance, in the oil, gas, and petrochemical concerns, thousands of kilometers of pipelines, pumps, pressure, and storage vessels are used to process, store, and transport products. These infrastructures are not only critical to the survival of these industries but also indirectly to the economy of a nation. However, because a large majority of these installations with their components are made of steel alloys and aluminium alloys they are certainly susceptible to corrosion or degradation. In most cases, these failures may result in product spillage which is invariably harmful to society as it represents a risk to human safety, hazard to the environment, and substantial loss of production, time and money. It is also bad publicity for such concerns as compensation and litigation may be involved. For these reasons, a lot of attention is paid to monitoring and inspection of these facilities. However, the period or duration at which these components are inspected can be prolonged or eliminated by incorporating sound corrosion protection techniques. Moreover, these techniques will reduce corrosion rate and by extension prolong inspection or monitoring time thereby reducing the cost of operation.

Corrosion can be reduced by infusing oil-soluble, water-dispersible, and filming amine-type corrosion inhibitors into transportation pipelines that can sufficiently disperse through stratified water layers. Many inhibitor misapplications occur because the features of the inhibitors are not examined prior to usage, and this has a significant impact on microbial corrosion. Bacteria also destroy the inhibitors, reducing their effectiveness and increasing corrosion. Organic compounds employed as typical oilfield corrosion inhibitors generate a film or protective barrier between the metal and the corrosive fluids due to their anodic, cathodic, or mixed-type action. Amino groups including imidazolines, amidoamines, and polythiols compounds are utilized as corrosion inhibitors in the petroleum sector. Aliphatic fatty-acid derivatives, imidazolines, quaternaries, and rosin derivatives are the four primary types, all of which contain long-chain hydrocarbons (C_{18}). However, no dual inhibitor exists that is both inhibitory and biocidal. As a result, creating a multifunctional inhibitor for a tropical pipeline is a great idea right now [13].

Corrosion is a significant issue. Corrosion is significant in three areas: economics, increased safety, and resource preservation. A leak of hazardous materials from a transport pipeline not only results in the loss of natural resources, but it also poses a major and dangerous environmental threat, as well as the risk of human casualties. While pipes are intended and built to retain their integrity, a variety of factors (such as corrosion) make it difficult to avoid leaks in a pipeline system over time [9].

Corrosion is an electrochemical reaction in which a current leaves a structure at the anode site, passes through an electrolyte, and returns to the cathode site. Differential potentials begin to emerge at various locations along the pipe. The current would exit the pipeline at that anode site, pass through the soil, and re-enter the pipeline at a cathode site, for example, because it is in soil with low resistivity relative to the remainder of the line. These potentials cause corrosion currents to exit the pipe and enter the earth at specific points.

Steel Pipelines serve a critical role in transferring gases and liquids across great distances from their sources to their eventual customers all over the world. There is currently roughly 460,000 km (285,000 miles) of common carrier pipelines in use in the United States, transporting about 46% of all crude oil and refined products moved in the country. In 1984, the United States had more than 1.6 x 10^6 km (10^6 miles) of natural gas pipelines in service, with about a quarter of them being interstate. There were roughly 2,800,000 km (174,000 miles) of liquid-transporting pipelines in interstate operation in the United States at the time. In 1984, the United States had around 1.6 x 10^6 km (10^6 miles) of natural gas pipelines in service, with over 25% of them being interstate. At the time, the United States had approximately 2,800,000 km (174,000 miles) of interstate pipeline carrying liquids. Pipelines are being built at a faster rate than ever before. In 1986, 9800 kilometers (6090 miles) of natural gas pipeline, 5740 kilometers (3567 miles) of crude oil pipelines, and 2660 kilometers (1652 miles) of refined products pipelines were built in the United States. The Indian Oil Corporation's cross-country crude oil and product pipeline network, which spans more than 9000 kilometers, fulfills the country's critical energy demands.

Massive sums of money are spent every year on corrosion damage prevention, monitoring, inspection, and repair. Internal corrosion control procedures frequently employ chemical treatment with corrosion inhibitors. There are oil-soluble, water-dispersible, water-soluble, limited-solubility, and volatile corrosion inhibitors on the market, each of which performs differently in different pipeline conditions. They can be applied in batches, and a strong protective film's long-lasting quality can last weeks or months.

Alternatively, they can be constantly metered into the pipeline in low concentrations using a continuous injection programmer, which lays down and maintains a thin film over time. The inhibitors will operate successfully as long as they can reach the pipe's wall and produce an effective film. When debris, corrosion products, and bacteria build up inside a pipe, they can consume inhibitors meant to cure the pipe's walls while simultaneously preventing the inhibitors from reaching the pipe's walls behind the deposits. As a result, when corrosion inhibitors are applied, pipelines should be as clean as possible [13].

Over the last decade, green chemistry has emphasized the importance of environmental preservation and human health in a cost-effective manner, with an emphasis on toxin avoidance, waste minimization, and the avoidance of hazardous or risky chemical use [3]. Natural materials were discovered to be safer with the development in environmental awareness [18]. Plant extracts of peels, seeds, stems, and leaves are commonly used not only because they are safe, but also because they are thought to have a lower economic cost than organic inhibitors [10].

Green corrosion inhibitors can achieve both anodic and cathodic activity by reducing electrochemical rates. The inhibitory process is usually associated to the adsorption of double and triple linked molecules with single electron pairs that can participate in and bond to the metal surface, such as nitrogen, oxygen, sulphur, and phosphorous components. At the metal's point of attachment, the electron density changes, delaying cathodic or anodic interactions [7].

Metals and their alloys are crucial in the food processing, manufacturing, and oil and gas industries. The importance of metals cannot be overstated, yet corrosion is a problem that continues to threaten their survival. As a result, the goal of this research is to locate a suitable plant extract that will minimize, slow down, or prevent metal corrosion.

2. Review of Related Work

Other alloys can't compete with steel and alloys' versatility as a construction material. It is the most frequently used construction material, with approximately 1.4 billion tons produced each year [17]. It is an important component of daily life because it connects all sectors, including housing, electricity, agriculture, and water supply. Structures, chemical plants, equipment, machinery, tools, transportation pipelines, pressure vessels, and storage tanks are only a few of its uses in our daily lives. As a result, steel is a major economic engine around the world [17]. Steel production will continue to rise, table 1 below shows world steel production in 2014 [11]. Developing regions such as Africa, Asia, and Latin America need to increase steel production, where steel will be critical in raising living standards. It is expected that sixty percent of the steel used in these areas will be for new infrastructure. The reprocessing of end-of-life of steel products and the conversion of fresh ore to steel will not be able to meet demand with this constant but ongoing expansion, as a result, appropriate corrosion protection and control procedures are required to maintain and extend the life of steel that is already in service.

Table 1. *World steel production in 2014.*

Country	Production 2014 (metric tons)*	% of world production
China	820,000,000	49.7
Japan	111,000,000	6.7
United States	88,000,000	5.3
India	83,000,000	5.0
Russia	71,000,000	4.3
South Korea	65,000,000	3.9
Germany	44,000,000	2.7
Brazil	34,000,000	2.1
Ukraine	26,000,000	1.6
France	17,000,000	1.0
United Kingdom	12,000,000	0.7
other countries	274,000,000	16.6
world total	1,650,000,000	100**

Source: [11].

Green corrosion inhibitors are substances when added to the process fluid, can slow down the corrosion reactions. Sometimes their inhibition mechanism is not straight to interpretation and study. The inhibition effect can be anodic, cathodic, or mixed. It is also worth specifying that some inhibitors are strictly specific to the metal or alloy and environment, and might not show any inhibition phenomenon with others. Anodic inhibitors usually act by forming a protective oxide film on the surface of the metal, cathodic inhibitors act by either slowing the cathodic reaction itself or selectively precipitating on cathodic areas to limit the diffusion of reducing species to the surface.

Most of the green corrosion inhibitors belong to the mixed class. Mixed-type inhibitors can accomplish a cathodic and anodic action at the same time by decreasing both their electrochemical rates. The mechanism of inhibition is often related to the adsorption of molecules with double/triple bonds or containing V and VI group elements such as N, P, S, O, which contain free-electron couples. The determination of the “class” is quite easy to be carried out. Other insights are instead necessary for the determination of the mechanisms, and this is the main lack in the literature.

The effects of roselle leaf (hibiscus sabdariffa (AELHS)) extraction and synthesis on mild steel corrosion in acidic baths (1.2 N HCl and 1.2 N H_2SO_4) were investigated using gravimetric techniques. The findings show that this chemical is more effective at preventing mild steel corrosion in 1.2 N H_2SO_4 than in 1.2 N HCl. The data is evaluated using the Langmuir, Frumkin, Florry–Huggins, and Langmuir–Freundlich isotherms; among these, the Langmuir isotherm fits the data well, with a correlation coefficient of > 0.99 in both acid settings [12].

AELHS is an effective medium carbon steel corrosion inhibitor, according to the findings. The four adsorption isotherms studied for the data were Langmuir, Frumkin, Florry–Huggins, and Langmuir–Freundlich. In both acid (HCl), (H_2SO_4), and alkaline conditions, the Langmuir isotherm matches the data well, with a correlation coefficient of over 0.99. The type of acid, the length of time, and the concentration of the acid all play a role in the inhibitor's inhibitory efficiency. In the presence of H_2SO_4, inhibitor adsorption on mild steel is more spontaneous than in the presence of hydrochloric acid, resulting in better inhibitor inhibitory characteristics in the H_2SO_4 environment. Overall, the AELHS inhibitor appears to perform significantly better in an H_2SO_4 environment than in a hydrochloric acid environment. [12].

El-Haddad [4] published a paper evaluating the corrosion inhibition effect of chitosan, purchased by Sigma Aldrich, onto copper surfaces, hence excluding any parallel effect due to other compounds in the used solution. Their results were more trustable in terms of electrochemical features and they classify the molecule as a mixed-type inhibitor acting preferably on the cathodic sites. Still, they used a Langmuir isotherm model to fit data, but they added a deeper computational study on the HOMO-LUMO orbitals and their hypothesis on the inhibition mechanism is more reliable due to the exact knowledge of the involved molecules, the only one that can play a role in the final inhibition effect.

3. Materials and Method

3.1. Azadirachta Indica Leaf Preparations

In November of 2021, *Azadirachta Indica* Leaves were harvested in Aba, Abia State, Nigeria's eastern area. The leaves were air - dried at room for 30 days at room temperature in the laboratory before being used to make the extract. The extraction was carried out using the reflux technique for 3 hours at steady heat (70°C) with ethanol as the extraction solvent. According to Nnanna et al., [14] the extract is then diluted into various concentrations: 0.1, 0.2, 0.3, 0.4, and 0.5 g/L using 1M H_2SO_4 solution as the corrosive media.

3.2. Metal Preparations

The experiment was carried out with medium carbon steel (C-1345) sample of chemical composition (C = 0.48%, Mn = 2.53%, Si = 0.64%, Cr = 0.46%, Ti = 0.15%, Co = 0.36%, Cu = 0.07%, Mo = 0.68% and Fe = 95.7%). The metal sheets were cut into coupons of dimension (20 x 20 x 4mm) were abraded with diverse grades of emery paper (120, 600 and 1200) and washed with detergent, degreased in ethanol and air dried before weighing.

3.3. Gravimetric Method

The pre-weighed coupons were exposed to diverse test solutions. The study was conducted with the diverse concentrations (0.1g/L to 0.5g/L) of the inhibitor (*Azadirachta Indica* Leave extracts) at room temperature. After the exposure period of between 4hrs to 20hrs, the coupons were retrieved, rinsed with plenty of water, dried and thereafter weighed. The weight loss was decided. The procedure repeated and the average result taken.

4. Results and Discussion

This study was carried out in order to identify a suitable substitute for some of the currently used inorganic and synthetic organic corrosion inhibitors, which are damaging to both humans and the environment. Potassium, magnesium, lead, chromates, and inhibitors containing a mixture of deadly anions (nitrates, phosphates, and fluorides) are no longer suitable or appropriate. As a result, a great number of inhibitors are currently utilized to protect metal surfaces from corrosion. Instead, eco-friendly inhibitors should be used. Unfortunately, there is a scarcity of knowledge about corrosion inhibitors that are safe for both humans and the environment. As a result of this research, corrosion inhibitor formulations made from *Azadirachta Indica* plant leaf extracts have been produced for use in sulphuric acid solutions to decrease low carbon steel corrosion.

Below are the results of the gravimetric experiment.

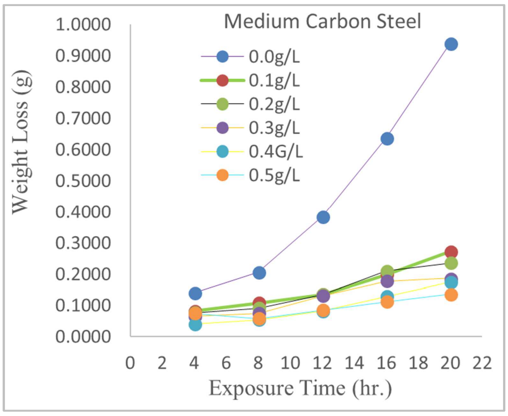

Figure 1. *Plot of weight loss versus Exposure time.*

Table 2. *Weight loss measurements for medium carbon steel in 1 M sulphuric acid in the absence and presence of Azadirachta Indica leaves extract.*

Ex Exposure	Weight loss at Different Concentration					
Time (Hrs)	Control (0.0g/L)	0.1g/L	0.2g/L	0.3g/L	0.4g/L	0.5g/L
4	0.1395	0.0821	0.0767	0.0659	0.0413	0.0739
8	0.2069	0.1076	0.091	0.0746	0.0536	0.0578
12	0.3843	0.1345	0.1346	0.1305	0.0816	0.0852
16	0.6372	0.1976	0.2104	0.1782	0.1278	0.1113
20	0.9386	0.2722	0.2347	0.1871	0.1747	0.135

Table 2 Illustrate the weight loss measurements for medium carbon steel as it degrades in 1M sulphuric acid in the absent of inhibitor and the medium carbon steel resisted to corrosion in the presence of inhibitor (*Azadirachta Indica* leaves extracts).

Weight loss versus exposure time for medium carbon steel corrosion in 1.0M H_2SO_4 in the absence and presence of various amounts of AZI leaf extract is shown in Figure 1.

The rate of weight loss in the controlled environment (1.0M H_2SO_4) was gradually increasing over time, as shown in the plots, and this was observed for 20 hours, but when the inhibitor leaf extracts were introduced, the rate of weight loss decreased significantly according to the different inhibitor concentrations. The adsorption of elements in the extract on the surface of the medium carbon steel test piece caused the weight loss to be reduced in the presence of the inhibitor.

Table 3. *Corrosion rate values for medium carbon steel in 1 M sulphuric acid in the absence and presence of Azadirachta Indica leaves extract.*

Time (t) of exposure (Hrs)	Corrosion rates for control experiment and different concentration of AZI s leaf extraction in corrosive media.					
	Control (0.0g/L)	0.1g/L	0.2g/L	0.3g/L	0.4g/L	0.5g/L
4	0.9700	0.5712	0.5336	0.4585	0.2873	0.5141
8	0.7200	0.3743	0.3165	0.2595	0.1864	0.2011
1 12	0.8900	0.3119	0.3121	0.3026	0.1892	0.1976
16	1.1100	0.3437	0.3659	0.3099	0.2223	0.1936
20	1.3100	0.3787	0.3266	0.2603	0.2431	0.1878

Table 3 shows the corrosion rate values when there was no inhibition to be increasing as exposure time increases, but when leaf extract was added there was noteworthy drop of corrosion rate.

The corrosion rate was calculated using eq. (2).

$$\Delta W = W_1 - W_2 \quad (1)$$

$$CR = \frac{K\Delta W}{At\rho} \quad (2)$$

Where K = Rate constant equal 8.78×10^4

W_1 = Weight before immersion in g

W_2 = Weight after immersion in g

ΔW = Weight loss in g

ρ = Density of material in g/cm^3

T = Exposure time in hours

A = Exposed area of coupon cm^2

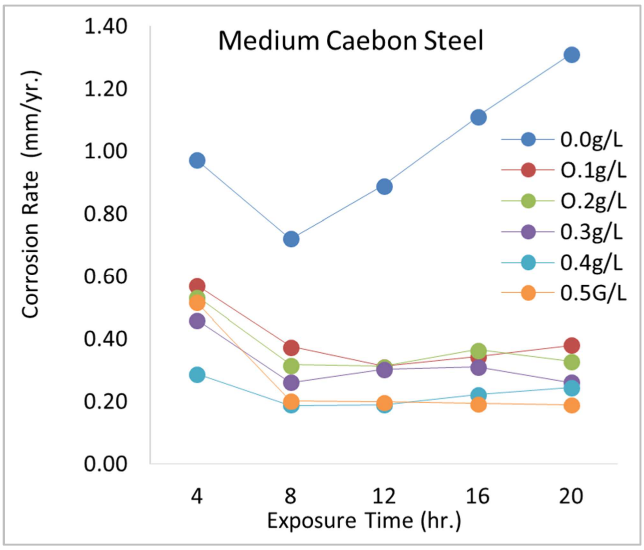

Figure 2. *Plot of Corrosion rate (mm/yr) (for control + various concentrations) versus exposure time (t).*

Figure 2 depicts the phenomenon where, in the absence of AZI leaf extract (control experiment), a behavioural pattern of medium carbon steel corrosion rate with time was found. Passivation occurred within the first four hours of metal exposure, followed by metal resistance to passivation, as seen by the control curve. The creation of coatings on the test piece's surface caused the corrosion resistance observed between 4 and 8 hours; this does not mean that corrosion has halted; merely that it has been postponed. The films succumbed to further deterioration after 8 hours due to the environment's severe corrosive nature. The corrosion rate values in the control experiment continued to grow after 8 hours with no signs of slowing down. Medium carbon steel degrades quickly, according to the findings. The inhibited corrosion curves for all concentrations of 0.1 to 0.5g/L showed a consistent pattern of the corrosion rate dropping steadily from 0 to 20 hours, demonstrating that the leaf has formed a protective layer on the test piece's surface, blocking the active site and thus preventing corrosion. The corrosion rate continued to reduce at 0.1g/L concentration until 16 hours, when a minor rise was noted, indicating that the extract concentration was depleting. From 8 to 20 hours, the 0.5g/L concentration of leaf extracts demonstrated the best corrosion rate values.

Table 4. *Efficiencies (%) at concentrations (g/L) for Gravimetric Method.*

Exposure Time (hr.)	Efficiencies at various concentrations (g/L)				
	0.1	0.2	0.3	0.4	0.5
4	41%	45%	53%	70%	47%
8	48%	56%	64%	74%	72%
12	77%	65%	66%	79%	78%
16	69%	67%	72%	80%	83%
20	71%	75%	80%	81%	86%

Table 4 Elucidates how efficiency increased with an increase in concentrations of inhibitor. The efficiencies was calculated using the formula.

$$\eta_{gravimetric} = (1-\frac{w_i}{w_o})\times100\% \quad (3)$$

Where W_i is the weight of test piece when dipped into corrosive environment with leaf extract and W_o is the weight of test piece when dipped into corrosive media without leaf extract.

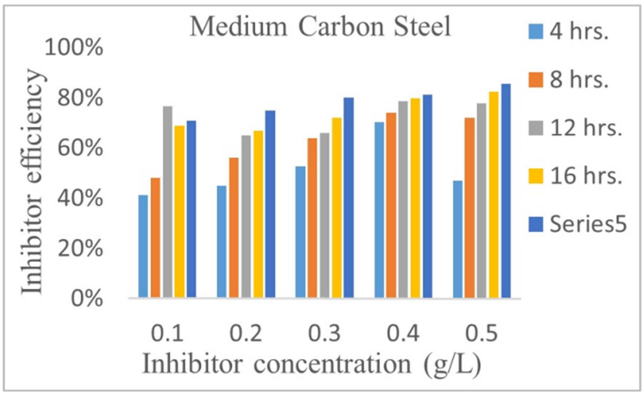

Figure 3. *% Efficiency versus Concentration of inhibitor (g/L).*

Figure 3 shows the inhibition efficiency of the inhibitor plotted against the concentration of the plant extract. It can be seen that *Azadirachta Indica* leaves extract is a good inhibitor of medium carbon steel corrosion in sulphuric acid. The inhibition efficiency increased with increase in inhibitor concentration. It can also be observed from table 2 that the corrosion rate reduced with increase in concentration of the plant extract.

5. Conclusion and Recommendations

5.1. Conclusion

In the light of the evidence gathered during the course of investigating the inhibitive properties of *Azadirachta Indica* Leaf extract on the Corrosion of Medium Carbon Steel in Sulphuric Acid, the following conclusion can be drawn:

i. In 1 M H_2SO_4 solutions, ethanolic extract of *Azadirachta Indica* Leaf is a suitable eco-friendly green inhibitor for medium carbon steel and can be used instead of hazardous chemicals.
ii. In the absence of *Azadirachta Indica* leaves extract in 1M of H_2SO_4 solution, gravimetric method reveals aggressive deterioration of the medium carbon steel coupon immersed in the corrosive environment, the corrosion rate and weight loss increases drastically.
iii. showing that the active site of the test piece was bare to acid attack.
iv. The addition of *Azadirachta Indica* leaf extract to 1M H_2SO_4 solution reduces the corrosion rate of medium carbon steel in the acid as elucidated by the weight loss measurement. The inhibition efficiency increases with increase in concentration of the plant extracts, the maximum inhibition efficiency was 86%.
v. Medium carbon steel was protected from corrosion when the plant extract was introduced by adsorption of compound of the extracts on the metal surface thereby creating a barrier for charge and mass transfer making the metal less susceptible to corrosion reaction.
vi. The findings show that *Azadirachta Indica* leaves extract is a very good corrosion inhibitor for medium carbon steel in sulphuric acid (H_2SO_4).

5.2. Recommendations

In view of the experienced gained on this research, the following are recommended for further research.

i. Further investigation is required to access the corrosion morphology so as to ascertain the active species in the adsorption layer.
ii. The researcher recommends that the need to grow *Azadirachta Indica* trees at commercial be encouraged since they have very high efficiency as inhibitors. Being good inhibitors, they can reduce corrosion rate. Moreover, they are human friendly and ecologically acceptable.
iii. Investigation of the inhibitive properties of *Azadirachta Indica* leaf extracts on medium carbon steel in sodium chloride (NaCl).
iv. To investigate the effect of temperature on *Azadirachta Indica* leaf extracts as a corrosion inhibitor of medium carbon steel in corrosive acid media.

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