

IMPACT OF NEEM (DOGONYARO) LEAVE EXTRACT AS A GREEN INHIBITOR TO CONTROL CORROSION OF MILD STEEL IN HCl ACID MEDIUM

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Abstract

The major process parameters of leaf extract of Neem (Dogonyaro) on mild steel corrosion in acidic medium (HCL) was investigated by the weight loss technique and optimized using the response surface approach (RSM). The elemental compositions of metal were determined using an Atomic Absorption Spectrophotometer (AAS). Phytochemical analysis was investigated to confirm the presence of organic constituents such as Alkaloids, Tannins, Terpenes, Glycosides, Saponins, and Flavonoids, which made the neem leaf extract act as a good inhibitor. The effect of the parameters such as inhibitor concentration, time, effect of temperature and medium concentration on corrosion rate in an acidic (HCL) medium was investigated. The results show that iron is the main component of mild steel, and that the presence of phytochemical components and functional groups in the extract makes it effective inhibitor. The optimal inhibition efficiency was 83.9 percent temperature: 47.3⁰C, inhibitor concentration: 12.10 mg/mol, time 7.50s, according to studies from the experimental design of neem. Within neem

inhibited acid media, the best feasible conditions yielded inhibition efficiency of 84.85 percent, respectively. This suggests that Neem leaf extract is a good inhibitor.

Key words: Optimization, Inhibitors, Neem, Hydrochloric acid, corrosion rate and weight loss.

INTRODUCTION

BACKGROUND OF THE STUDY

Corrosion can be defined as an irreversible reaction of a material with the environment, which usually (but not always) results in a degradation of the material or its properties. It is the inevitable deterioration (thermodynamically favoured) of materials by the chemical interaction with their environments. It is the returning of the materials to its original form (stable state) to the mother earth. There are several aspects of corrosion: the material, the environment, and the material properties (Uhlig, 2000; Fontana, 1999; Bardal, 2003).

The degradation of metals has a negative influence on the populace and is apparently an issue of concern as it influences the economic cost and also creates safety awareness from the damages such as in pipelines, building, bridges, waste water system, and even our residence. A recent study in USA of industrial sectors, predicts the cost of corrosion would rise over \$1.1 trillion in 2016 (Joshua, 2016). These estimates were based on a landmark study by NACE (National Association of Corrosion Engineers) that estimated (direct) corrosion costs were \$276B in 1998 as reported in the NACE Corrosion Costs Study.

Several methods used in the controlling of corrosion in structures are available. The most frequently used techniques include organic and metallic protective coating; corrosion resistant alloys, plastics and polymers; corrosion inhibitors and cathodic production technique which are used on piping, underground storage system and offshore facilities (Ajayi *et al*, 2011). However, there is no consensus regarding which method assesses corrosion levels of structures most accurately, that is why the technique for corrosion detection will remain an issue no matter how corrosion inhibition is being carried out.

The rate and extent of corrosion depend mainly on the nature of the metal and the nature of the environment (Krishnamurthy *et al*, 2014). Thus the position of the metal in the galvanic series; relative areas of the anode and cathode; purity of the metal; physical state of the metal; nature of the oxide film; solubility of the products of corrosion; temperature; humidity; pH; nature of the electrolyte; concentration of oxygen and formation of oxygen concentration cells affect corrosion (Bardal, 2003; Roberge, 2008).

STATEMENT OF THE PROBLEM

A lot of researchers has done many works on corrosion in acidic medium, precisely like hydrochloric acid, sulphuric acid etc but enough work up till now is yet to be done on the impact of corrosion control activities using the neem leave plant extracts, to portray industrial interest towards use. This is the focus of this study.

AIM AND OBJECTIVE OF THE RESEARCH

The aim of these research efforts is to study the impact of corrosion inhibition activities using concentration of the neem leaf plant extracts to control corrosion of mild steel in HCl acid medium.

Specific objectives are:

1. To Study the extraction of juice from the neem leaf plant.
2. To characterize the neem leaf plant extracts using analytical and instrumentation.
- 3 To verify the metallic composition of the mild steel using Atomic Absorption Spectrometer, AAS.
4. To determine the corrosion behavior of mild steel in acidic (HCl) environment.
5. To determine the effects of process parameters, concentration of the neem leaf plant extracts, contact time, and temperature and concentration of the medium on the rate of corrosion of mild steel using the weight loss method and to optimize the inhibition process using response surface methodology (RSM).

Research Questions

1. How can we extract juice from the neem leaf?
2. How can we characterize the neem leaf extract using analytical and instrumentation
3. What is the metallic constituents of the mild steel using atomic absorption spectrometer?

4. How does mild steel respond when exposed to an acidic environment?
5. In order to improve the inhibition process using response surface methodology (RSM), what effects do process parameters, concentration of neem leaf extract, contact time, temperature, and medium concentration have on the rate of corrosion of mild steel using the weight loss method?

SCOPE OF THE STUDY

This research is limited to the synthesis of neem leaf plant extracts for corrosion inhibition and optimization investigations. Corrosion rate when weight loss is used.

SIGNIFICANCE OF THE STUDY

- (1) The goal of this study is to promote the use of locally grown plants as corrosion inhibitors and to create the best possible circumstances for their usage in both the oil and non-oil industries.
- (2). If corrosion is control it reduces the overall economic cost.
- (3). If corrosion is control it creates a certain level of safety from the areas such as in pipelines, building, bridges, waste water system, and even our residence to prevent damaging effects of corrosion.
- (4). This study, which uses a central composite design of response surface methodology to optimize the use of neem leaf extracts in an acidic medium to control mild steel corrosion, is anticipated to offer suggestions for new ways to make green inhibitors by turning plant waste into wealth and managing it in the environment.

MATERIALS AND METHODS:

From Amokwu, Udi, Enugu State, Nigeria, neem leaf will be gathered. The voucher for mild steel would be bought in Kenyatta Market in Enugu, Nigeria's Achara Layout. We will buy acid of the analytical grade from Gerald Chemicals Ltd. in Enugu, For analysis, the following tools must be used: AAS, SEM, weighing scale, beakers, heating mantle, ethanol, and stopwatch. Nigeria's Ogbete Main Market.

METHODS:

Extraction of juice from the leaves

To get rid of undesirable stuff, the plant leaf will be cleaned thoroughly with water. After being ground up, the samples will be dried and weighed. Prior to use, the weighted inhibitors will be kept in desiccators. Each ground sample will be combined with 70g of ethanol and sealed firmly for 48 hours to prevent evaporation. The extracts will next be filtered to provide high concentration yields. In order to remove the ethanol, the filtered solutions will be heated in a rotary evaporator setup for 25 minutes at 69°C.

Characterization of the extracts

The extracts will be subjected to phytochemical laboratory analysis at Projects Development Institute, PRODA, Enugu, Nigeria, and phytochemical instrumentation analysis at NARICT, Zaria, Nigeria, with results being printed from spectroscopy to identify the compounds, functional groups, and their structures. American Society for Testing and Materials (ASTM International) will be used for the laboratory analysis. Tests on the instrumentation: neem leaf extracts will be analyzed using Fourier Transformation Infrared spectroscopy in order to identify any active functional groups that are present.

Phytochemical analysis

The method described by Eddy and Ebenso (2008) will be used for the phytochemical analysis of the ethanol and aqueous extracts of the plant samples. Both the foaming test and the Na_2CO_3 test will be used to identify saponin. Testing with ferric chloride and bromine water will be performed to identify tannin. Leberman's and Salkowski's tests will be utilized to identify cardiac glycosides, while Dragendorff, Hagger, and Meyer reagents will be used to identify alkaloids.

Metal Specimen preparation

Using AAS, the resulting mild steel will be examined to ascertain its metallic content. By using the technique utilized by Awe et al., (2015), it will be used to get ready for the corrosion experiment. The mild steel specimens will be mechanically cut into dimensions of 3.0 cm × 3.0 cm with a thickness of 1.2 mm (with a surface area of 9.0 cm²). Before anything else, the mild steel will be mechanically polished to sufficiently remove any mill scale from the mild steel sample using emery paper of grades ranging from 400 to 1200. The specimen will undergo a thorough cleaning with distilled water, 100% ethanol degreasing, dipping into acetone to prevent corrosion, and air drying. Before usage, the dried specimens will be kept in desiccators.

Experimental Technique

Awe et al., (2015) method will be used to conduct weight loss measurements under total immersion using 250 ml capacity beakers containing 100 ml prepared acid (HCl) solution, which shall be kept at 30°C to 70°C. With the use of an acid resistance plastic clip and under the

necessary circumstances, the mild steel must be weighed and dropped in various acid concentrations. The coupons will be obtained every so many seconds, such as every two, four, six, eight, and ten. The mild steel coupons must be taken out after each exposure period, properly cleaned using emery paper to remove the corrosion product, rinsed with distilled water, and dried in acetone as previously mentioned. After reweighing the mild steel, the weight loss in grams was calculated using the difference between the mild steel's pre- and post-immersion weights. With varying inhibitor concentrations in the solution, the process will be repeated. In both the absence and presence of the inhibitors, the corrosion rates ($\text{g}/\text{cm}^2\text{h}$) will be calculated. Table 1 displays the several variations of the components utilized in the experiment. The difference between each coupon's weight before and after immersion, as reported by Awe et al., (2015), will be used to compute weight loss.

$$\Delta W = W_b - W_a \quad (1)$$

is the weight before immersion; is the weight after immersion. While the corrosion rate ($\text{g}/\text{cm}^2\text{h}$) in absence and presence of inhibitors shall be calculated using equation 2

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

Where ΔW is the weight loss (g) after exposure time t (h), A is the area of the specimen (cm^2), t is time of exposure in hours and CR is the corrosion rate at each exposure time. The corrosion rate obtained in the absence and presence of inhibitor shall be used to calculate inhibition efficiency (IE %) as in equation 3.

$$IE(\%) = \frac{CR_1 - CR_2}{CR_1} \times 100 \quad (3)$$

Where E (%) is inhibition efficiency, R_1 is the corrosion rate of mild steel in absence of inhibitors; R_2 is the corrosion rate of mild steel coupons in the presence of inhibitors.

Optimization of the inhibition process using response surface methodology (RSM)

The experiment will be planned using Design Expert software (version 10), and the inhibitory conditions will be optimized. This study's experimental design, which consists of 30 experiments, is a two-level, four-factor full factorial design. For the optimization investigation, the concentration of the extract (%v/v), exposure time (hour), and temperature ($^{\circ}\text{C}$) at a constant acid chloride medium were chosen as independent parameters. The answer chosen was inhibition efficiency, IE (%) from mild steel alloy corrosion inhibition utilizing specific plant extracts. To forecast a good estimation of errors, six replications of the center points were employed, and the experiments were carried out in a randomized order. The levels of each factor, both actual and coded, are as displayed. The codes for the coded values were -1 (minimum), 0 (centre), +1 (maximum). The definition of alpha is the distance from the center, which may be inside or outside of the range. It is important to highlight that the program employs the idea of coded values to investigate significant terms; as a result, an equation based on coded values was employed to examine how the factors affected the replies. Equ. 1 depicts the empirical equa. as follows:

$$Y = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{i=1}^4 \sum_{j=i+1}^4 \beta_{ij} X_i X_j + \sum_{i=1}^4 \beta_{ii} X_i^2 \quad (1)$$

Where Y is the response factor (corrosion rate), x_i is the i th independent factor term, β_0 is the intercept, β_i is the linear model coefficient, β_{ii} is the quadratic model coefficient, and β_{ij} is the linear model coefficient for the interaction between β_i and β_j . Equ. 2 was used to calculate the process variables' coded values:

$$x_i = \frac{X_i - X_0}{\Delta X} \quad (2)$$

Where x_i is the coded value of the i th variable, X_i is the uncoded value of the i th test variable, X_0 is the uncoded value of the i th test variable at the center point, and ΔX is the change or intervals between the uncoded values. Based on the results of the trials, levels for each factor were chosen.

Table 1: Range of each factor in actual and coded form for corrosion inhibition of in acidic medium.

Factor	Units	Low level	High level	$-\alpha$	$+\alpha$	0 level
Conc. of Extract (A)	(mg/100ml)	10(-1)	20(+1)	5(-2)	25(+2)	15
Temperature (C)	°C	40(-1)	60(+1)	30(-2)	70(+2)	50
Conc. of media (D)	Mol/dm ³	1.5(-1)	2.5(+1)	1(-2)	3(+2)	2

RESULTS AND DISCUSION

Yield of extract from the leaves

From each of the 60g of dried neem leaves, 35g of neem leaf extract was extracted. According to Awe et al. (2015), these indicate a 58% yield, indicating that the leaf contains a significant amount of extract that can act as green inhibitors.

Phytochemical analysis

The phytochemical components of the ethanol extract of neem extract are displayed in Table 2. The findings show that anthraquinone is absent but saponin, tannin, terpenes, flavonoids, perpenes, glycoside, and alkaloids were found. The phytochemical constituents of the ethanol

extract of these leaves may be responsible for the inhibition of mild steel corrosion, as the majority of these phytochemicals have electron-rich bond or heteroatoms in their chemical structures that aid in their electron-donating ability. Other studies have found similar results about the ethanol extract of certain leaves' ability to suppress the corrosion of mild steel and aluminum (Ebenso et al.,2006, El-Shamy et al.,2020, Alaneme et al.,2015, Hassan et.,2016, Makkar et al., 1996).

Table 2. Phytochemical Constituents of Ethanol Extracts of Leaves

S/N	Phytochemicals	Neam leaf Extract (%w/w)
1	Saponins (% w/w)	6.61
2	Terpenes (% w/w)	9.59
3	Tannins (mg/100g)	9.59
4	Flavonoid (% w/w)	5.84
5	Phlobatannins (% w/w)	-
6	Anthraquinones (% w/w)	-
7	Glycoside (mg/100g)	1.59
8	Alkaloids (% w/w)	6.34

Table 3: Metal Composition of Mild steel Using *Atomic Absorption Spectroscopy (AAS)*

Metals	Composition (Wt %)
Magnesium	0.89
Phosphorus	0.70
Carbon	0.49
Silicon	0.05
Iron	97.87

Impact of inhibitor concentration on corrosion rate

The graph in Fig. 2. illustrates the fluctuation in mild steel corrosion rate in an acidic media when different amounts of ethanol extract from neem (*Azadirachta indica*) leaves were present or

absent. The figure showed that as the extract content increased, the rate at which mild steel corroded in the medium reduced. This could be due to the fact that as the extract's concentration increased, more extract ingredients adsorbed to the mild steel's surface, forming a barrier that stops further corrosion and mass transfer. The findings (Patel et al., 2014, Baran et al., 2014, and Hawraa et al., 2018) are consistent with this outcome.

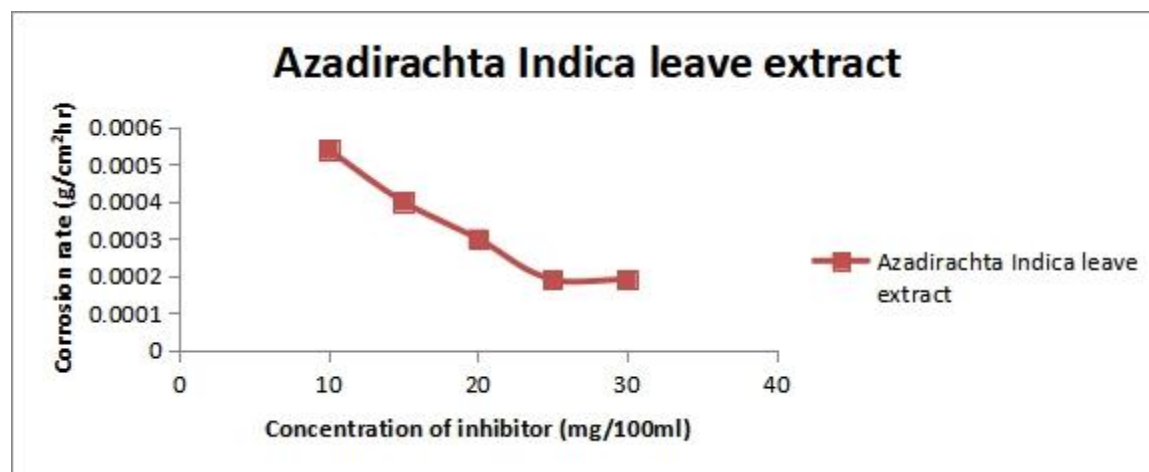


Fig 2. Impact of inhibitor concentration on corrosion rate in the presence and absence of different inhibitors.

Temperature's impact on the rate of corrosion

As seen in Fig. 3. the effect of temperature on the rate of mild steel corrosion in free acid and in the presence of various inhibitor concentrations (plant extract) was investigated in the temperature range of 30 °C to 70 °C under certain conditions. It was discovered that when temperature rose, mild steel corroded more quickly in both the presence of an inhibitor and in a free acid solution. This is to be expected since mild steel corrodes more quickly at higher temperatures due to an increase in the average kinetic energy of the interacting molecule. For example, the rate of corrosion in controlled acid solutions fell more than in unrestrained acid solutions. Nonetheless, the plant extract's ability to mitigate the mild steel's corrosion rate was the reason for the inhibited acid solution's superiority over the uninhibited one. This result was consistent with studies at the similar temperature range with a decrease in the corrosion rate from Vasudha et al. (2014) and Gaber et al. 2020. According to the study's findings, mild steel corrodes less quickly as leaf extract concentrations are increased (NorZakiah et al., 2014). This

suggests that the mild steel's inhibitory efficiencies in an acidic medium are increased by an increase in leaf extract (Leelavathi et al., 2013, Okafor 2007, Siddiqui et al 1984).

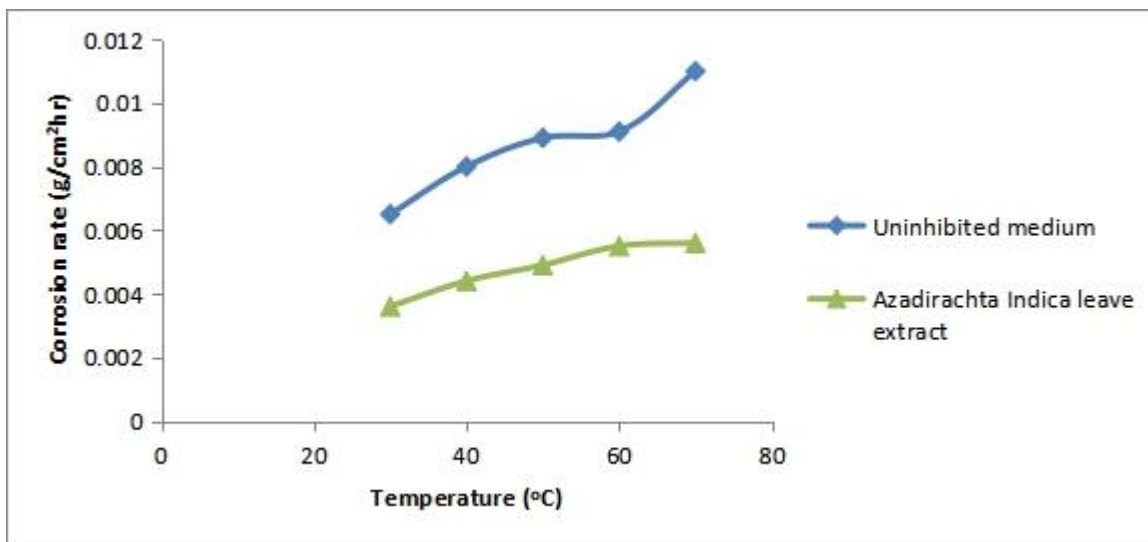


Fig.3. Temperature's impact on the rate of corrosion in presence and absence of different inhibitors.

Impact of medium concentration on corrosion rate of mild steel

The impact of medium concentration on mild steel corrosion rates in the presence and absence of different inhibitors under diverse circumstances is shown in Fig. 3. The pace at which unrestricted media corrode rises with the medium's concentration. The inhibited medium's corrosion rate first increased noticeably before declining. It is plausible that the concentration of inhibitor in the solution was adequate to offset the caustic nature of the highly concentrated acidic medium. Similar findings were obtained by (Rajesh et al., 2018) both in the presence and absence of the leaf extract. The study's conclusions showed that the inhibitor extract's presence offers rather effective protection when compared to the unrestricted substrate.

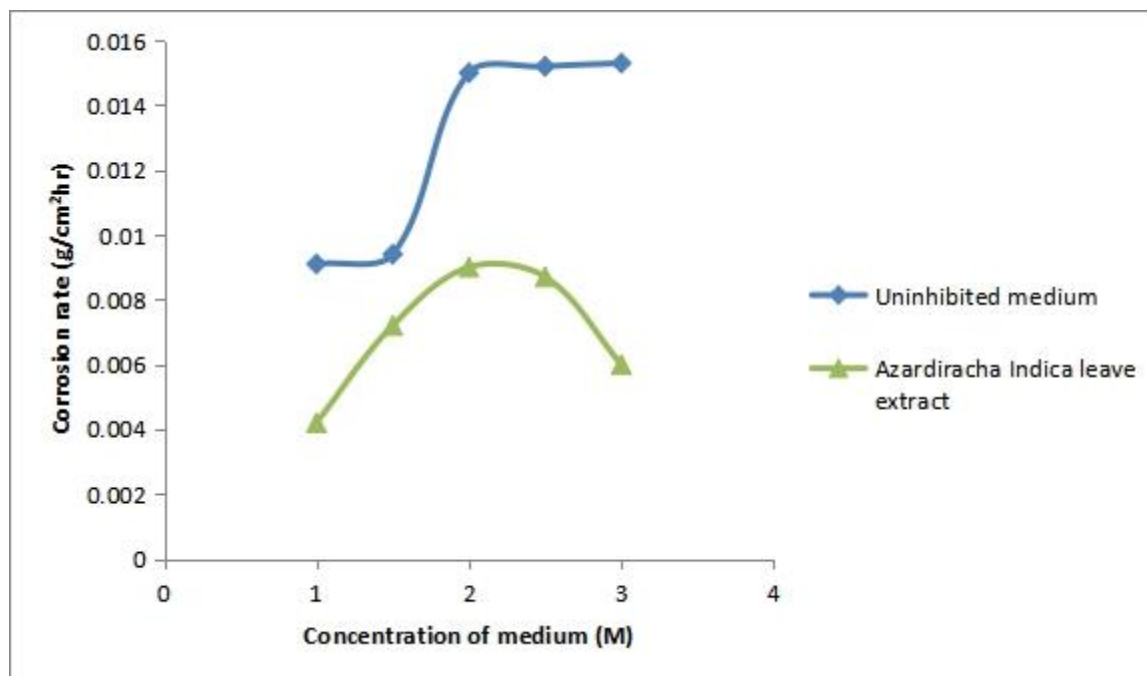


Fig. 3: Impact of medium concentration on corrosion rate of mild steel in the presence and absence of certain inhibitors.

SURFACE MORPHOLOGICAL STUDIES OF THE MILD STEEL.

Using a Sigma Field Emission Scanning Electron Microscope, the surface morphology of mild steel was examined. Figures 4 and 5 display pictures at 200 μ m of the surface of the mild steel in its untreated state and in the presence of inhibitors, respectively. (Muthukrishnan and associates, 2014). According to Anadebe et al. (2018) and Odejobi and Akinbulumo (2016), a SEM image aids in the examination of surface contaminations, offers qualitative chemical assessments, and identifies crystalline structures. A fast oxidized surface in the uninhibited media was shown to be an observational structural difference between the mild steel and the extracts (Leelavathi and Rajalakshmi, 2013, Mousavi et al., 2011, Munis et al., 2020, Olawale et al., 2018, Pal et al., 2019). According to Chang et al. (2020), Fu et al. (2010), Shukla et al. (2009), Vijayalakshmi et al. (2011), the SEM images also showed that the mild steel specimen immersed in the presence of inhibitor was in better condition, with a smooth surface covered with adsorbed inhibitor. In

contrast, the metal surface immersed in blank acid solutions was rough and appeared to be full of pits, cracks, and cavities.

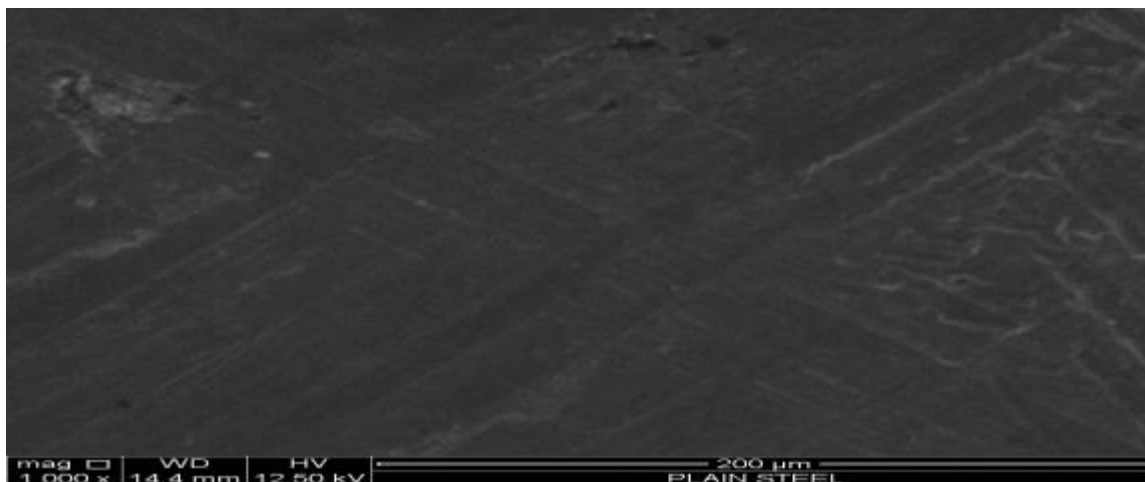
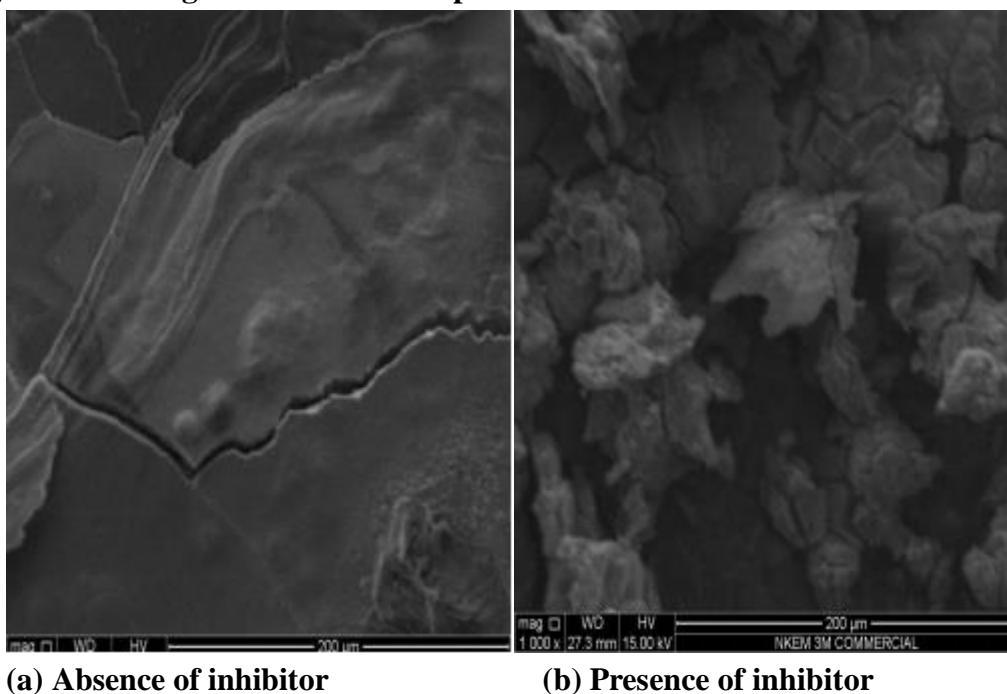


Fig. 4: SEM images of mild steel coupon



(a) Absence of inhibitor

(b) Presence of inhibitor

Fig. 5: SEM images of absence and presence of *Azadirachta indica*(Dogonyaro).

Conclusion;

The study concludes that mild steel corrosion in acidic media is inhibited by the ethanol extract of *Azadirachta indica* (dogonyaro) leaves. The photochemical components of the extracts were the cause of the ethanol extract of the leaf's inhibitory efficacy. The ideal parameters are: inhibitor concentration of 18.38 mg/100 ml, time of 5.55 h, and temperature of 47.9 oC; inhibitor concentration of 12.30 mg/100 ml, time of 7.90 h, and temperature of 47.4 oC. Inhibition efficacy in *Azadirachta indica* leaf inhibited acid media was 84.9% percent under ideal conditions. This suggests that leaf extract can prevent mild steel from corroding in an acidic environment. According to the study, harmless organic inhibitors are never favored over ecologically acceptable inhibitors.

RECOMMENDATIONS

Nigeria should take advantage of the economic feasibility of manufacturing environmentally acceptable inhibitors and using them to prevent metal corrosion. Oil firms and other industrial sectors could implement green inhibitor strategies to reduce or manage corrosion. The government should fund more research on green corrosion inhibitors in order to promote technological domestication and advance industrialization.

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