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Probing the Inhibitive Potency of Azadirachta Indica (Neem) Leaf on High Carbon Steel Corrosion in 1M Sulphuric Acid (H_2SO_4)

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***Abstract*:- Using gravimetric method in 1.0M sulphuric acid, the inhibitive characteristics of Azadirachta Indica leaf extract for the corrosion of high carbon steel were examined. The leaves were gathered, dried, and powdered to a micron size for gravimetric analysis. Using the ethanol reflux method, the leaf extract was obtained, and the weight of the extracted leaves was calculated. The amounts for various extract concentrations were likewise calculated, added to the acidic medium, and the high carbon steel coupons submerged there for four hours at a time. The inhibitory efficiency was estimated after obtaining the weight loss values for each metal coupon. The weight loss and corrosion rate results showed that Azadirachta indica leaf extract functions as an effective inhibitor, with weight loss from 0.411g to 0.166g and corrosion rate dropping from 2.86mm/yr. to 0.23mm/yr. with an increase in concentration of the plant extract from 0.0g/L to 0.5g/L. Using the approach employed, it was proven that Azadirachta Indica leaf extract is a potent inhibitor of corrosion of high carbon steel, with an increase in inhibition effectiveness with increasing inhibitor concentration. Gravimetric method was used to get inhibition efficiency values ranging from 73% to 89%. Azadirachta Indica Leaf Extract is an excellent alternative to conventional corrosion inhibitors because it is less harmful to human and the environment. This study has shown that Azadirachta indica leaf extract, when administered at the right concentration, can lengthen the service life of high carbon steel.**

Key words: Azadirachta Indica, corrosion. Potency, Efficiency and Inhibition.

1. INTRODUCTION

A natural process known as corrosion is the degradation or destruction of a material's qualities as a result of interaction with its surroundings. Corrosion can cause structural and equipment failures in plants that are typically expensive to repair, result in the loss of tainted products, cause environmental harm, and possibly have a negative impact on human health (Nurul and Jain, 2013). Some people claim that the term should only apply to metals, yet corrosion engineers frequently need to take both metals and non-metals into account. Corrosion is defined as the attack of one molten metal on another molten metal (liquid metal corrosion) and the corrosion of ceramics, rubber, and other nonmetallic materials. It can also occur when paints and rubber deteriorate due to sunlight or chemicals, fluxing, or the lining of the steel-making furnace (Samina et al., 2011).

In many industries, structures, and public utilities like water and sewage supply, metal corrosion is a major issue (Nnanna et al., 2014). It depends on the material being corroded whether the process is quick or gradual. Metal corrosion can be seen as an extractive form of metallurgy. Although many other metals produce their oxides when corrosion takes place, the term "rusting" is only used to refer to steel and iron corrosion (Samina et al., 2011). Corrosion inhibitors are chemical substances that, when added to corrosive media, slow down the corrosion process and maintain its rate to a minimum. They are typically used in tiny concentrations. Among these, organic compounds with heteroatoms like nitrogen, oxygen, sulphur, and phosphorus, electrons in triple or conjugated double bonds, and compounds that are readily adsorbed onto the surface of metal have the capacity to operate as effective corrosion inhibitors (Nurul and Jain, 2013). The use of natural items like essential oils as corrosion inhibitors for metals in acid cleaning operations has become increasingly popular as a means of locating corrosion inhibitors that are both easily available and safe for the environment. An essential oil's ability to prevent corrosion is strongly tied to the phytochemical components of the oil, which include polar and nonpolar hydrophilic and hydrophobic hydrocarbon molecules with one or more functional groups (Andreani et al., 2016).

Despite its propensity to corrode in aqueous solutions, particularly in acidic environments, mild steel—the most important iron alloy—finds widespread use in industries, building materials, and machinery due to its low cost and outstanding mechanical qualities [Nurul and Jain, 2013]. High carbon steels and mild steels are categorized as ferrous metals (they contain a large percentage of iron). Iron-carbon alloys are essentially what carbon steels are. Mild steel plates and rod-sections have been employed for many years as structural members in bridges, buildings, pipelines, heavy vehicles, as welded plate construction for ships and storage vessels, among many other uses. The least ductile of all the carbon steels, high carbon steel is harder and stronger while having a larger carbon content than mild steel. It is primarily employed in the production of metal-cutting instruments such hammers, saws, forging die blocks, axes, knives, and drills.

Azadirachta Indica (AZI) gum is naturally obtained from AZI trees by induced or accidental damage. AZI gum has an amber color and is transparent, brilliant, and flavorless. It is also soluble in cold water. It is a byproduct produced by the metabolic processes of plants and trees. AZI has been utilized for commercial purposes in order to exploit its gum, which is utilized in numerous sectors. AZI cake is a byproduct of the AZI oil business and is used as fertilizer, insecticide, and feed for cattle. Making soap frequently involves the use of AZI oil. The popularity of medicated AZI soaps is rising. In Europe and India, toothpaste with a AZI base is very popular. Numerous dental care and oral hygiene products are made from AZI. Gum and tannins from AZI bark are used in tanning, dyeing, and other processes. In fermentation industries and for the production of methane gas, AZI seed pulp is employed as a rich source of carbohydrates (Baran et al., 2019; Gualdrón et al., 2013). The primary goal of this work is to use the gravimetric method to examine the high carbon steel corrosion-inhibiting characteristics of AZI in acidic condition.

High carbon steel, which has a carbon content ranging from 0.6 to 1.4 percent, is the strongest type of steel. Even though cast iron is only mentioned when it contains more than (0.2-4.5%) percent carbon, it is utilized to manufacture a variety of cutting tools. It is crucial to keep in mind that a rise in carbon may, in certain circumstances, cause steel corrosion to increase given that the resistance of the metal to corrosion varies when it is heat-treated (Dwivedi et al., 2017).

1.1 Corrosion inhibitors from Plant Extract

The progressive degradation of metals and alloys brought on by the action of air gases, moisture, and other chemicals is known as corrosion. The life of metallic and alloy materials is prolonged by the rate at which a corrosion inhibitor spreads in a small volume of water, hence increasing the life of the metal exposed to that water. Plant extracts, also referred to as "green corrosion inhibitors," can be utilized to prevent corrosion in metal. Compounds that are naturally generated by plants are created.

The majority of naturally occurring substances are chosen because they are safe for the environment, cost-effective, and widely accessible. Some not only have chemical, biological, and physical characteristics, but also complicated molecular structures. Corrosive media are supplemented with low-concentration inhibitors to postpone the reaction between the metal and the corrosive elements in the medium.

1.2 Inhibition phenomenon

Plant extracts are utilized to prevent corrosion because their inhibitory effects are related to the adsorption of inhibitor molecules on metal surfaces. Adsorbing organic molecules involves the employment of physical, electrostatic, and chemical methods (Harvey et al., 2018). Lowering the geometric size of the reaction zone on the metal surface is how adsorption inhibitors work. If the rate of activation energy barriers differs for anodic and cathodic reactions, they can also reduce corrosion through the electro-catalytic effect or the products of their reaction (Abd El-Lateef et al., 2019). Physical adsorption is the process by which an increase in temperature and the occurrence of electrical attraction between inhibitor molecules containing electron donor atoms like (O, N, S, and P), heterocyclic rings, and metal atom orbitals cause the adsorption of inhibitor molecules to increase (Faisal et al., 2018).

The adsorption of inhibitor molecules activates the inhibitory mechanism of metal surfaces. The type of metal, its surface, the medium charge, and the chemical composition of the inhibitor all have an impact on the adsorption phenomenon (Vinutha et al., 2016). As a result, the creation of bonds between the orbitals of metal atoms and the sp- electron pairs that are present on the nitrogen and oxygen atoms of heterocyclic rings may be the cause of the adsorption of inhibitor chemicals. When water molecules are forced off of metal surfaces, electrostatic interactions, for instance, between the positively charged nitrogen atom and the negatively charged metal surface may encourage inhibitor adsorption. Some inhibitors' ability to prevent corrosion may occur from either ring removal from particular sub-inhibitors (acidic or alkaline) as a result of the materials' resilience to oxidation (Ryl et al., 2019). At some doses, a modest number of inhibitors is sufficient to prevent their adsorption.

2. REVIEW OF RELATED WORK

The studies linked to various plant extracts as corrosion inhibitors on steels and alloys show the research efforts targeted at creating long-lasting green inhibitors, and they are described below.

AZI is notable for its biological and chemical properties. It is among nature's most productive suppliers of secondary metabolites. More than 300 natural products have been separated from various tree sections to date, and every year, more substances are added to the list. By examining the inhibitory capabilities of AZI specifically for mild steel, aluminum, and tin, the current work seeks to expand the applicability of plant extracts for metallic corrosion inhibition as a contribution to the current interest in green corrosion inhibitors. The potential of AZI extract as a corrosion inhibitor on metal surfaces, particularly those made of carbon steel and its alloys, aluminum, and tin, is covered in the current article. The chemical makeup of AZI, the impact of temperature on inhibitory effectiveness, and computational study linked to AZI adsorption on metals have all been thoroughly studied. The understanding of the adsorption process involved and the subsequent inhibitory effect of plant extract against metal corrosion would be improved by this work (Sanjay et al., 2015).

The efficiency of AZI gum as a mild steel corrosion inhibitor in 1.0M nitric acid (HNO_3), sulphuric acid (H_2SO_4), and hydrochloric acid (HCl) solutions at 298K and 313K was examined using the gravimetric method. At different inhibitor concentrations (0.50, 1.00, 1.50, 2.00, and 2.50% w/v), weight loss was seen. The amount of surface coverage and inhibitor effectiveness were calculated using the weight measurements. In order to analyze the spontaneity and enthalpy of the corrosion process, the Gibbs free energy and heat of adsorption were computed along with the activation energy using the Arrhenius equation. Adsorption isotherms, including Langmuir, Temkin, and Fraudlish, were used to get insight into a potential adsorption mechanism (Abdulmudallib et al., 2018).

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For the preservation and protection of carbon steel-reinforced concrete structures, the construction industry has fervently called for cutting-edge green inhibitory measures. The impact of Azadirachta indica leaf extract as a possible inhibitor of carbon steel corrosion in reinforced concrete under corrosion in saline simulated media was assessed for the first time. Three inorganic commercial inhibitors were tested to compare in accordance with the standards defined by Stratful for half-cell potential in a simulated chloride environment in order to evaluate the corrosion inhibition behavior of the AZI natural organic extract. Additionally, the impact of the AZI treatment on the integrity of the concrete was observed after it underwent changes in temperature, slump, weight, air content, compressive strength, and chloride ion penetration. The findings indicated that neither the physicochemical characteristics nor the concrete integrity were changed by the AZI treatments. After 182 days of testing, we had a 95% enhanced long-term corrosion prevention. Our latest findings provide the building sector a fresh, potential "green" route for conserving carbon steel in reinforced concrete (Benjamin et al., 2021).

This study looked at the ability of two biological components, AZI and an enzyme, to reduce the corrosion of mild steel. The corrosion losses in mild steel exposed to saline solutions with or without inhibitors over a period of 576 days were evaluated using the weight loss analysis method, and the corrosion rates, inhibitors' efficacy, and surface coverage were identified. The findings shown that after 400 days of continuous exposure to the same solution, the rate of mild steel corrosion generally did not change appreciably. When enzyme was added to low and high saline solutions, the corrosion rate of mild steel was dramatically reduced compared to when saline solutions were used alone. Application of the enzyme resulted in high corrosion inhibition efficacy, and its effective concentration was discovered to be 2 wt.% in both low and high saline solutions. However, it was discovered that the corrosion inhibition of AZI was more effective in low saline solution, and in this environment, an optimum concentration of 2 wt.% was effective. However, in high saline solution, a higher concentration of 10 wt.% is required for effective corrosion inhibition (Udoh et al., 2022).

Due to the corrosion and damage, it produces in most industrial processes, especially in the oil and gas industries, there have been considerable losses at high costs for a long time. Since erosion has a catastrophic effect on the environment and poses a major threat to people and animals, it requires long-term solutions. As a result, there is a wealth of literature on the topic of corrosion. Thus, the need for natural or synthetic organic corrosion inhibitors, which are frequently affordable and safe because they biodegrade, as demonstrated by earlier studies. Metals can be protected from corrosion in a variety of corrosive conditions by organic molecules with many heterogeneous atoms and double and triple bonds in their molecular structures. Numerous scholars have published a lot of study each year on the manufacture or extraction of corrosion inhibitors, as well as on the classification and inhibitory mechanisms. The current study compares the various types of extracted or manufactured coumarins used as corrosion inhibitors with one another in terms of their effectiveness in preventing corrosion, as well as the various types of corrosion, inhibitors, mechanisms of action, and measuring corrosion inhibitor efficiency (Kadhim et al., 2021).

Chemical techniques were used to investigate the AZI leaf extracts' capacity to reduce corrosion in sea water. Extracts from AZI prevented mild steel from corroding in sea water. Weight loss, electrochemical impedance spectroscopy (EIS), linear polarization, and potentiodynamic polarization methods have all been used to examine the extract of AZI leaves. The Nyquist plots demonstrated that when the concentration of AZI increased, charge transfer resistance increased and double layer capacitance decreased. As the extract concentration grew, the effectiveness of the inhibition increased as well. AZI's leaf extract was discovered to have a higher 98% inhibitory efficacy (Dr. Sribharathy et al., 2018).

The gasometric method was used to examine how AZI seed extract in 0.5M, 1.0M, and 1.5M H_2SO_4 inhibited corrosion of mild steel and copper. Based on the volume of hydrogen evolved, zero order, half order, and first order models were fitted to the data. These findings demonstrate that a zero order kinetic model may be used to simulate the volume of hydrogen evolved during the corrosion of mild steel and copper in an H_2SO_4 media, and inhibition lowers the instantaneous volume of hydrogen evolved (V_0) and kinetic rate constant (k). The gasometric method's results also showed that solutions using mild steel corroded at a higher rate than solutions using copper (Ekeke et al., 2019).

3. MATERIALS AND METHOD

3.1. *Azadirachta Indica* Leaf Preparations

AZI leaves were gathered at Aba, Abia State, eastern Nigeria, in October 2021. Before being used to create the extract, the leaves were air-dried in the laboratory for 30 days at room temperature. With ethanol serving as the extraction solvent, the extraction was carried out using the reflux technique for 3 hours at constant heat (70 °C). According to prior research, the extract is next diluted into a range of concentrations using 1M H_2SO_4 solution as the corrosive medium: 0.1, 0.2, 0.3, 0.4, and 0.5 g/L (Nnanna et al., 2014).

Table 3.1: Results of quantitative tests carried out on the extract AZI leaf:

Phytochemicals	AZI (mg/g)
Tannin	3.1
Steroid	9.91
Flavonoid	2.28
Saponin	5.81
Terpenoid	15.85
Glycoside	26.78

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According to Table 3.1 below, Glycosides make up the majority of the phytochemical components in AZI leaf extract, with Tannins, Steroids, Flavonoids, Saponin, and Terpenoid, making up the remainder. Tannins, according to Ayeni et al. (2012), act as a barrier to ion diffusion in both cathodic and anodic metal ion processes in solution, reducing the rate of corrosion.

3.2 Metal Preparations

In the experiment, a high carbon steel (HCS) sample of grade (AISI, TS O-1) was used, and its chemical composition was as follows: C is between 0.85 and 1%, Si is 3.94 %, Mn is 1.71 %, Cr is 0.88 %, Ti is 0.29 %, Co is 0.68 %, Cu is 0.07 %, Mo is 0.68 %, and Fe is 91.76 %. To prepare the metal sheets for weighing, they were cut into 20 x 20 x 4 mm coupons, abraded with 120, 600, and 1200 grit emery paper, cleaned with soap and ethanol to get rid of any grease, and then left to dry on the air.

3.3 Weight loss Method

Various test solutions were applied to the pre-weighed coupons. The experiment was done at room temperature and employed AZI leaf extract inhibitor doses ranging from 0.1g/L to 0.5g/L. The test coupons were removed from the test solutions at 2 hours interval for a total immersion period of 20 hours for the various solution. The test pieces after the immersion period were dipped in nitric acid to end the reaction, washed with water, removed the water with ethanol, and then quickly dried with acetone so that corrosion wouldn't commence before it could be reweighed. The weight loss was calculated. The technique was repeated, and an average result was obtained.

4. Results and Discussion

Green inhibitors (plant extracts) have been found to be a good option for corrosion inhibitors due to their availability, biodegradability, low cost, and lack of toxicity to humans and the environment. This study has shown that using AZI leaf extract in the proper concentrations can extend the life of high carbon steel in sulphuric acid conditions.

Below are the results of the weight loss experiment.

Table 4.1: Weight Loss Measurements for High Carbon Steel in 1.0M Sulphuric Acid in the Absence and Presence of AZI Leaf Extract.

S/N	Exposure Time (Hrs)	Control (0.0g/L)	Weight loss (g) at Different Concentration 0.1g/L	0.2g/L	0.3g/L	0.4g/L	0.5g/L
1	4	0.4111	0.2585	0.2464	0.1691	0.1353	0.2218
2	8	0.7012	0.3718	0.2944	0.2538	0.1892	0.232
3	12	0.8367	0.2566	0.2538	0.2833	0.1399	0.2225
4	16	0.9079	0.2544	0.2723	0.1843	0.1832	0.1866
5	20	1.4706	0.3965	0.3394	0.2108	0.2048	0.1662

Table 4.1 displays the weight loss data for high carbon steel during degradation in 1.0 M sulphuric acid both in the presence and absence of an inhibitor (AZI leaf extracts). The table shows that as the extract concentration rises, weight loss decreases.

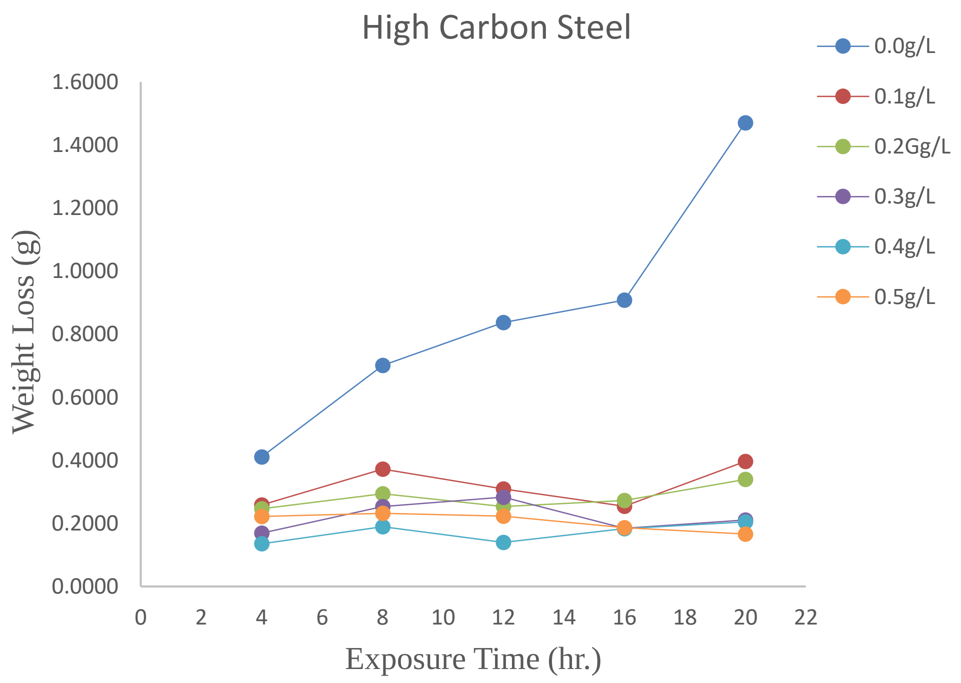

Figure 4.1 : Plot of Weight Loss (g) versus Exposure Time (t) for MCS with AZI leaf extract as inhibitor

The outcomes of the gravimetric experiment using AZI leaf as an inhibitor for HCS are shown in Figure 4.1. The graphs below illustrate the corrosion of HCS in 1.0M H2SO4 in the absence and presence of varying concentrations of AZI leaf extract as a function of weight loss versus exposure time. The rate of weight loss in the controlled environment (1.0M H2SO4) increased over time, though there was a significant loss between 16 and 20 hours, indicating that the HCS will not survive for a long time. Weight loss decreased after administration of the inhibitor leaf extracts and remained stable for around 16 hours before it slightly increased, showing that the inhibitor concentration was dwindling. For information on weight loss and exposure time, see table 4.1 above.

Table 4.2: Corrosion Rate Values for High Carbon Steel in 1.0M Sulphuric Acid in the Absence and Presence of AZI Leaf Extract.

Time (t) of exposure (Hrs)	Corrosion rates (mm/yr.) for control experiment and different concentration (g/L) of AZI leaf extraction in corrosive media.					
	Control (0.0g/)	0.1g/L	0.2g/L	0.3g/L	0.4g/L	0.5g/L
4	2.8599	1.7983	1.7142	1.1764	0.9413	1.5430
8	2.4391	1.2933	1.0240	0.8828	0.6581	0.8070
12	1.9403	0.5950	0.5885	0.6570	0.3244	0.5160
16	1.5790	0.4425	0.4736	0.3205	0.3186	0.3245
20	2.0461	0.5517	0.4722	0.2933	0.2850	0.2312

The corrosion rate increases as exposure duration increases when there is no inhibition, as shown in Table 4.2, but when leaf extract is administered, the corrosion rate sharply drops.

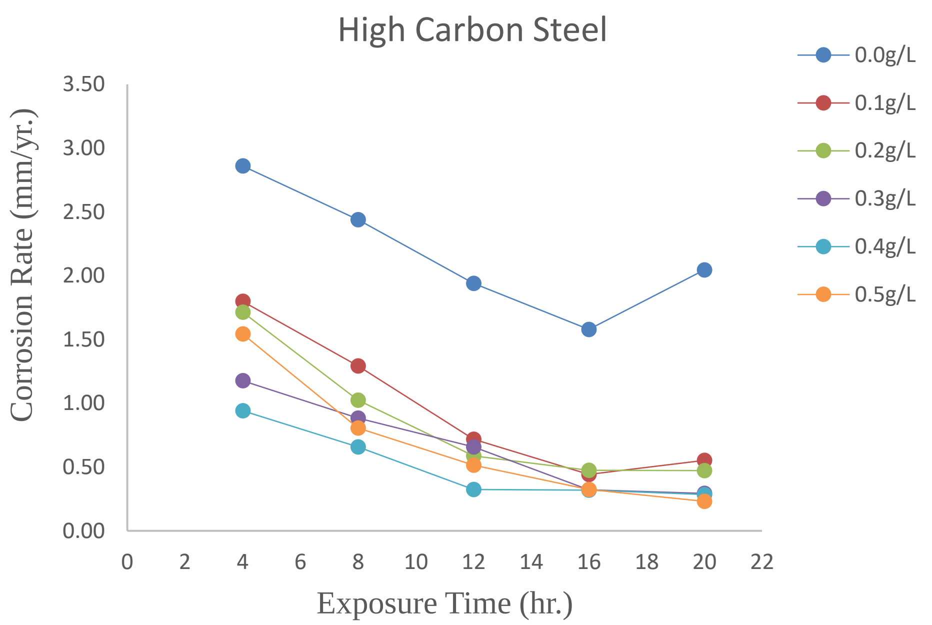

Figure 4.2: Plot of Corrosion Rate (mm/yr.) (for control + various concentrations) versus Exposure Time (hr.) for HCS with AZI leaf extract as inhibitor.

The graph shows the trend of high carbon steel CR over time in the presence of AZI leaf extract (control experiment) and lack of it (experiment) (figure 4.2). The control curve demonstrates that there was resistance to corrosion for the first 16 hours as a result of the test piece's surface developing a protective layer of film, which slowed the rate of corrosion. The test piece succumbed to further corrosion after 16 to 20 hours, indicating that the protective layer had totally disappeared. The corrosion significantly decreased and maintained a lower trend with the addition of different concentrations of AZI Leaf extracts (0.1 to 0.5g/L), indicating that the test pieces were shielded against deterioration. For values of CRs (control + various concentrations) vs. exposure time, see table 4.2.

Table 4.3: Efficiencies for High Carbon Steel (%) at Exposure Time (hr.) for Gravimetric Method.

Efficiencies (%) and 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	37%	40%	59%	67%	46%
8	47%	58%	64%	73%	67%
12	69%	70%	66%	83%	73%
16	72%	70%	80%	80%	79%
20	73%	77%	86%	86%	89%

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As concentrations increased, Table 4.3 shows how efficiency increased, but it also illustrates how efficiency declined as the inhibitor concentration was depleted during the process.

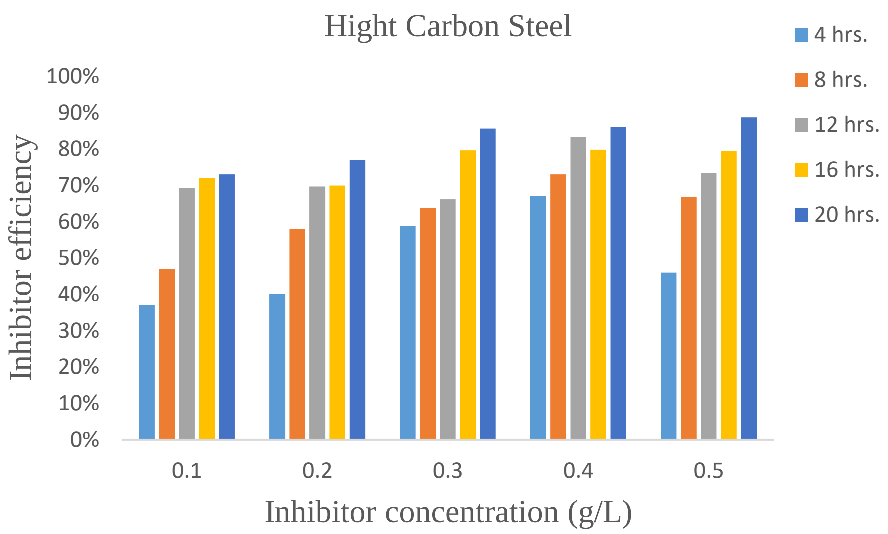

Figure 4.3: Efficiency (%) versus Concentration of inhibitor (g/L) for HCS with AZI leaf extract as inhibitor

The inhibitor's inhibitory effectiveness is displayed versus the concentration of the plant extract in Figure 4.3. It is clear that AZI leaf extract works wonders to prevent HCS from corroding in sulfuric acid. The effectiveness of inhibition increased as inhibitor concentration was raised. Efficiency and concentrations are displayed in Table 4.3 above.

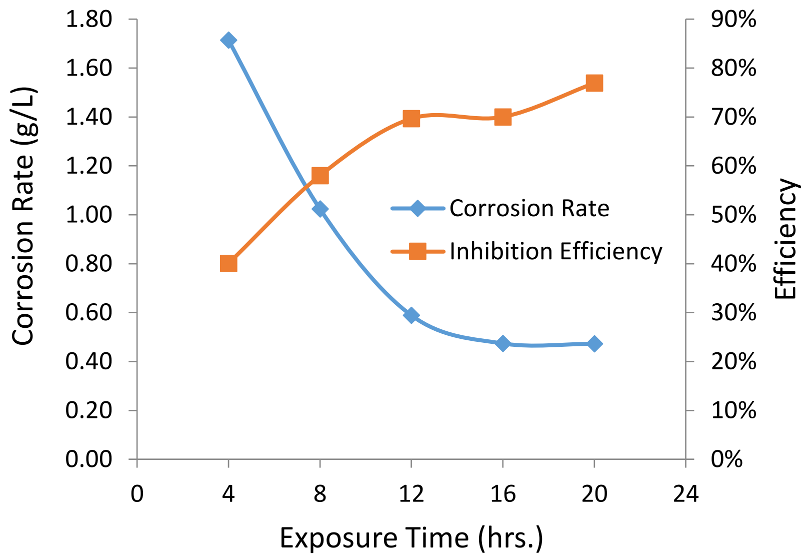

Figure 4.4: Plot of Corrosion Rate (g/L), Efficiency (%) versus Exposure Time (hrs.) at 0.1g/L Inhibitor Concentration

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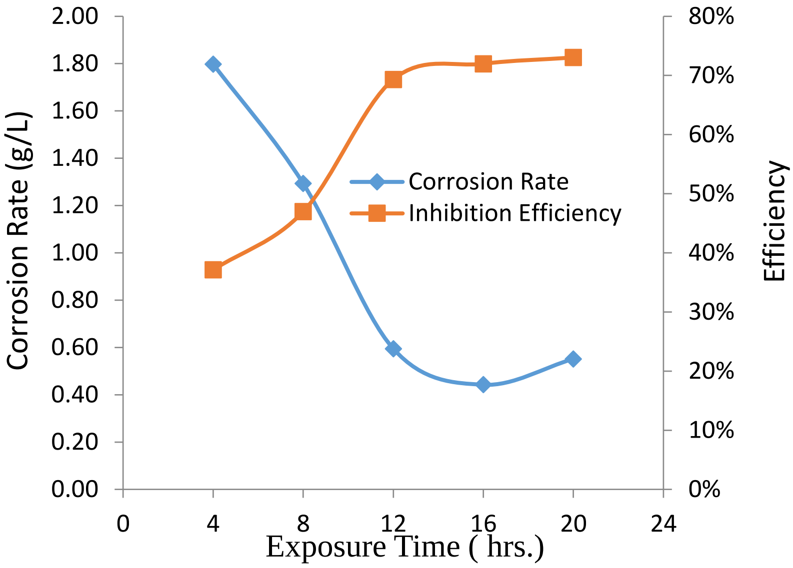

Figure 4.4: Plot of Corrosion Rate (g/L), Efficiency (%) versus Exposure Time (hrs.) at 0.2g/L Inhibitor Concentration

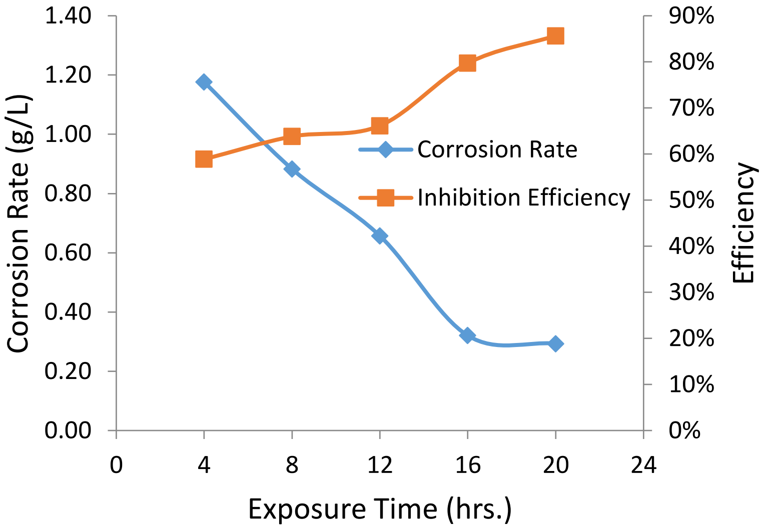

Figure 4.5: Plot of Corrosion Rate (g/L), Efficiency (%) versus Exposure Time (hrs.) at 0.3g/L Inhibitor Concentration

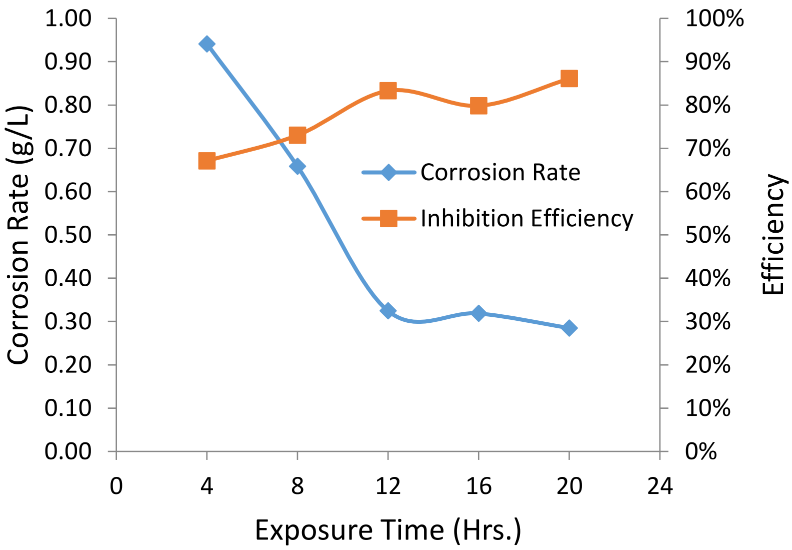

Figure 4.6: Plot of Corrosion Rate (g/L), Efficiency (%) versus Exposure Time (hrs.) at 0.4g/L Inhibitor Concentration

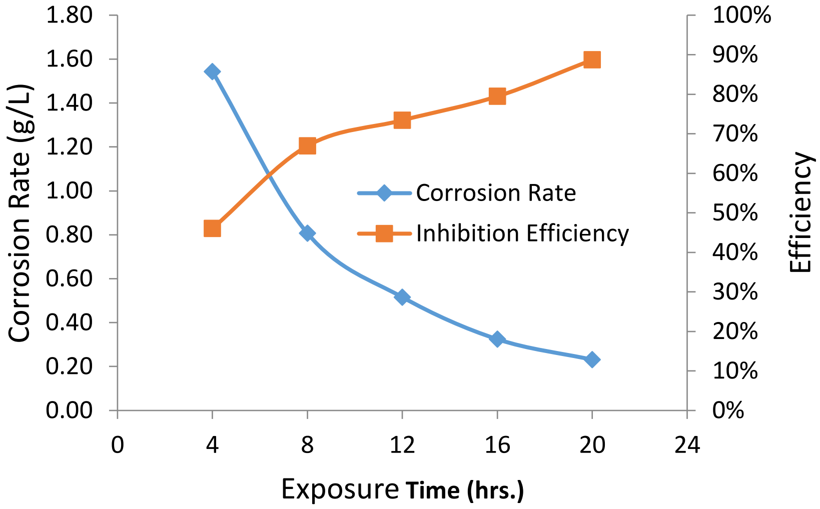

Figure 4.7: Plot of Corrosion Rate (g/L), Efficiency (%) versus Exposure Time (hrs.) at 0.5g/L Inhibitor Concentration

Figures 4.4 to 4.7 illustrate the test pieces' behavior when different amounts of AZI leaf extract were added. It is obvious that as the leaf extract is added, the corrosion rates decline while the effectiveness of the inhibition increases, demonstrating that the test coupons are being protected. However, it was noticed that the corrosion rates at 0.4g/L concentration increased slightly between hours 12 and 16 before declining again between hours 16 and 20. This could be the result of localized corrosion between hours 12 and 16. These findings indicate that AZI is a reliable corrosion inhibitor for high carbon steel.

5. CONCLUSION AND RECOMMENDATIONS

5.1. Conclusion

Based on the information gathered while studying the inhibitive potency of Azadirachta Indica Leaf Extract on the Corrosion of High Carbon Steel in Sulphuric Acid, the following conclusion can be drawn:

a. A suitable environmentally safe green inhibitor for high carbon steel that can be used in place of harmful chemicals is Azadirachta indica leaf ethanolic extract.

b. In the absence of Azadirachta indica leaf extract in a 1M H_2SO_4 solution, the gravimetric method shows aggressive degradation of the high carbon steel coupon immersed in the corrosive environment. Both the corrosion rate and weight loss increase significantly. showing how acid attack could damage the test piece's active site. However, when the inhibitor was added, there was a noticeable decrease in the rate of corrosion, indicating the leaf extract had created a protective film on the test piece.

c. The weight loss measurement shows that the rate of high carbon steel corrosion in a 1M H_2SO_4 solution is slowed down by the addition of Azadirachta Indica leaf extract. The maximum inhibitory efficacy was 89%, and when plant extract concentrations are raised, it will get better.

5.2. Recommendations

The findings of Green Corrosion Inhibitors' performance evaluation might be done better. For instance, standard deviation calculation, which calls for repeatability testing, can be highly useful in identifying flaws and inaccuracies in the research. Additionally, comparing various outcomes that have been published in the literature using statistical analysis yields useful information.

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