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# Effect of Neem and Bitter Leaf Extract on the Corrosion Penetration Rate of 0.19% Medium Carbon Steel in Hydrochloric Acid

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**ABSTRACT**

*An important economical method of protecting a metallic material like steel against deterioration from corrosive attack is by efficient application of inhibitors. At the core of this research is investigation of the corrosion responses or behavior of the medium carbon steel subjected to degrading media of hydrochloric acid of varied concentration. It was inhibited with Neem and Bitter-leaf extracts individually as well as Neem-Bitter leaf mixture which is the synergized extract. Varied concentration of Neem–Bitter leaf extracts as well as the Synergized extract, were tested as corrosion inhibitors for steel coupons in 1.5 M, 2.0 M and 2.5 M hydrochloric acid and subsequently analyzed via weight loss techniques. The maximum percentage inhibition efficiency was recorded as 90% at 1.5 M hydrochloric acid on the 28$^{th}$ day of immersion. This is an indication that the synergistic effect of neem-bitter leaf formulation used as inhibitor can produce a better result in reducing corrosion of hydrochloric acid when compared with the individual extracts of neem and bitter leaf.*



## 1. INTRODUCTION

Owing to its versatility due to carbon presence, steel has been heavily employed at the core of constructions and several installations. Field experience has shown that the sharpest impediment to the health of steel in service is corrosion mostly induced by acidic and environmental attack (Bailey and Peterson, 2011). Traces of acid left behind decomposes electrochemically into uninhibited hydrogen ions and preferentially discharged oxygen species which precipitates a differential potential that drives corrosion (Okafor and Ebenso, 2007). Steel has been a core engineering alloy most especially from the outset of the industrial revolution of many years ago. Properties like microstructural integrity, mechanical duty and crystalline tenacity has led to its increased participation in the small, medium and heavy industries (Bailey and Peterson, 2011).

In this research, the plant extracts of *Vernonia amygdalina* (Bitter leaf) and *Azadirachta indica* (Neem) were used because of the phytochemical presence of potentially sufficient unshared electrons in the polar groups in the continuous functional chain (Onyeka and Nwambekwe, (2007). Neem contains a recurring phenol-based compound with thousands of nitrogen atoms in the spatial chains known to prevent oxidative deterioration on the metal, Bitter-leaf similarly contains benzene based structural compounds of pyridine with several heteroatoms of oxygen, sulphur and potassium which extends or reduces evolution of hydrogen gas to form corrosion cells on the metal. (Loto et. al., 2014). Bitter leaf and Neem have high density presence of lone pair heteroatoms, these pairs are spatially attached to the contained compounds of alkaloid, terpenes, tannins, and anthraquinones on their benzene-based chain (Onyeka and Nwambekwe, 2007). These lone pairs practically reduce the electron deficit on the deteriorating steel surface by creating passivation or near inert film.

## 2. MATERIALS AND METHODS

### 2.1. Materials

The materials used include 0.19% medium carbon steel, procured from Cutix steel dealership in Lagos State. The bitter leaf and Neem extract, 37% analytical grade (AR) concentrated hydrochloric acid. Equipment used include high precision electronic weighing balance, industrial desiccator. Avery Denilson electromechanical tensile testing machine model 250S with sensitive clip-on extensometer.

### 2.2. Methods

#### 2.2.1. Preparation of Neem and Bitter leaf extracts

Neem plant (seed and leaves) were collected, washed, then chopped and squeezed in de-ionized water to produce a dark green juice. About 250 ml was finally recovered by refluxing with 10 ml of ethanol which was reported to stimulate the release of active anti-corrosion (Zucchi and Omar, 1985). Same process was followed for bitter-leaf. About 125 ml each of bitter leaf and neem extract were then mixed to form a synergized extract for the work.

#### 2.2.2. Preparation of hydrochloric acid

Analytical grade (AR) concentrated stock solution of hydrochloric acid was diluted with double distilled water to yield concentrations of 1.5 M, 2.0 M and 2.5 M.

#### 2.2.3. Preparation of 0.19% medium carbon steel corrosion specimen

Preparation of the medium carbon steel was based on ASTM G1-G4 and ASTM RP 00175 which is the standard practice for preparing corrosion specimen (ASTM G1- 03, 2011). A cylindrical medium carbon steel of diameter 14 mm was mechanically cut into 30 coupons of individual length 10 cm employed for the weight loss investigation. Another 30 coupons for weight loss were cut to length of 50 cm and machined. These samples were mounted on the grips of a vice and abrasively treated with silicone or Emeryl cloth of different grit size. 240 grit grade was firstly used to rid the metal surface of inhomogeneous surfaces and for the removal of saw cuts. It was subsequently followed by 320, 400 and 600 grit abrasive papers to further remove strain raisers that could facilitate corrosion. Final surface neatness was arrived by polishing to mirror finish by the careful use of a grinding wheel (Onyekpe, 2002). The specimen then cleaned with dry pieces and swabbed in an etching solution acetone, then stored in a desiccator prior to usage.

### 2.2.4. Corrosion studies

Beakers labelled A, B, C and D were filled with 1.5 M, 2.0 M, 2.5 M hydrochloric acid molarity and de-ionized water and 30 medium carbon steel samples each were carefully immersed in these beakers and covered for an immersion period of 98 days. Another set of four beakers were filled with 1.5 M, 2.0 M, 2.5 M hydrochloric acid molarity and de-ionized water respectively and 30 medium carbon steel samples each were carefully immersed in these beakers. Also, 10 ml of neem extract was added to the beakers for every 14 days which was the withdrawal period for the samples, withdrawal of samples in this interval continued till the 98th day. The same process was maintained for the other extracts of bitter leaf and synergized extracts in their own media.

### 2.2.5. Weight loss analysis

The difference in weight between the pre-weighed (before immersion) samples and when withdrawn from their native corrosive media of hydrochloric acid is the basis for the weight loss technique. The samples withdrawn from the corrosive media as well as from the inhibited media were weighed in a high precision electronic balance. To reduce margin of error in measured outcome, averages of three samples were taken to be the valid weight loss. Weight loss values were obtained from Equation (1).

$$\text{Weight loss} = W_a - W_b \tag{1}$$

$W_a$= Weight of carbon steel specimen before immersion, $W_b$= Weight of carbon steel specimen after immersion

### 2.2.6. Corrosion penetration rate

The corrosion penetration rate of 0.19% carbon steel is best explained in terms of the rapidity with which electrons leaves the metal within the hydrochloric acid media resulting to sectional decrease in thickness. This was investigated for effect on the samples in the absence and presence of the Neem and Bitter leaf extracts. It is calculated using Equation (2).

$$CPR = \frac{KW}{\rho_{AT}} \tag{2}$$

Where K is the corrosion constant equivalent to 87.6, W= Weight loss value in g, $\rho$=Density of the carbon steel = 7.85 g/cm$^3$, A= Cross-sectional area of the cylindrical steel =$\pi d^2/4$

### 2.2.7. Inhibition efficiency

To determine the level of corrosion susceptibility of the steel in acid when the Neem bitter complex is applied, calculation of percentage efficiency was done using Equation (3).

$$\text{I.E} = \left[\frac{W_1 - W_2}{W_2}\right] 100 \tag{3}$$

Where I.E = is the inhibition efficiency. $W_1$= is the weight loss of steel coupons immersed in different hydrochloric acid concentration without inhibitor. $W_2$= is the weight loss of metals in inhibitor medium.

## 3. RESULTS AND DISCUSSION

### 3.1. Weight Loss Investigation

#### 3.1.1. Samples immersed in ordinary hydrochloric acid

Figure 1 shows increase in weight loss with the samples immersed in the hydrochloric acid with the varying acid concentration of 1.5 M, 2.0 M and 2.5 M. Thus, weight loss can be said to be directly related to the rate of decomposition of the samples based on the aggressiveness of the media. For instance, on the 98$^{th}$ day of immersion, 1.5 M concentration of hydrochloric acid resulted in the weight loss of 0.52g of medium carbon steel, 2.0 M of acid concentration led to 0.67 g loss and 0.72 g was the deficit in 2.5 M of the acid.

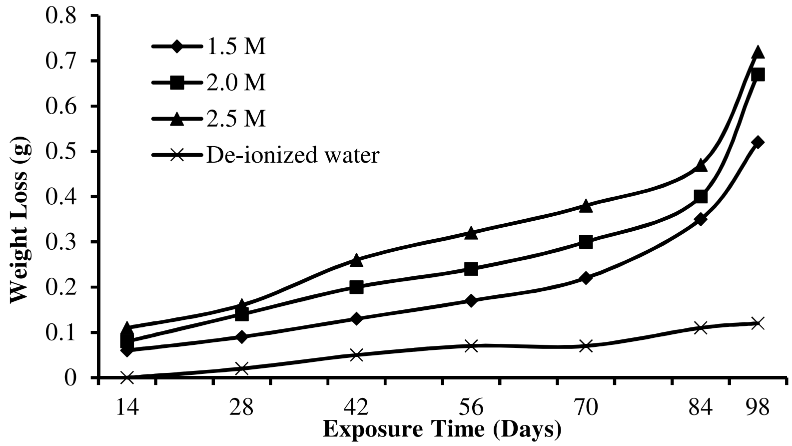


Figure 1: Weight loss against exposure time for samples immersed in ordinary hydrochloric acid

#### 3.1.2. Samples immersed in hydrochloric acid inhibited with Neem extract

Figure 2 shows that hydrochloric acid equally corroded the samples inhibited with Neem but at a slightly reduced rate especially when compared to the results for samples immersed in ordinary hydrochloric acid in Figure 1. The weight loss of samples for the first 14 days immersed in 1.5 M hydrochloric acid inhibited by 10 ml of Neem inhibitor was 0.04 g. On the 98$^{th}$ day of immersion, the weight loss had increased to 0.38 g unlike samples immersed in ordinary hydrochloric acid of same concentration which was 0.06 g and 0.52g respectively for samples withdrawn on the 14$^{th}$ and 98$^{th}$ day respectively. For coupons immersed in 2.5 M of hydrochloric acid at 10 ml of Neem inhibitor, the weight loss was reduced to 0.08g while it was 0.11 g for the samples in ordinary hydrochloric acid medium. The reduction in the weight loss values for the neem inhibited samples arose from the electrostatic adsorption of neem molecules onto the metal. This is to suggest that application of Neem extract on the samples gave rise to a reduced dissolution rate of the samples (Umoren et al., 2013). It equally led to the temporary passivity the medium carbon steel metal possessed in hydrochloric acid (Umoren et al., 2013).

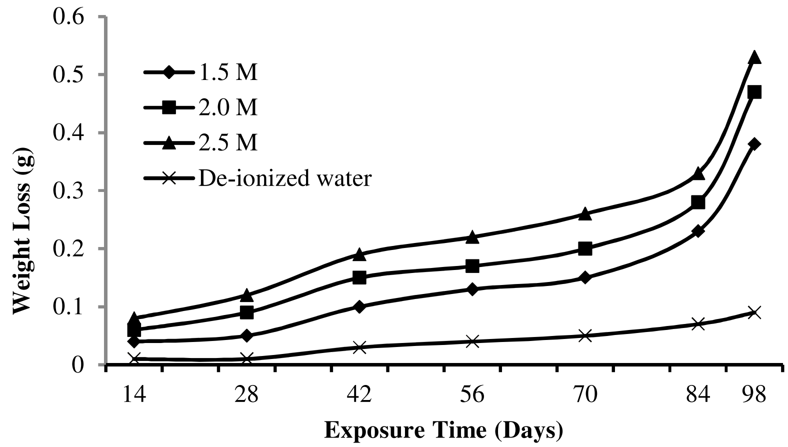


Figure 2: Weight loss against exposure time for samples immersed in hydrochloric acid inhibited with neem extract

### 3.1.3. Samples immersed in hydrochloric acid inhibited with bitter leaf extract

Figure 3 shows that bitter leaf inhibited samples were also attacked by the hydrochloric acid but at a reduced rate if particularly compared to Figure 1. On the 98$^{th}$ day of immersion in 2.5 M hydrochloric acid inhibited with bitter leaf extract, the weight loss of the metal was 0.58 g compared to the 0.72 g obtained from the samples immersed in ordinary hydrochloric acid of same concentration. This slight reduction in weight loss value arose from the adhering of Bitter-leaf compounds onto specific sites on the medium carbon steel samples. As a result, the samples became slightly passive and led to lower dissolution rate in hydrochloric acid and invariably lower weight loss (Onyeka and Nwambekwe, 2007).

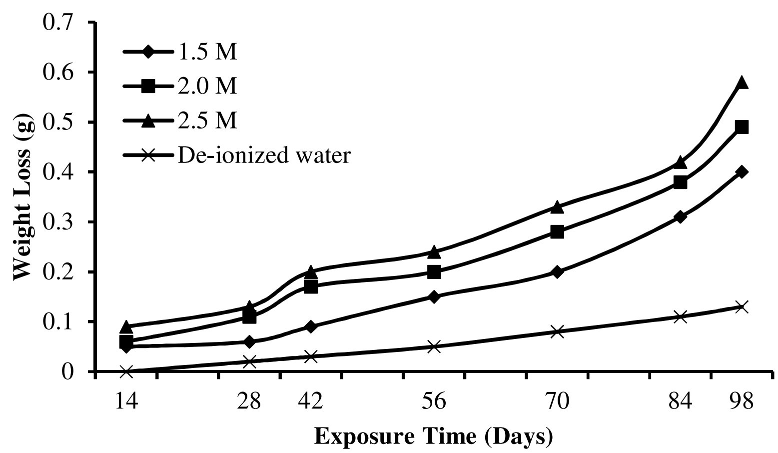


Figure 3: Weight loss against exposure time for samples immersed in hydrochloric acid inhibited with bitter-leaf extract

### 3.1.4. Samples immersed in hydrochloric acid inhibited with synergized extract

Figure 4 shows that the synergized extract produced a marked reduction in the weight loss values of coupons when compared to the weight loss values offered individually by neem extract as well as bitter leaf extract. For instance, in 2.5 M of hydrochloric acid, by the first 14 days of immersion, weight loss of coupons was

0.11 g in the ordinary hydrochloric medium and weight losses for the inhibitors of Neem and Bitter-leaf was recorded as 0.08 g and 0.09 g respectively while for the synergistic inhibitor the weight loss was 0.04 g. In the same vein, the 98th day of sample immersion in 2.5 M hydrochloric acid produced a weight loss of 0.72g in the medium without any inhibitor and weight losses of 0.53 g and 0.58 g in neem and bitter leaf inhibitors respectively while for the synergized extract the weight loss was 0.26 g. These points to the synergistic effect of the polar compounds from the extracts of Neem and Bitter-leaf in facilitating a better adsorption process and greatly slowing down the acid attack (Onyeka and Nwambekwe, 2007).

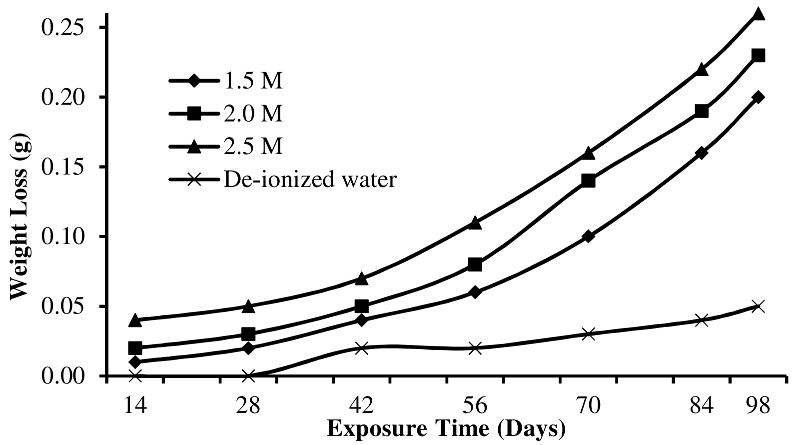


Figure 4: Weight loss against exposure time for samples immersed in hydrochloric acid inhibited with synergized extract

### 3.2. Corrosion Penetration Rate

#### 3.2.1. Samples immersed in ordinary hydrochloric acid

The corrosion penetration rate defines the rapidity at which decomposition of the medium carbon steel takes place within the media of hydrochloric acid. From figure 5, the corrosion penetration rate shows a trend of fluctuation from the first 14 days of immersion up to the 98th day.

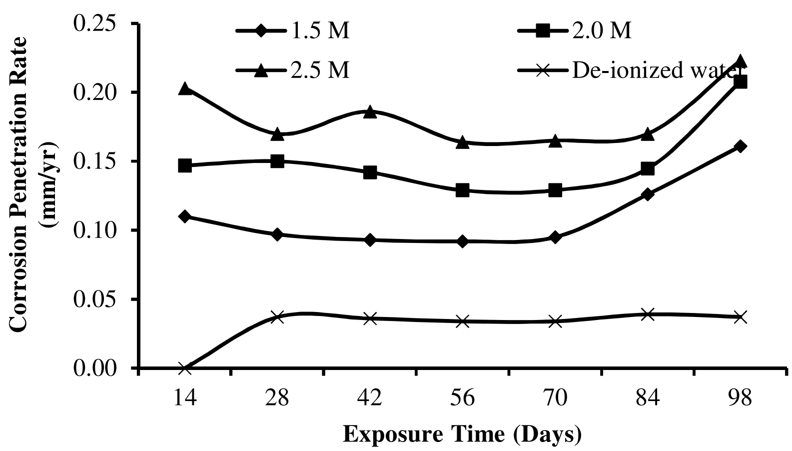


Figure 5: Corrosion penetration rate against exposure time of samples in hydrochloric acid media

For 2.5 M hydrochloric acid, the metal samples showed a rapid corrosion penetration rate of 0.203 mm/yr for the initial 14 days and the rate decreased again and shot high to 0.223 mm/yr on the 98th day of immersion and retrieval. The rate of decomposition of metal was very high due to persistent release of hydrogen ion from the acid onto the metal (Onyeka and Nwambekwe, 2007).

### 3.2.2. Samples immersed in hydrochloric acid inhibited by neem extract

In Figure 6, the corrosion penetration rate inhibited by only neem extract followed identical fluctuation trend with those in the acid without inhibitor. The adsorption of neem compounds onto the metal surface resulted in the lower corrosion penetration rate values compared to the un-inhibited samples in Figure 5. The corrosion penetration rate was 0.203 mm/yr. on the first 14 days for the samples in the blank acid media and 0.173 mm/yr for Neem inhibited samples in the same 14 days. Again, it was 0.223 mm/yr for samples in the blank media of hydrochloric acid at the 98th day as against 0.163 mm/yr obtained for the samples protected by the Neem inhibitor.

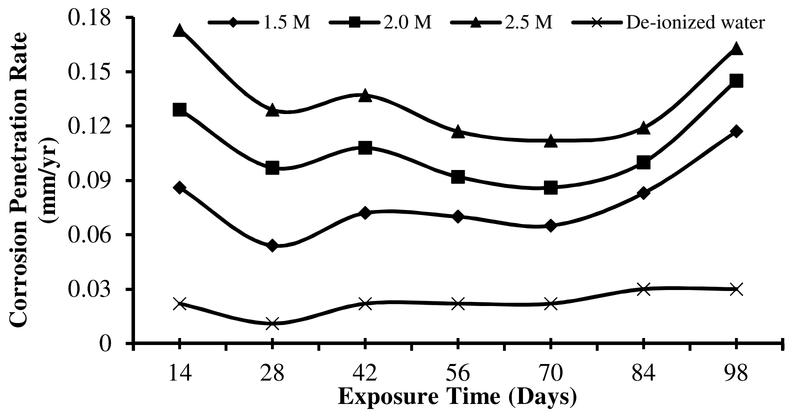


Figure 6: Corrosion penetration rate against exposure time of coupons inhibited by Neem

### 3.2.3. Samples immersed in hydrochloric acid inhibited by bitter leaf extract

The corrosion penetration curve in Figure 7 shows sinusoidal behavior as the exposure time of coupons to media of hydrochloric acid inhibited with Bitter-leaf increases.

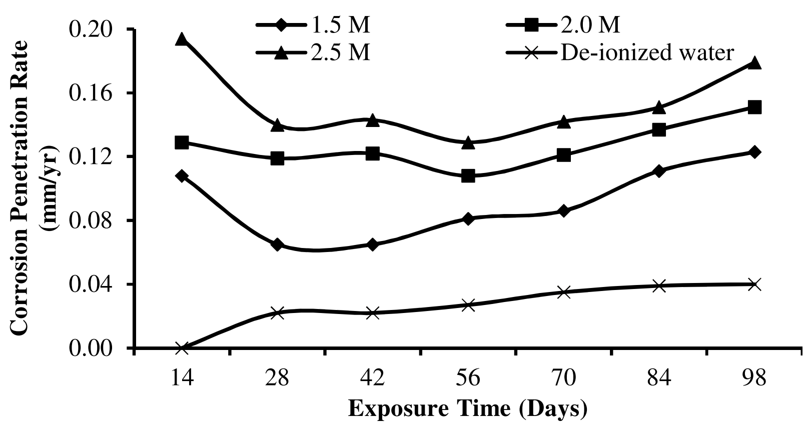


Figure 7: Corrosion penetration rate against exposure time of samples inhibited by Bitter-leaf extracts

This behavior is explained as originating from the passive modification by the inhibitor at the interface between the metal substrate and the acidic environment. It breaks down at a certain potential but restored by the increment in the inhibitor concentration (Onyeka and Nwambekwe, 2007). The 2.5 M hydrochloric acid medium bearing coupons inhibited with bitter leaf extract yielded 0.194 mm/yr for the first 14 days and 0.179 mm/yr on the 98$^{th}$ day. Correspondingly, the acid solution setup without inhibitor at 14 days and 98 days respectively showed rate of penetration as 0.203 mm/yr and 0.223 mm/yr. Continuous comparison with the samples immersed in hydrochloric acid shows that bitter leaf brought reduction to the rate of penetration of hydrogen and dissolved oxygen (Onyeka and Nwambekwe, 2007).

### 3.2.4. Samples immersed in hydrochloric acid inhibited with synergized extract

Figure 8 show that the Synergized inhibitor significantly mitigated corrosion rate at the studied concentration. The inhibitor film has been proven by study to facilitate rust inhibition by modifying the environment's corrosivity at the metal surface (Faska et. al., 2007). For 2.5 M concentration, there was a drop to 0.086 mm/yr and 0.080 mm/yr respectively for the first 14 days of immersion as well as the 98th day.

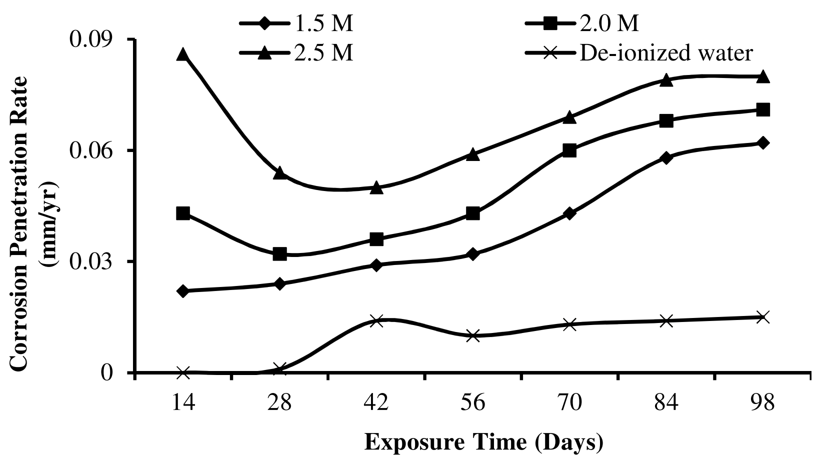


Figure 8: Corrosion penetration rate against exposure time for samples inhibited by synergized extract

## 3.3. Percentage Inhibition Efficiency Analysis

Corrosion inhibition efficiency is the measure of competence of an inhibitor to mitigate corrosion in a corrosive environment (Umoren et al., 2008). From Figure 9, the Neem inhibitor could individually produce the highest Percentage inhibition efficiency of 44.44% at the given temperature and inhibitor concentration on the 28$^{th}$ day of immersion. This is for sample soaked inside 1.5 M hydrochloric acid, the Neem from these results is averagely efficient in protecting the samples in hydrochloric acid. The Bitter leaf extract inside the media of hydrochloric showed low percentage inhibition efficiency of 30.76% on the immersed steel samples. The best inhibition efficiency for the samples was at 1.5 M hydrochloric acid on the 28$^{th}$ day of immersion. The corrosion data based on the calculated inhibition efficiency for bitter leaf extract inhibitor produced low inhibition efficiency on the average while the Synergized extract produced the highest percentage inhibition efficiency of 90%. Comparing the inhibition efficiencies of the three extracts the synergized formulation was the best because it produced the highest efficiency and gave a better protection to the samples in the acidic media.

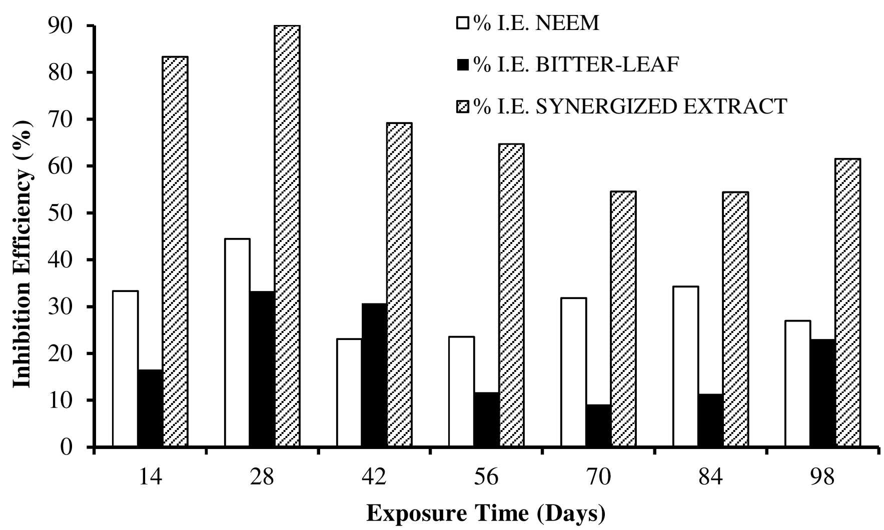


Figure 9: Corrosion inhibition efficiency against exposure time of neem, bitter leaf and the synergized extract in 1.5 M hydrochloric acid

## 4. CONCLUSION

From the results above, it can be concluded that the synergistic effect of Neem-bitter leaf formulation used as inhibitor produced a better result in reducing corrosion of hydrochloric acid when compared with the individual extracts of Neem and Bitter-leaf. This was evident from the reduced corrosion penetration rate and the low weight loss values. Also, at 1.5 M hydrochloric acid, the percentage inhibition efficiency was about higher and more efficient than the individual extracts of neem and bitter leaf. It can therefore be said that using synergized extract of Neem and Bitter-leaf clampdown corrosion more than Neem or Bitter-leaf could individually.

## 5. ACKNOWLEDGMENT

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## 6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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