



## Corrosion Inhibition Study of Ethanolic Extracts of Neem Leaves (*Azadirachta indica*) on Zinc Metal In 0.1 M HCl and 0.1 M NaOH

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**Abstract:** Corrosion inhibitors use are prevailing in many industries to reduce the corrosion of metals and alloys in contact with a corrosive environment. Natural extracts have been generally used to protect metal materials from corrosion. The efficiency of these extracts as corrosion inhibitors is conventionally evaluated through electrochemical tests, which include techniques such as weight loss measurement. In this study the effect of extract of Neem (*Azadirachta indica*) leaves on the corrosion inhibition of zinc metal in 0.1M HCl and 0.1M NaOH solutions was studied. Soxhlet techniques were used for neem leaf extraction. While corrosion inhibition of zinc metal was studied using electrochemical and weight loss techniques. The experiment was performed in the test solution containing 0.1M HCl, 0.1M NaOH, and the neem extract of varying concentrations. The 0.1M solution was prepared by dissolving analytical reagents (AR) grade of HCl (37%) and 0.1M solution of NaOH base (40%) supplied in doubly distilled water. 1 ppm–5 ppm of the neem extract, which serves as the inhibitors, was also prepared. 100 mL of the test solution was used for the weight loss measurement. The result shows that neem extract was found to inhibit zinc corrosion in 0.1M HCl and 0.1M NaOH solutions at all temperature studies. An increase in the concentration (ppm) of extract solution decreases the rate of zinc corrosion in 0.1M of HCl and 0.1M NaOH solutions. Hence, it increases the inhibition efficiency. Corrosion rate increases with time but decreases with an increase in the concentration of extract solution. Finally the neem leaves extract was found to be an excellent potential corrosion inhibitor.

## 1. Introduction

Corrosion is the gradual destruction of material resulting from exposure and interaction with the environment. It is a major problem that must be confronted for safety, environment, and economic reasons (Pandian and Mathur 2008, Thompson *et al.*, 2017). Corrosion occurs when a substance or its qualities deteriorate owing to interactions between the substance and its surroundings. The tendency of a metal to corrode is determined by its grain structure, its composition as created during alloying, or the temperature developed during manufacture for deformation of a single metal surface

(Khadraoui *et al.*, 2014; Abdulrahman *et al.*, 2015; Beniken *et al.*, 2022). Given the importance of the environment in rusting, corrosion mechanisms can be as diverse as the settings, to which a substance is exposed, making them difficult to comprehend. Corrosion inhibitors can lower the high expense of replacing rusted metals, which is connected with corrosion. One of the ways of preventing/reducing corrosion of metals is the addition of inhibitors to the solution in contact with the metal surface in order to reduce the rate of corrosion (Quraishi *et al.*, 2010; Zarrouk *et al.*, 2012; Ogunleye *et al.*, 2020; Arrousse *et al.*, 2021).

Among the many methods of corrosion control/prevention, the use of corrosion inhibitors (Nagalakshmi and Sivasakthi, 2019, Nwosu *et al.*, 2014, Thakur and Kumar, 2021, Kumari and Lavanya, 2024, Solmaz *et al.*, 2023, Golshani *et al.*, 2022) is very much sought-after. Corrosion inhibitors are substances which, when added in small concentrations to corrosive media, decrease or prevent the reaction of the metal with the media. It would be more feasible to avoid corrosion rather than strive to eliminate it entirely. Several efforts have been made using corrosion preventive measures and the use of green corrosion inhibitors is one of them (Hossain, Asaduzzaman and Kchaou, 2021, Rani and Basu, 2012, Abdel-Karim and El-Shamy, 2022). The use of green inhibitors for the control of corrosion of metals and alloys which are in contact with acidic/basic environment is an accepted and growing practice. (Sanyal, 1981) in his review has given a vivid account of organic corrosion inhibitors including the classification and mechanism of action. He has attributed the corrosion inhibition potential to the donation of lone pair of electron to metal atoms. A number of heterocyclic compounds (Sheetal *et al.*, 2023, Goni *et al.*, 2021, Merdas and Hayal, 2021, Ahmed, Ali and Khadom, 2019) have been reported as corrosion inhibitors and the screening of synthetic heterocyclic compounds is still on-going.

Though many synthetic compounds showed promising anticorrosive activity, most of them are highly toxic to both human beings and environment. The safety and environmental issues of corrosion inhibitors arisen in chemical industries has always been a global concern. These inhibitors may cause temporary or permanent damage to internal organs for instance, kidneys or liver, or to disturb a biochemical process or to disturb an enzyme system at some site in the body. The toxicity may manifest either during the synthesis of the compound or during its applications. These toxic effects have led to the use of natural products as anticorrosion agents which are eco-friendly and harmless. Recently, many alternative eco-friendly corrosion inhibitors have been developed, they range from rare earth elements (De Damborenea *et al.*, 2014; Verma *et al.*, 2022) to organic compounds (Garcia *et al.*, 2011; Patel and Mehta, 2012; Palou *et al.*, 2014) found in plants.

Given their success story in literature, plant extracts have the potential to replace synthetic organic and inorganic inhibitors. The mechanism of action of green inhibitors depends on the structure of the active ingredient, and thus, many researchers have postulated many theories to explain this phenomenon (Barouni *et al.*, 2014; Ahmed, *et al.*, 2024).

(Saleh *et al.*, 1982) reported that *Opuntia ficus indica* and Aloe eru (leaves) and of Orange, Mango and Pomegranate (fruit-peels) give adequate protection to steel in 5% and 10% HCl at 25 and 40 °C. (Srivatsava *et al.*, 2018), also, tobacco, black pepper, castor oil seeds, acacia gum and lignin can be good inhibitors for steel in acid medium. In fact, the first patented corrosion inhibitors used were either natural product such as flour, yeast and so on or by products of food industries for

restraining iron corrosion in acid media (Sharma *et al.*, 2008). (Cabrera *et al.*, 2001) found that molasses treated in alkali solution inhibit the corrosion of steel in HCl used in acid cleaning (Mobin, Basik and Aslam, 2019). (Srivatsava and Sanyal, 1973) studied the performance of caffeine and nicotine in the inhibition of steel corrosion in neutral media. (Khamis *et al.*, 2016) has proved the use of herbs (such as coriander, hibiscus, anis, black cumin and garden cress) as new type of green inhibitors for acidic corrosion of steel (Ogunleye, Adeyemi and Oyegoke, 2011).

The corrosion inhibition activity in many of these phytochemicals could be due to the presence of heterocyclic constituents like alkaloids, flavonoids, terpenoids and soon. A brief insight of using Neem as corrosion inhibitor can be visualized by bibliometry using Scopus and VOSviewer to show the most authors and the countries developing researches on this kind of natural plants.

## **2. Methodology**

### **2.1 Sourcing and preparation of Neem (*Azadirachta Indica*) Leaves Extract**

The leaves of Neem (*Azadirachta Indica*) were sourced from Kogi State University, Anyigba. Then wash and air dried at room temperature for three weeks and 4 days. The air dried leaves were blended with electrical blending machine into powdery form. The sample of the powered leaves was taken to the department of pure and industry chemistry Kogi State University for further analysis.

#### **2.1.1 Preparation of the Extract of the leaves**

Using Soxhlex extraction method, the dried grinded leaves was weigh to be 200 g and was wrapped into an emery paper. About 950 mL of ethanol was poured into the round bottom flask, followed by the grinded leaf which was early wrapped into the extraction chamber.

The solvent was then heated from the bottom flask, evaporated, and passes through the condenser where it condenses and flow down to the extraction chamber and extracts the leaves by coming in contact consequently. When the leaves of solvent in the extraction chamber reach the top of the siphon, the solvent and the extracted plant material flow back to the flask.

The entire process continues repeatedly until the leaves are completely extracted. The extract was later subjected to a rotary evaporator in order to separate the solvent from the crude extract, at this point the volume of the original solvent used reduces.

#### **2.1.2 Preparation of the Zinc Metal Sample**

The Zinc metal samples with 2.0 cm x 2.0 cm dimension were mechanically cut and a hole of 2 mm diameter near the top corner of each coupon for weight loss experiment was drilled into it. Prior to the experiment, the coupons were washed with distilled water, degreased in acetone, dried and stored in desiccators.

## **2.2 Corrosion Study**

### **2.2.1 Test Solution**

In this present study, experiment was performed in the test solution containing 0.1M hydrochloric acid, 0.1M sodium hydroxide and the neem extract of varying concentrations. The 0.1M solution was prepared by dissolving analytical reagents (AR) grade of hydrochloric acid (37 %) and 0.1M solution of sodium hydroxide base (40 %) supplied in doubly distilled water. The concentration range of 1 ppm – 5 ppm of the neem extract which serves as the inhibitors was also prepared. The volume of

the test solution used for the weight loss measurement was 100 mL. From the initial and final weights of the zinc, the weight loss, corrosion rate (CR, ghr<sup>-1</sup> cm<sup>-2</sup>) inhibition efficiency (I.E %) and the degree of surface coverage (Ø) of the inhibitors were calculated using the formula (Eddy *et al.*, 2009):

$$\text{Corrosion rate} = \frac{\Delta W}{AT} \dots \dots \dots (a)$$

$$\text{Inhibition efficiency} = 1 - \frac{CR_1}{CR_2} \times 100 \dots \dots \dots (b)$$

$$\text{Surface coverage} = 1 - \frac{CR_2}{CR_1} \dots \dots \dots (c)$$

When Δw is the weight loss in gram, CR<sub>1</sub> and CR<sub>2</sub> are the corrosion rates of the rates of the zinc coupons in absence and presence of inhibitors respectively. A is the cross sectional area in cm<sup>2</sup> and t is the exposure time in hours.

### 2.2.2 Weight loss measurement

Weight loss measurement was carried out to determine the effects of time, concentration and temperature on the corrosion of the extract.

### 2.2.3 Effect of increase in concentration of inhibitor

The concentration of the extract was varied from 1 ppm – 5 ppm in 100 mL of 0.1M HCl and 0.1M NaOH at room temperature for 5days. Each sample was retrieved after every 24 hrs, washes with distilled water, rinsed in accentor, dried and reweighted. The averages of the triplicate analysis were recorded.

### 2.2.4 Effect of Time

The zinc coupons in triplicate were immersed in 100 mL breakers which separately contained 1 ppm – 5 ppm of the extract respectively, contained in 100 mL of 0.1M HCL and 0.1M NaOH and without the inhibitor (blank) for a period of 5days.

Previously weighted zinc sheet coupons were each suspended in each of the breakers with the help of a rope. The zinc coupons were retrieved from the solutions at 24 hrs interval. Progressively for 120 hrs (5days). Each retrieved coupon was immersed in 100 mL of distilled water, washed properly to remove the corrosion product, dried in acetone and then reweighted. The weight loss was determined by subtracting the final weight (after suspension). Averages of the triplicate analysis were recorded.

### 2.2.5 Effect of temperature with respect to corrosion

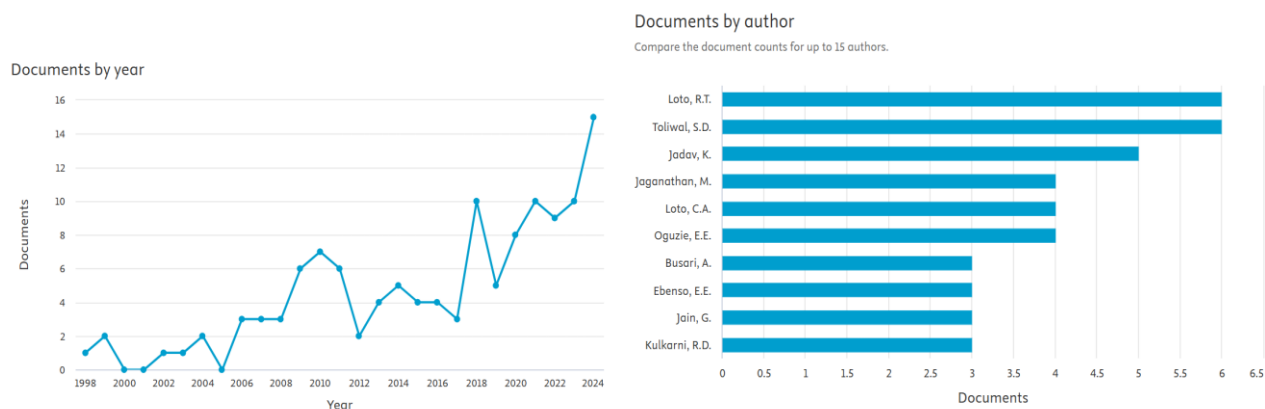
The zinc coupons were each suspended in a beaker with the aid of a rope containing 100 mL of the 0.1M HCl and 0.1M NaOH with 4ppm the extract concentrations and without the inhibitor (blank). The beaker was immersed in water bath at various temperatures (303, 313, 323, 333 and 343 k) for 4 hours. Each retrieved coupon was immersed in 100 mL of distilled water, washed properly to remove the corrosion product, dried in acetone and then reweighted. The weight loss was then determined.

## 3. Results and Discussion

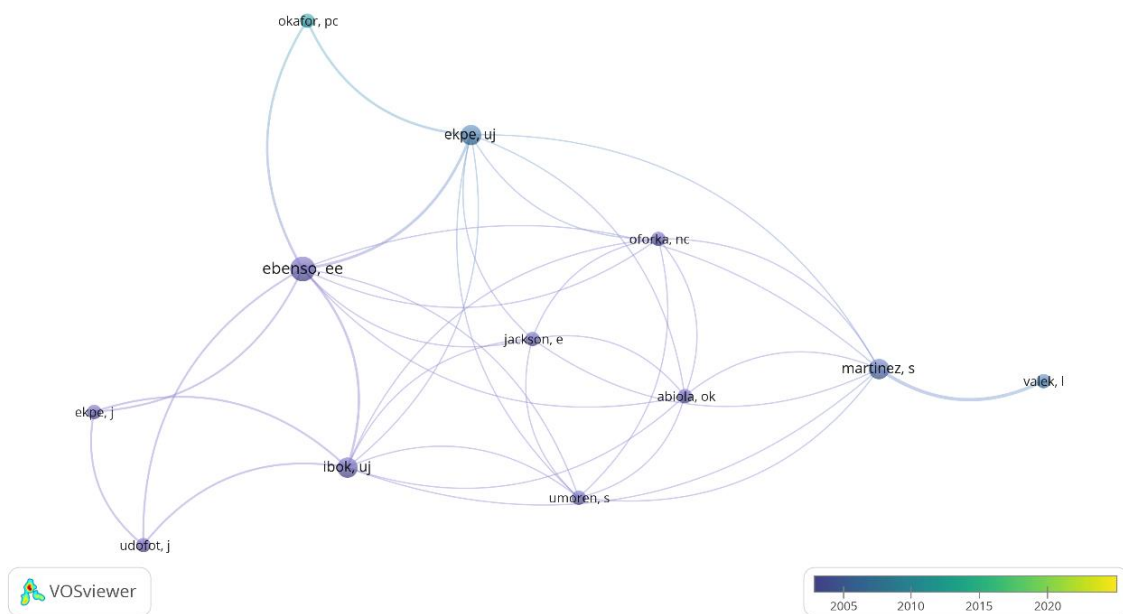
The Scopus database and VOSviewer may be excellent tools for mapping the research area in an easy and understandable pattern for many users, evaluating or reorienting the deciders in various organizations, several funders, sponsors, practitioners, and other academicians and social scientists

(Alharti *et al.*, 2023; N'daye *et al.*, 2022; Rodriguez-Marin *et al.*, 2022). Search through Scopus databases “Corrosion and Neem or *Azadirachta Indica*” gave 125 documents during more than 25 years, as shown in **Scheme 1 (a)**. Scopus offers several analyses as the most published authors are also shown in **Scheme 1 (b)**, indicating that both Loto R.T. from Covenant University, Ota, Nigeria and Toliwal, S. D. Institute of Science & Technology for Advanced Studies & Research, Vallabh Vidyanagar, India, publishing 7 documents. The most cited authors are: Oguzie’s paper published in Corrosion Science (2008, 372 citations), (Oguzie, 2008) followed by that of (Valek & Martinez, 2007) (156 citations), the use of *Azadirachta* as copper corrosion inhibitor in 0.5M sulphuric acid cited 149 times (Ofakor, Ebenso & Ekpe, 2010), (**Scheme 2**).

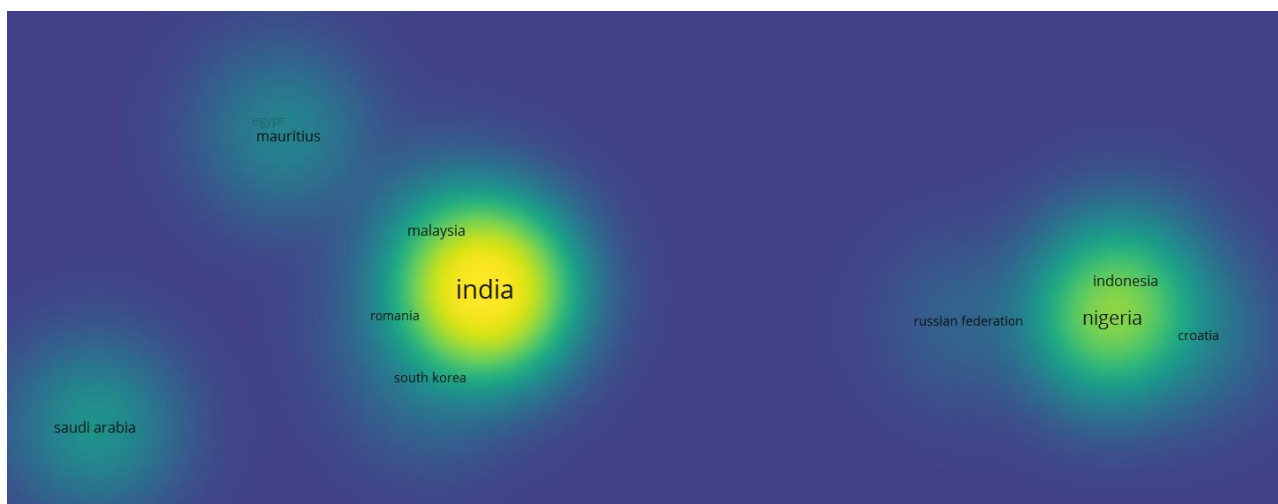
Based on the analysis performed on co-authorship, India was identified as having the highest affiliation linked to Romania, Malaysia & South-Korea countries. The list was followed by Nigeria's collaboration with Indonesia, Russia and Croatia. The latest country is Saudi Arabia (**Scheme 3**).



**Scheme 1: (a):** Evolution of the publication (1998-2024) and **(b)** the authors most published



**Scheme 2:** A bibliometric map fabricated according to the co-authorship in network visualisation mode (VOSviewer)



**Scheme 3:** A bibliometric map fabricated according to the countries in density visualization mode (VOSviewer)

#### 4.1.1 Effect of concentration and immersion time.

The weight loss of analysis of Zinc in 0.1M HCl and 0.1M NaOH is presented in Tables 1 and 2 above, the plot of weight loss against time and weight loss against concentration in the presences and absences of inhibitors in 0.1M HCl solution (figure 1 and 2) and that of NaOH solution in (figure 3 and 4) shows that, there's a decrease in weight as the concentration of the inhibitor increase (1 ppm- 5 ppm), and also the weight loss increase as time increase for both medium.

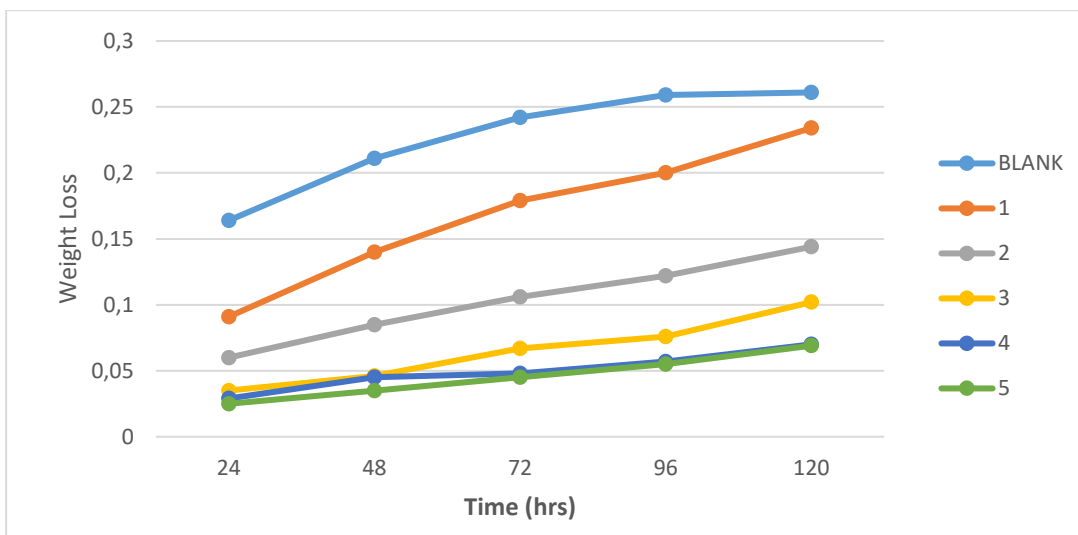
**Table 1:** Variation in weight loss in the presence and absence of Inhibitor in 0.1M HCl

| Weight loss in the presence and absence of Inhibitor in 0.1M HCl |                     |            |       |       |       |       |
|--|---------------------|------------|-------|-------|-------|-------|
| Inhibitor  | Concentration (ppm) | TIME (HRS) |       |       |       |       |
|  |                     | 24         | 48    | 72    | 96    | 120   |
|  | Blank               | 0.164      | 0.211 | 0.242 | 0.259 | 0.261 |
|  | 1                   | 0.091      | 0.140 | 0.179 | 0.200 | 0.234 |
| Neem (Azadirachta Indica) Extract                                | 2                   | 0.060      | 0.085 | 0.106 | 0.122 | 0.144 |
|  | 3                   | 0.035      | 0.046 | 0.067 | 0.076 | 0.102 |
|  | 4                   | 0.029      | 0.045 | 0.048 | 0.057 | 0.070 |
|  | 5                   | 0.025      | 0.035 | 0.045 | 0.055 | 0.069 |

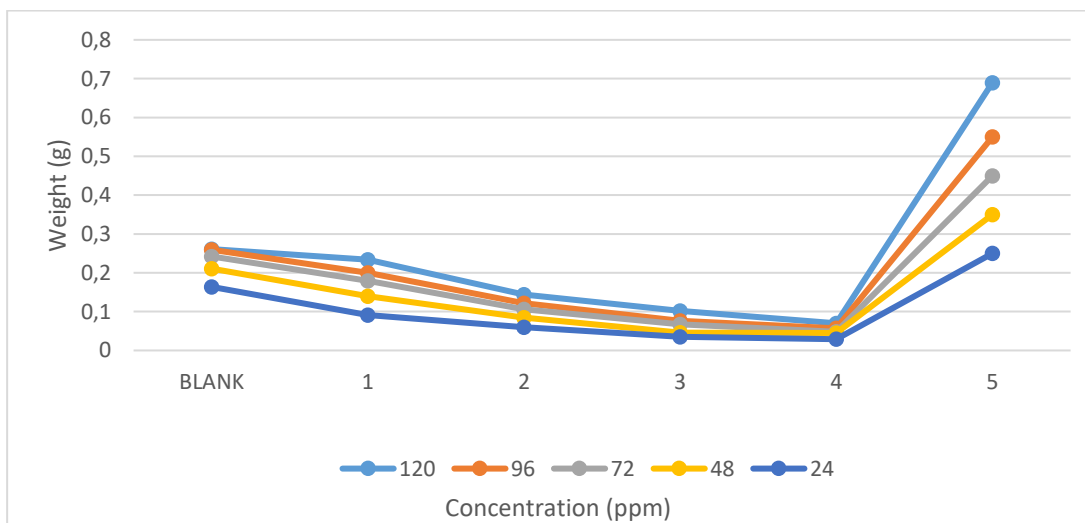
**Table 2:** Weight loss in the presence and absence of Inhibitor in 0.1M NaOH

| W eight loss in the presence and absence of Inhibitor in 0.1M NaOH |                     |            |       |       |       |       |
|--|---------------------|------------|-------|-------|-------|-------|
| Inhibitor  | Concentration (ppm) | TIME (HRS) |       |       |       |       |
|  |                     | 24         | 48    | 72    | 96    | 120   |
|  | Blank               | 0.020      | 0.026 | 0.038 | 0.047 | 0.062 |
|  | 1                   | 0.017      | 0.019 | 0.024 | 0.032 | 0.052 |
| Neem(Azadirachta Indica) Extract                                   | 2                   | 0.012      | 0.017 | 0.022 | 0.028 | 0.039 |
|  | 3                   | 0.009      | 0.013 | 0.018 | 0.025 | 0.030 |
|  | 4                   | 0.006      | 0.010 | 0.014 | 0.020 | 0.025 |
|  | 5                   | 0.001      | 0.006 | 0.009 | 0.016 | 0.020 |

Which implies that, the decrease of weight as the concentration increase may be due to the adsorption of the hetero-atom presence in the Neem leave extract on the surface of the Zinc metal. And this indicate that the inhibitor were effective in acid and in base medium as the concentration increase from 1ppm – 5ppm.

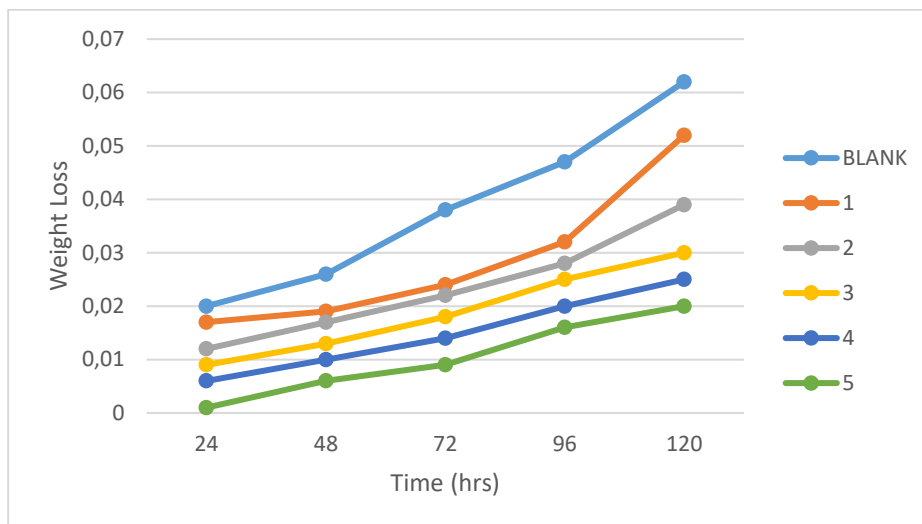


**Figure 1:** Variation of corrosion rate of zinc with time in 1 M HCl

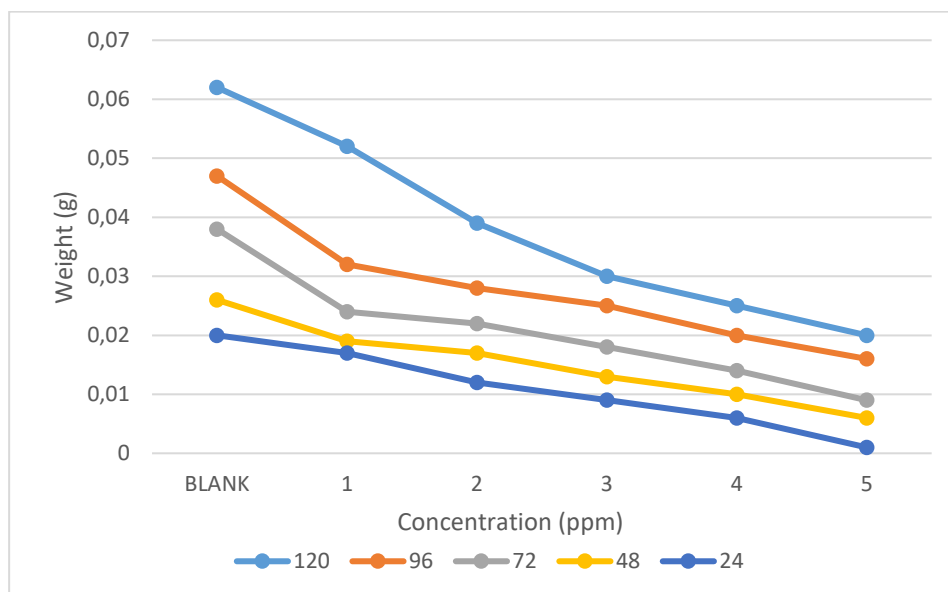


**Figure 2:** Variation of corrosion rate of zinc in 1 M HCl at different concentrations

Table 3 and 4, shows decrease in the corrosion rate from low concentration to high concentration in 0.1M HCl containing the inhibitors and without the inhibitor (blank) as well as 0.1M NaOH solution. This is as a result of adsorption effect of the hetero-atom on corrosion rate of zinc and with a consequent increase in the surface coverage as well as the inhibition efficiency (Table 3 and 4). And this indicate that the rate at which corrosion rate for Zinc in HCl, H<sub>2</sub>SO<sub>4</sub> and NaOH decrease on the addition of more of the inhibitor (Neem leave extract), which is in line with a report work by (Nahlé *et al.*, 2010; Sharma *et al.*, 2009). This process blocks the active site and causes a decrease in the rate of corrosion attack when the inhibitor concentration increases.



**Figure 3:** Variation of corrosion rate of zinc with time in 1 M NaOH.



**Figure 4:** Variation of corrosion rate of zinc in 1 M NaOH at different concentrations.

**Table 3:** Corrosion parameters for zinc in 0.1M HCl.

| Corrosion Rate, Inhibition Efficiency, and Surface Coverage for 24 hrs of immersion in 0.1M HCl |                                     |                          |                      |
|---|-------------------------------------|--------------------------|----------------------|
| Concentration (ppm)   | Corrosion Rate g/cm <sup>2</sup> /h | Inhibition Efficiency(%) | Surface Coverage (Ø) |
| Blank   | $1.71 \times 10^{-3}$               | -                        | -                    |
| 1   | $0.95 \times 10^{-3}$               | 179                      | 0.44                 |
| 2   | $0.63 \times 10^{-3}$               | 270                      | 0.63                 |
| 3   | $0.37 \times 10^{-3}$               | 461                      | 0.78                 |
| 4   | $0.30 \times 10^{-3}$               | 569                      | 0.82                 |
| 5   | $0.26 \times 10^{-3}$               | 656                      | 0.84                 |



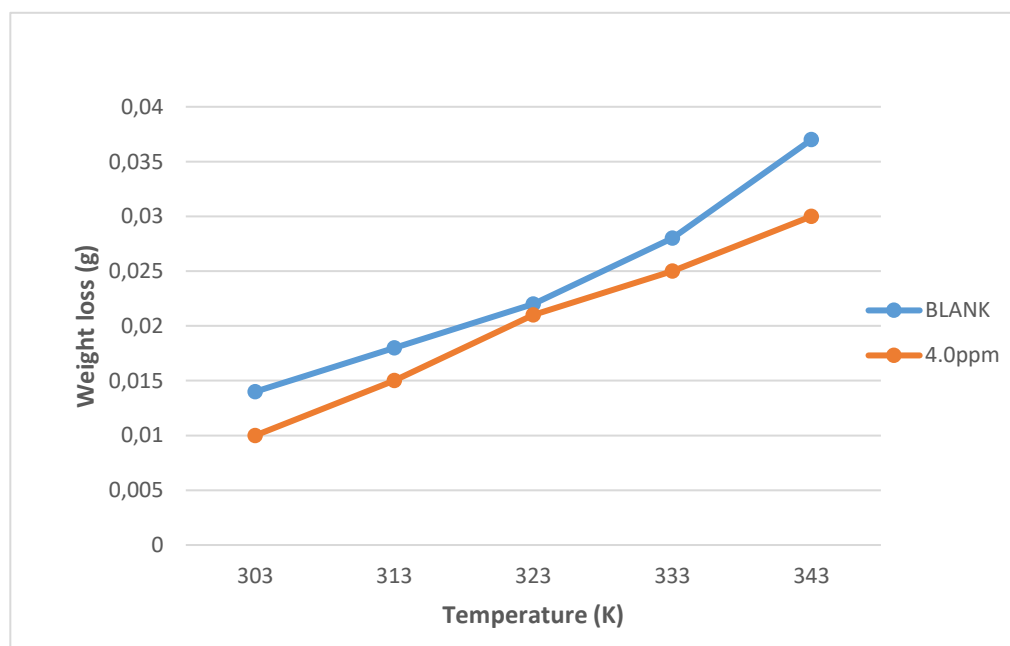
**Table 4:** Corrosion parameters for zinc in 0.1M NaOH

| Corrosion Rate, Inhibition Efficiency, and Surface Coverage for 24 hrs of immersion in 0.1M NaOH |                                     |                          |                      |
|--|-------------------------------------|--------------------------|----------------------|
| Concentration (ppm)  | Corrosion Rate g/cm <sup>2</sup> /h | Inhibition Efficiency(%) | Surface Coverage (Ø) |
| Blank  | $2.08 \times 10^{-4}$               | -                        | -                    |
| 1  | $1.77 \times 10^{-4}$               | 116                      | 0.15                 |
| 2  | $1.25 \times 10^{-4}$               | 165                      | 0.39                 |
| 3  | $0.94 \times 10^{-4}$               | 220                      | 0.55                 |
| 4  | $0.63 \times 10^{-4}$               | 329                      | 0.69                 |
| 5  | $0.10 \times 10^{-4}$               | 407                      | 0.95                 |

Table 5 and 6 (Figure 5 and 6) shows that weight loss increase with increase in temperature, this implies that the increase in temperature will enhance corrosion therefore corrosion materials are to be prevented from excessive temperature. It was form that, the rate of corrosion of zinc in the blank and in the inhibited acid and base solution increase as the temperature increase.

**Table 5:** Effect of temperature and weight loss in 0.1M HCl

|                            | Effect of temperature and weight loss in 0.1M HCL |       |       |       |       |
|----------------------------|---|-------|-------|-------|-------|
|                            | Temperature (k)                                   |       |       |       |       |
|                            | 303   | 313   | 323   | 333   | 343   |
| Blank                      | 0.014   | 0.018 | 0.022 | 0.028 | 0.037 |
| Azadirachta indica Extract | 0.010   | 0.015 | 0.021 | 0.025 | 0.030 |

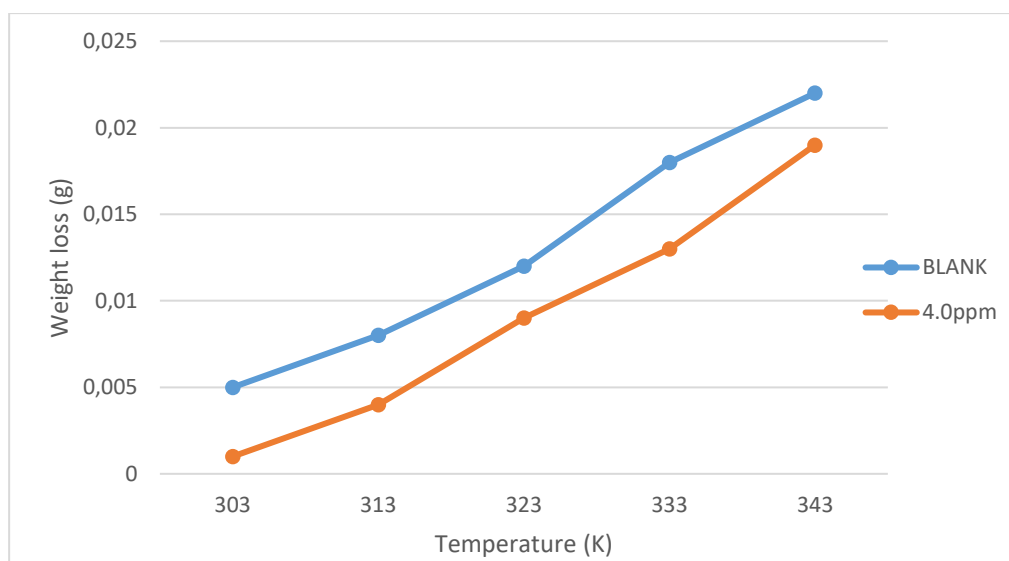


**Figure 5:** Variation of corrosion rate of zinc in 1 M HCl at different temperatures

**Table 6:** Effect of temperature and weight loss in 0.1M NaOH

| Effect of temperature and weight loss in 0.1M NaOH  |                 |       |       |       |       |
|---|-----------------|-------|-------|-------|-------|
|   | Temperature (k) |       |       |       |       |
|   | 303             | 313   | 323   | 333   | 343   |
| <b>Blank</b>  | 0.005           | 0.008 | 0.012 | 0.018 | 0.022 |
| <b>Neem (<i>Azadirachta indica</i>)<br/>Extract</b> | 0.001           | 0.004 | 0.009 | 0.013 | 0.019 |

The examination of literature showed that the ethanolic extract is rich in polyphenols, flavonoids ... various molecules were characterized as quinic acid, malic acid, tr-Aconitic acid, Gallic acid, Chlorogenic acid, Protocatechuic acid, Tannic acid, tr-caffeic acid, Vanillin, p-Coumaric acid, Rosmarinic acid, Rutin, Hesperidin, Hyperoside, 4-OH Benzoic acid, Salicylic acid, Myricetin, 18: Fisetin, Coumarin, Quercetin, Naringenin, Hesperetin, Luteolin, Kaempferol, Apigenin, Rhamnetin, Chrysin... (Boukeloua *et al.*, 2024). The inhibitory effect of extract is generally explained by the synergistic intermolecular effect of the molecules due to the presence of aromatic rings, double and triple bonds and heteroatoms (S, N, O...) (Lrhoul *et al.*, 2023)

**Figure 6:** Variation of corrosion rate of zinc in 1 M NaOH at different temperatures

### Conclusion

The inhibitive properties of Neem Leave extract on zinc metal surface corrosion in 0.1m of HCL and 0.1M NaOH at varying temperature, time and concentration using weight loss method was revealed that the extract was found to inhibit zinc corrosion in 0.1M HCl and 0.1M NaOH solutions at all temperature studies, increase in the concentration (ppm) of extract solution decreases the rate of zinc corrosion in 0.1M of HCL and 0.1M NaOH solutions. Hence, increases the inhibition efficiency. Finally, corrosion rate increase with time but decreases with increase in the concentration of extract solution.

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**Disclosure statement:** *Conflict of Interest:* The authors declare that there are no conflicts of interest.

*Compliance with Ethical Standards:* This article does not contain any studies involving human or animal subjects.

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