

Potentials of Azadirachta Indica (Neem) Leaf Extract as Corrosion Inhibitor of Medium Carbon Steel in Sulphuric Acid

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ABSTRACT:

The inhibitory activities of Azadirachta Indica leaf extract on medium carbon steel corrosion behavior were observed in electrolyte solutions at room temperature and the inhibition effectiveness was estimated using the potentiodynamic polarization technique. Before being ground to micron size for potentiodynamic polarization investigation, the leaves were collected, dried, and ground. Reflux ethanol extraction was used to extract the leaves, and the weight of each leaf that was removed was calculated. The quantities for different extract concentrations were also calculated and put to the acidic medium, where the test for different extract concentrations ranging from 0.1 to 0.5g/L was conducted using the medium carbon steel coupons. Utilizing I_{corr} and $I_{corr(i)}$ values for corrosion current densities, the inhibition efficiency was computed. As the concentration of the plant extract rose (from 0.0g/L to 0.5g/L), the corrosion rates decreased (from 4.58 mm/yr. to 0.29 mm/yr.). Azadirachta indica leaf extract is a superb inhibitor of medium carbon steel corrosion, with inhibition efficiency rising from 72% to 90% as the inhibitor concentration rose, according to the potentiodynamic polarization method. Due to their availability, biodegradability, low cost, and lack of toxicity to humans and the environment, plant extracts make good corrosion inhibitors. This study discovered that Azadirachta Indica leaf extracts can increase the service life of medium carbon steel when utilized in the right concentration.

Keywords: Corrosion, Azadirachta Indica Leaf, Concentration, Potentiodynamic Polarisation and Inhibition.

1. INTRODUCTION

According to the American Society of Testing and Materials (ASTM) International, corrosion is the result of a chemical or electrochemical

reaction between a substance, typically a metal, and its surroundings that accelerates the loss of the substance's qualities (Brycki, Kowalczyk, Szulc, Kaczerewska, & Pakiet, 2018). The International Organization for Standardization (ISO) also describes it as the physiochemical interaction between a metal and its surroundings that alters the metal's characteristics and may seriously affect the metal, the environment, or the technical system's ability to function (ISO 8044, 2015). Metal atoms are found in chemical compounds in nature, and variations in the electrolyte can cause corrosion. The resistivity of the soil, oxygen concentrations, moisture content, and various ion concentrations are examples of such differences.

In the gas and oil industry, internal pipeline corrosion is considered to account for 40% of all corrosion. One method for preventing corrosion in pipelines that transmit fluids (water and petroleum products) is the application of a protective surface coating. Coating a pipeline that is already damaged and full with petroleum product is not viable with this expensive approach. One of the most effective and economical solutions to this issue is the application of corrosion inhibitors. Therefore, there is an urgent need to identify a reliable corrosion inhibitor (Muthukumar, 2014).

Metal corrosion control has been crucial, particularly in the production industry (Rani & Basu, 2012). Corrosion inhibitors have historically been regarded as the first line of

defense against internal corrosion in the oil and gas production and processing sectors. Corrosion can cause infrastructure and machinery failures in plants, which are typically expensive to fix, expensive in terms of lost or polluted products, expensive in terms of environmental harm, and possibly even expensive in terms of human safety. An accurate evaluation of the factors influencing a structure's corrosion and rate of degradation is necessary to make decisions about the structure's or component's long-term integrity. With this knowledge, a wise choice can be made regarding the kind, cost, and timeliness of any corrective measures to be taken (Hou, Ma, Du, Zhang, & Zheng, 2017).

Although there are numerous ways to control corrosion, using corrosion inhibitors is the most practical technique utilized in both industrial settings and academic research to protect metals and their alloys against strong environmental attack. The majority of commercial corrosion inhibitors used in sectors like oil and gas are multi-component inhibitor solutions with capabilities for both nitrogen and sulphur. These inhibitors can be costly to create, and are stable and effective in corrosive settings, but can be toxic, endangering human health and the environment. Multi-component inhibitor systems are also persistent and call for expensive techniques to remove them because they are often present alongside other heavy metals, chromates, nitrites, and phosphates. (Abbas et al., 2014).

In the beginning, sodium arsenite (NaAsO_2) and sodium ferrocyanide ($\text{Na}_4\text{Fe}(\text{CN})_6$) were used as inorganic inhibitors to prevent corrosion caused by carbon dioxide (CO_2) in oil wells, but due to the frequency and effectiveness of the treatments, many organic chemical formulations that frequently contained film-forming amines and their salts were developed (Ibrahim et al., 2012). These corrosion inhibitors are made because they work well at a variety of temperatures, get along with protected materials, dissolve well in

water, are inexpensive, and have a low level of toxicity (Brycki et al., 2018). Organic corrosion inhibitors attach to the surface and create a barrier film that repels water and prevents deterioration.

Metal degradation is one of the key elements affecting the systems' dependability in the chemical, automotive, and transportation industries. For instance, thousands of kilometers of pipes, pumps, pressure vessels, and storage tanks are used to process, store, and transport goods in the oil, gas, and petrochemical industries. These infrastructures are essential to a nation's economy in addition to being essential to the existence of these sectors. But since the vast majority of these installations and their parts are composed of steel and aluminum alloys, they are undoubtedly prone to corrosion or degradation. The majority of the time, these failures may lead to product leakage, which is invariably bad for society because it poses a threat to environmental safety, a risk to human safety, and a significant loss in output. Additionally, the potential involvement of issues like compensation and legal action is bad publicity. The monitoring and inspection of these facilities receive a lot of attention as a result. By implementing sound corrosion prevention procedures, it is possible to extend or even do away with the time that these components need to be inspected. Additionally, by slowing down corrosion, these solutions will increase inspection or monitoring times, which will decrease operation costs.

By infusing corrosion inhibitors that are water- and oil-soluble and can sufficiently spread through stratified water layers into transportation pipelines, corrosion can be reduced. Because the properties of the inhibitors are not assessed before use, many incorrect applications of inhibitors take place, which has a substantial impact on microbial corrosion. Inhibitors are also destroyed by bacteria, which lessens their potency and speeds up corrosion. Organic compounds

employed as typical oilfield corrosion inhibitors provide a film or protective barrier between the metal and the corrosive fluids as a result of their anodic, cathodic, or mixed-type activity. In the petroleum industry, amino groups such as imidazolines, amidoamines, and polythiol compounds are used as corrosion inhibitors. Aliphatic fatty acid derivatives, imidazolines, quaternaries, and rosin derivatives are the four primary types, all of which contain long-chain hydrocarbons (C₁₈). There is no dual inhibitor that combines biocidal and inhibitory effects, though. As a result, the time is now for developing a multipurpose inhibitor for a tropical pipeline (Muthukumar, 2014).

The issue of corrosion is really important. Corrosion has a significant impact in three areas: economy, improved safety, and resource preservation. In addition to the loss of natural resources, the leakage of hazardous materials from a transport pipeline carries the risk of deadly environmental effects as well as human casualties. Although pipes are built and engineered to retain their integrity, a variety of factors (such as corrosion) make it challenging to prevent leaks from occurring in a pipeline system over the course of its lifetime (Koch, 2017).

An electrochemical process called corrosion occurs when a current exits a structure at the anode site, travels through an electrolyte, and then returns to the structure at the cathode site. As the pipe moves along, differences in potential start to emerge. For instance, because it is in soil that has a lower resistivity than the rest of the pipeline, the current might exit the pipeline at that anode site, travel through the soil, and then re-enter the pipeline at a cathode location. As a result of these potentials, corrosion currents are created, which exit the pipe and enter the soil in specific places.

Steel pipelines are essential for transporting gases and liquids across long distances from their sources to their final consumers anywhere in the world. Approximately 460,000 km

(285,000 miles) of common carrier pipelines are now in operation in the US, moving 46% of all crude oil and refined products carried in the nation. More than 1.6 x 10⁶ km (106 miles) of natural gas pipelines were in use in the United States in 1984, with about a quarter of them being interstate. At the time, there were approximately 2,800,000 km (174,000 miles) of interstate liquid transportation pipeline in operation in the United States. Around 1.6 x 10⁶ km (106 miles) of natural gas pipelines were operational in the United States in 1984, with more than 25% of them being interstate. There were about 2,800,000 km (174,000 miles) of interstate liquid pipeline in the United States at the time. More pipelines are being constructed than ever before. In the United States, pipelines for natural gas, crude oil, and refined goods totaled 9800 kilometers (6090 miles), 5740 kilometers (3567 miles), and 2660 kilometers (1652 miles) respectively in 1986. The country's vital energy needs are addressed by the more than 9000 km cross-country crude oil and product pipeline network of the Indian Oil Corporation (Muthukumar, 2014).

Huge quantities of money are used each year to prevent, monitor, inspect, and repair corrosion-related damage. Internal corrosion control programs frequently involve chemical treatment with corrosion inhibitors. Available corrosion inhibitors include those that are oil-soluble, water-dispersible, water-soluble, limited-solubility, and volatile. Each one performs differently depending on the pipeline's specific conditions. Strong protective films can be applied in batches and have a long-lasting quality that can persist for several weeks or months. Alternately, they can be continuously injected into the pipeline using a continuous injection programmer in low concentrations, which creates and preserves a thin layer over time. As long as the inhibitors can create a potent film and enter the pipe, they will function properly. Inhibitors used to treat the pipe's walls can be devoured by debris, corrosion products, and bacteria that build up inside the pipe. This also prevents the inhibitors

from penetrating the pipe's walls behind the deposits. As a result, pipelines should be as clean as possible prior to applying corrosion inhibitors (Muthukumar, 2014).

1.1. Corrosion Implication on Human life, Environment and safety

Corrosion can have an unthinkable effect on human life and safety, yet because of liability concerns or simply because the evidence disappeared following the catastrophic event, this destructive phenomena has gone unrecognized as the main cause of many fatal occurrences. The collapse of the Silver Bridge is among the most dangerous and well-known corrosion accidents (Fiori-Bimbi et al., 2015). On December 15, 1967, this bridge, which linked Point Pleasant, West Virginia, with Kanauga, Ohio, suddenly collapsed into the Ohio River, killing 46 people. Stress corrosion cracking (SCC) and corrosion fatigue were found to be at blame for this disaster. The night of December 2-3, 1984, in Bhopal, India, saw one of the worst industrial accidents in terms of the number of people killed and hurt. At Union Carbide India Limited, an unfortunate seepage of water (500 liters) brought on by corrosion of pipelines, valves, and other safety systems led to the release of methylisocyanate (MIC) and other dangerous reaction products into the surrounding environs. 3000 people died and an estimated 500,000 people were injured in this catastrophe (Aljourani et al., 2010). The collapse of the swimming pool roof in Uster, Switzerland in 1985 is another famous corrosion disaster. Twelve people were killed when the stainless-steel rods holding up this swimming pool's ceiling broke due to SCC (Vermeirssen et al., 2017).

1.2. Review of Related Work

The adaptability of steel and alloys as a construction material is unmatched by other alloys. The most common building material is produced in excess of 1.4 billion tons annually (World Steel Association, 2013). It connects all industries, including housing, power,

agriculture, and water supply, making it a crucial part of daily life. It is used in a variety of things in our daily life, including buildings, chemical plants, machinery, tools, transportation pipelines, pressure vessels, and storage tanks. Steel is a significant economic driver worldwide as a result (World Steel Association, 2013). Global steel production in 2014, indicating that steel production will continue to increase (Mineral Commodity Summaries, 2015). Steel production must be increased in developing nations like those in Africa, Asia, and Latin America where it will be essential to raising living standards. It is anticipated that new infrastructure will account for 60% of the steel used in these regions. In order to maintain and increase the life of steel that is already in service, appropriate corrosion protection and control procedures are needed because the reprocessing of end-of-life steel products and the production of steel from fresh ore will not be able to keep up with demand.

Green corrosion inhibitors are chemicals that can sluggish corrosion reactions when applied to the process fluid. Their inhibitory method may not always be easily understood and studied. Anodic, cathodic, or mixed inhibitory effects are possible. It is also important to note that some inhibitors are completely unique to the metal or alloy and environment, and they may not exhibit any inhibitory phenomena with other substances. While cathodic inhibitors often work by selectively precipitating on cathodic sites to stop the diffusion of reducing species to the metal's surface, anodic inhibitors typically work by producing a protective oxide film on the metal's surface.

The majority of green corrosion inhibitors fall within the mixed category, by lowering both of their electrochemical rates, mixed-type inhibitors can carry out a cathodic and anodic action simultaneously. The adsorption of molecules with double/triple bonds or those having V and VI group elements, such as N, P, S, and O, which contain free-electron couples, is frequently linked to the mechanism of

inhibition. The process of determining the "class" is rather simple. The key gap in the literature is that more insights are required for determining the mechanisms.

Inhibitory effects of roselle leaf (*Hibiscus sabdariffa* (AELHS)) on mild steel corrosion in acidic baths (1.2 N HCl and 1.2 N H₂SO₄) were investigated using gravimetric techniques after the plant's extraction and synthesis. The findings of this investigation show that this chemical is more effective at preventing mild steel corrosion in 1.2 N H₂SO₄ than 1.2 N HCl. The data are evaluated using the Langmuir, Frumkin, Florry-Huggins, and Langmuir-Freundlich isotherms; the Langmuir isotherm matches the data the best, with a correlation coefficient of > 0.99 in both acid environments (Murthy & Vijayaragavan, 2014).

The results demonstrate the efficiency of (AELHS) as a medium carbon steel corrosion inhibitor. Four adsorption isotherms—Langmuir, Frumkin, Florry-Huggins, and Langmuir-Freundlich—were looked at for the data. With a correlation coefficient of above 0.99 in both the acidic (HCl), (H₂SO₄) and alkaline conditions, the Langmuir isotherm adequately fits the data. The effectiveness of the inhibitor depends on the type of acid, how long it is exposed for, and the acid's concentration. The inhibitor inhibitory properties in the H₂SO₄ environment are better because inhibitor adsorption on mild steel occurs more spontaneously in the presence of H₂SO₄ than in the presence of hydrochloric acid. Overall, the H₂SO₄ environment appears to be much more favorable for the AELHS inhibitor's performance than the hydrochloric acid environment. 2014 (Murthy & Vijayaragavan).

El-Haddad (2013) released a report testing the chitosan corrosion inhibition impact on copper surfaces, which excluded any parallel effects brought on by other compounds in the utilized solution. Chitosan was obtained by Sigma Aldrich. In terms of electrochemical characteristics, their findings were more

reliable, and they categorize the molecule as a mixed-type inhibitor working mostly on cathodic sites. They still used a Langmuir isotherm model to fit the data, but they also added a more in-depth computational analysis of the HOMO-LUMO orbitals. As a result, their hypothesis on the inhibition mechanism is more accurate because they know precisely which molecules are involved, the only ones that can have an impact on the inhibition effect in the end.

2. MATERIALS AND METHOD

2.1. Azadirachta Indica Leaf Preparations

Azadirachta indica (AZI) leaves were gathered in November 2021 in Aba, Abia State, in the eastern part of Nigeria. Before being used to create the extract, the leaves were air-dried at room temperature for 30 days in the laboratory. The extraction was performed using the reflux technique with ethanol as the extraction solvent for 3 hours at constant heat (70 °C). The extract is subsequently diluted into several concentrations using 1M H₂SO₄ solution as the corrosive medium, according to Nnanna et al. (2012). These concentrations are 0.1, 0.2, 0.3, 0.4, and 0.5 g/L.

2.2. Metal Preparations

A medium carbon steel (C-1345) sample with the following chemical composition was used in the experiment: C = 0.48%, Mn = 2.53%, Si = 0.64%, Cr = 0.46%, Ti = 0.15%, Cu = 0.07%, Mo = 0.68%, and Fe = 95.7% was used for this experiment. The metal sheets were cut into 20 x 20 x 4 mm coupons, abraded with different emery paper grades (120, 600, and 1200), washed with detergent, degreased with ethanol, and allowed to air dry before being weighed.

2.3. Potentiodynamic Polarisation (PDP) experiment

The polarization samples were made from MCS and had dimensions of 2 cm x 2 cm x 4 cm. They were subsequently sealed with epoxy

resin, leaving only one square surface (1.0 cm²) exposed. The exposed surface was cleaned with acetone, rinsed with distilled water, and allowed to air dry. The electrochemical polarization experiment was run at a scan rate of 0.333 mV s⁻¹ in the potential range of -1000 to 2000 mV. Each test was repeated three times to ensure that the systems were repeatable.

The Materials Studio 4.0 software's DFT electronic structure tools Forcite and Dmol3 were used for all theoretical computations (Accelrys, Inc.). Using the Stern-Geary Equation, the current density was calculated after the necessary parameters, potentials, and related currents were registered.

$$i_{corr} = \left(\frac{\beta_a \beta_c}{2.303(\beta_a + \beta_c)} \right) / R_p \quad (1)$$

where, i_{corr} = the corrosion current density, β_a & β_c = slopes of anodic and cathodic of Tafel slopes, respectively and R_p = Material's resistance of the test sample will be found by taking the slope of the potential versus current.

Then, the corrosion rate (CR mm/yr.) can now be calculated with the equation below

$$\frac{i_{corr} \times E_q \times 10 \times 3.15 \times 10^7}{F\rho} \quad (2)$$

where, E_q = Equivalent mass of metal exposed to corrosion (g), ρ = Density of the metal sample (g/cm³) and F = Faraday constant (96,500 C)

The inhibition efficiency of PDP was calculated using Equation below.

$$IE_{Taf}(\%) = \left(\frac{I_{corr} - I_{corr(i)}}{I_{corr}} \right) \times 100\% \quad (3)$$

where, $I_{corr(i)}$ is the current density when inhibited and I_{corr} is current density without inhibition.

3. RESULTS AND DISCUSSION

3.1 PDP Measurements

Below are the results of the PDP experiment.

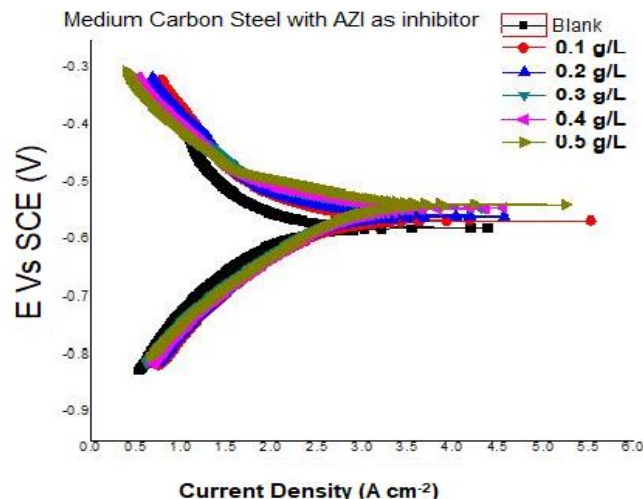


Figure 1: Plot of Potential (V_{SCE}) versus Current Density (Acm⁻²) for PDP Experiment with AZI as inhibitor for MCS.

Figure 1 shows the potentiodynamic curves for MCS electrodes in 1M H₂SO₄ both in the absence and presence of different doses of AZI. Anodic polarization becomes more positive while cathodic polarization becomes more negative. Following the addition of the extract, there was both a dramatic decrease in current and an increase in polarization resistance, both of which indicated the formation of protective coatings. As the concentration of extract increases, the corrosion progressed more slowly and inhibition was more effective.

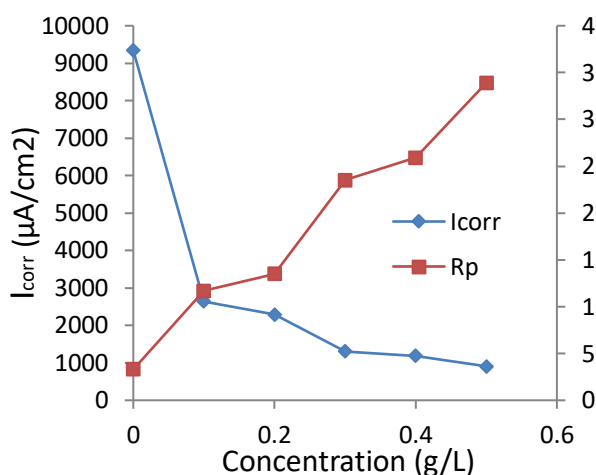


Figure 2: plot of corrosion current density (I_{corr}) and polarization resistance (R_p) against different concentration (g/L).

Figure 2 depicts a plot of polarization resistance (R_p) and corrosion current density (I_{corr}) against AZI leaf extract concentrations ranging from 0.1 to 0.5g/L. I_{corr} and $I_{corr}(i)$ are the corrosion current densities in the absence and presence of an inhibitor (extract), respectively, according to equation (3). Looking at the graph, it was clear that the I_{corr} values gradually fell as the concentration of the extract increased. The corrosion rate was 0.45 mm/yr. at the concentration of 0.5 g/L. As a consequence, it is confirmed that medium carbon steel of grade (AIAI, C - 1345) can be effectively inhibited by AZI leaf extract in an acidic environment. Additionally, when the leaf extract was added, it was observed that the polarization resistance started to rise as the extract concentration increased, indicating the formation of protective films on the test piece and the subsequent reduction in corrosion current as a result of the test coupon's non-oxidation, which rendered it resistant to further corrosion.

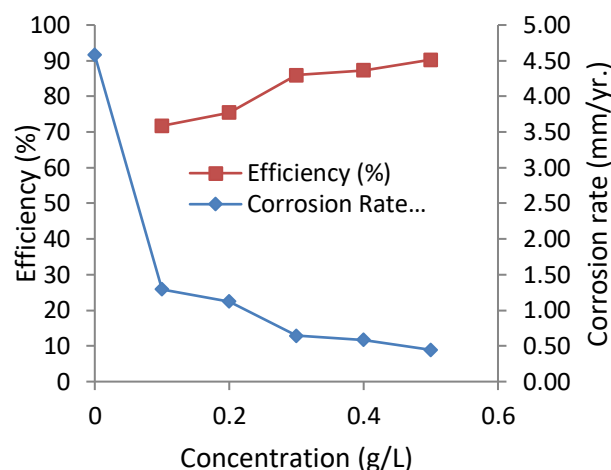


Figure 3: plot of efficiency (%), corrosion rate (mm/yr.) versus concentration (g/L).

Figure 3 shows the plot of efficiency, corrosion rate versus concentration of the leaf extract, when the experiment was run without an inhibitor, the corrosion rate significantly increased; however, when the leaf extract was added to the acidic media, the corrosion rate dramatically decreased, falling from 4.58 to 1.29 mm/yr at 0.1g/L and continuing to decline until 0.45 mm/yr at 0.5g/L, supporting the claim that the corrosion rate decreases as extract concentration increases.

4. CONCLUSION AND RECOMMENDATIONS

4.1 Conclusion

The following inference can be made in light of the data amassed while researching the inhibitive properties of Azadirachta Indica Leaf extract on the Corrosion of Medium Carbon Steel in Sulphuric Acid:

- i. Azadirachta indica leaf ethanolic extract is a suitable environmentally benign green inhibitor for medium carbon steel and can be used in place of hazardous compounds in 1 M H_2SO_4 solutions.
- ii. The potentiodynamic polarization method reveals aggressive deterioration of the medium carbon steel coupon

immersed in the corrosive environment in the absence of *Azadirachta Indica* leaves extract in 1M of H₂SO₄ solution. The corrosion rate increased sharply, indicating that the active site of the test piece was bare to acid attack.

- iii. The potentiodynamic polarization measurement demonstrates that adding *Azadirachta Indica* leaf extract to a 1M H₂SO₄ solution slows the pace at which medium carbon steel corrodes in the acid. The maximal inhibitory efficacy was 90%, and expected to improve as plant extract concentrations are increased.
- iv. When a plant extract was added, medium carbon steel was shielded from corrosion by the compound of the extracts adhering to the metal surface, blocking the transfer of charge and mass and making the metal less prone to corrosion reaction.

4.2 Recommendations

The following topics are suggested for more research in light of the knowledge gathered from this study.

- i. To determine the active species in the adsorption layer, more research is needed to access the corrosion morphology.
- ii. Investigation of the inhibitive properties of *Azadirachta Indica* leaf extracts on medium carbon steel in saline environment.
- iii. To examine how temperature affects the ability of *Azadirachta Indica* leaf extracts to prevent medium carbon steel from corroding in corrosive acid solutions.

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