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Comparison of the Inhibitive Effects of Neem (*Azadirachta indica*) Leaves Extract and Benzamide in Tetraoxosulphate (VI) Acid Solution

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ABSTRACT
A comparison of the inhibitive effects of Neem (Azadirachta indica) leaves extract and Benzamide in 0.5 M tetraoxosulphate (VI) acid was undertaken using the weight loss method of corrosion measurement. This was aimed at determining which of the inhibitors will function optimally in 0.5 M H_2SO_4 acid solution. Neem leaves extract offered better corrosion inhibition performance in the acid medium compared to Benzamide. The corrosion rate of mild steel in the acidic environment with Neem leaves extract as an inhibitor was lower compared to the corrosion rate of mild steel in the acidic medium with Benzamide as an inhibitor.The inhibition efficiencyof Neem leaves extract was higher compared to Benzamide. The mean corrosion rate of mild steel in the acidic environment with Benzamide and Neem leaves extract as inhibitors were 74.06 mm/day and 66.29 mm/day respectively. The mean inhibition efficiency of Neem leaves extract and Benzamide were 21.37 % *and 10.21* % *respectively in 0.5 M H_2SO_4 acid solution from the results of weight loss measurement. For optimum corrosion inhibition performance, Neem leaves extract is recommended.*

KEYWORDS: Benzamide, Comparison, Efficiency, Inhibition, Neem leaves.

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1.0 INTRODUCTION
The most effective way of mitigating the corrosion of metals in acidic media is by the use of synthetic corrosion inhibitors. Several synthetic corrosion inhibitors have been used to mitigate the corrosion of metals in various media. Benzamide acted as a mixed inhibitor for mild steel in tetraoxosulphate (VI) acid (Loto *et al.*, 2017). An inhibition efficiency of up to 70% at concentration 4 g 200 mL^{-1} H_2SO_4 over forty-eight (48) hours of exposure time was achieved using the inhibitor. N-(phenylcarbamothioyl) Benzamide affected both anodic and cathodic processes and their efficiency increased with concentration, with maximum efficiency of 95.22 % at 100 ppm (Gopiraman *et al.*, 2012). According to Lgaz *et al*. (2020), 2-mercaptobenzimidazole derivative proved effective in mitigating the corrosion of mild steel in hydrochloric acid solution at different temperatures. Benzotriazole showed good performance as a corrosion inhibitor in citric acid solution due to the presence of heteroatom and unsaturated bond that cause effective adsorption process leading to the formation of an insoluble protective surface film which suppresses the metal dissolution reaction (Matheswaran & Ramasamy, 2010). The use of synthetic corrosion inhibitors is being restricted by governments all over the world because of its adverse effect on man and the environment (Ezemonye *et al*., 2010). Attention is now shifting towards plant materials that are cheap, readily available and environmentally friendly (Okafor *et al*., 2010; Hassan *et al*., 2016). Several investigations have been conducted using plant extracts as corrosion inhibitors for metals in different media.

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Citrus aurantium leaves extract acted as an inhibitor for mild steel in sulphuric acid medium (Hassan *et al*., 2016). Maximum inhibition efficiency of the extract was found to be 89% at optimum concentration of 10 m/L at 40 ^oC. According to Ghaidani *et al.* (2020), Hibiscus leaves extract is a potential green corrosion inhibitor for mild steel in 0.5 M tetraoxosulphate (VI) acid medium. *Polyalthia longifolia* inhibited the corrosion of mild steel in 1 M HCl and an inhibition efficiency of about 70 % was achieved with 1.0 mL of the extract (Zubairu *et al*., 2021). Valek and Martines (2007) reported that Neem leaves extract inhibited copper corrosion in 0.5 M sulphuric acid better than already proven good inhibitors (2-acetamino-5-mercapto-1, 3, 4-thiadiazole and 1, 2, 3-benzotriazole).

From the above discussions, it is evident that little has been done to compare the corrosion inhibition performance of plant materials and synthetic corrosion inhibitors. Therefore, this work is aimed at comparing the corrosion inhibition performance of extract from Neem leaves and Benzamide in an acidic medium.

2.0 MATERIALS AND METHODS

2.1 Materials & Equipment

The materials and equipment used for the study were: mild steel sheet, plastic containers (one-liter size), strings, cotton wool, emery cloth (P60 and P220), distilled water, latex gloves, fresh Neem *(Azadirachta indica*) leaves, bowls, filter papers, Benzamide, tetraoxosulphate (VI) acid (H_2SO_4), ethanol, electronic balance and sieve.

2.2 Extraction of Active Ingredients in Neem Leaves Powder

The leaves were shade dried for fourteen (14) days, to enrich the active principles in them by reducing their water content (Saratha *et al*., 2009). The dried leaves were converted to powder by pounding using a mortar and a pestle.
Extraction of the active ingredients in the powdered Neem leaves was done using the reflux method (Mejeha *et al*., 2010; Oguzie *et al*., 2004). Sixty (60) grammes of powdered Neem leaves were refluxed in 1000 mL of 0.5 M tetraoxosulphate (VI) acid solution for three hours (Mejeha *et al*., 2010). The resulting mixture in the flask was filtered using a filter paper. The filtrate was taken as the stock solution.

2.3 Preparation of Corrosion Test Specimens

Forty-four rectangular test specimens each of dimensions (40mm x 30mm x 3mm) were prepared from a mild steel sheet. The percentage chemical composition of the mild steel sheet used for the preparation of the corrosion test specimens are: 98.82 wt% Fe, 0.143 wt% C, 0.124 wt% Si, 0.541 wt% Mn, 0.0204 wt% P, 0.307 wt% Cr, 0.00097 wt% Mo, 0.00788 wt% Ni, 0.0158 wt% Cu, 0.0224 wt% Al, 0.00027 wt% Nb, 0.00339 wt% Ti and 0.00134 wt% V. A 3mm diameter hole was drilled along the longitudinal end for suspension in the corrodent as shown in Figure 1. The sharp edge formed during the cutting and drilling operations were smoothened using a hand file. The surfaces of the test specimens were further polished with abrasive paper using water as a lubricant, to produce a smooth surface and to remove any trace of contaminants. The test specimens were degreased in ethanol, washed thoroughly in deionized water and air dried (Ehujuo *et al*., 2014; Eddy *et al*., 2011). After weighing, the test specimens were kept in a desiccator until needed for corrosion studies.

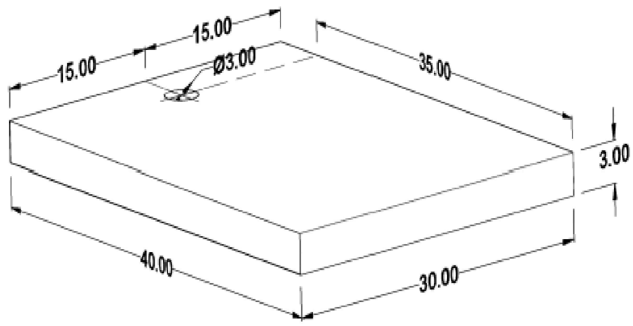

Fig 1 Corrosion test specimen (Coupon)

Journal of Newviews in Engineering and Technology (JNET)
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2.4 Preparation of Corrosion Medium

The corrosion environment used for this work was 0.5 M tetraoxosulphate (VI) acid solution. 0.5 M tetraoxosulphate (VI) acid solution was prepared by diluting 22.6 mL of concentrated tetraoxosulphate (VI) acid solution in 1000 mL of distilled water.

2.5 Corrosion Measurement

2.5.1 Weight Loss Measurement

The weight loss method used by Elachi *et al.* (2016) was adopted for determining the weight loss of mild steel specimens in the acidic medium. The required concentrations of the inhibitors (2 g/L, 4 g/L, 6 g/L, 8 g/L and 10 g/L) were respectively measured from the stock solution of Neem leaves extract and Benzamide and were respectively added to each of the containers containing one litre the corrodent in each set. No inhibitor was added to the eleventh container in each set. The pre-weighed test specimens were totally immersed in each of the plastic containers containing both the corrodent and the extracts. After every two days, samples were retrieved from the corrodent(s), observed, and washed to remove corrosion products. Samples were again subjected to absolute washing in absolute ethanol with a cotton wool, air dried and weighed to determine the final weight. The difference in weight of the coupon was taken as the weight loss. The average weight loss value was determined by adding the weight loss value obtained from the first set of experiment to the corresponding weight loss value obtained from each coupon in the second set of experiment and the result divided by two (2). The average weight loss value was recorded as the weight loss. The corrosion rate was computed using Equation 1 (Fontana, 1987).

$$\text{CR} = \frac{534W}{\text{DAt}} \qquad (1)$$

where CR is the corrosion rate in mm/day, W is the weight loss in grammes, A is the sectional area in mm^2, D is the density of the metal sample in g/mm^3 and t is the exposure time of the mild steel coupons in days.

The inhibitor efficiency (η %) of the extract was computed using Equation 2 (Oruene *et al*., 2021).

$$\eta\ (\%) = \left(\frac{W_o - W_i}{W_o}\right) \times 100 \qquad (2)$$

where W_i is the weight loss in grammes in the presence of inhibitor and W_o is the weight loss in grammes in the absence of the inhibitor.

3.0 RESULTS AND DISCUSSION

3.1 Effect of Exposure Time on Corrosion Rate

Figure 2 is the result of the effect of exposure time on corrosion rate in 0.5 M tetraoxosulphate (VI) acid solution containing Benzamide as an inhibitor. Corrosion rates were observed to decline as exposure time increased from day two (2) to eight (8) and subsequently corrosion rates increased with increase in exposure time at concentrations of 0 g/L, 2 g/L and 4 g/L. The decrease in corrosion rates after the second day could be due to the formation of passivating corrosion complexes that created a barrier between the metal and the acidic environment (Osarolube *et al.*, 2008). It could also be as a result of the decline in the strength of the acid due to corrosion complexes formed in the acidic solution (Idenyi et al., 2004 as cited in Osarolube *et al.*, 2008; Loto *et al*., 2017). The increase in corrosion rates observed on day eight (8) to ten (10) could be due to rise in temperature of the environment due to the reactions between the metal and the acid. Increase in temperatures increase the rate of corrosion reaction by increased atomic vibrations and diffusional movements (Loto *et al*., 2011; Godwin-Nwakwasi *et al.*, 2017) in acid medium. At concentrations of 6 g/L and 10 g/L of Benzamide, corrosion rates were observed to decrease as exposure time increased from day two (2) to day six (6) and subsequently, corrosion rates increased with increased exposure time. Corrosion rate decreased as exposure time increased from day two (2) to day four (4) after

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e- ISSN: 2795-2215

which an exposure, increase in corrosion rate was observed on the sixth day of exposure at 8 g/L of

Benzamide. In addition, a decrease in corrosion rate was observed as exposure time increased from day six (6) to day eight (8) and subsequently, corrosion rate increased with time at the same concentration of Benzamide. Figure 3 is the result of the effect of exposure time on the corrosion rate of mild steel in 0.5 M tetraoxosulphate (VI) acid solution containing Neem leaves extract as an inhibitor. Corrosion rate was observed to decrease as exposure time increased from day two (2) to day eight (8) and subsequently it began to increase with increased exposure time at concentration of 0 g/L. Corrosion rates were observed to decrease as the exposure time increased from day two (2) to six (6) and subsequently it began to rise with increased exposure time at 2 g/L and 10 g/L of Neem leaves extract. At extract concentrations of 4 g/L, 6 g/L and 8 g/L, corrosion rates were observed to decline as the exposure time is increased from day two to day four, after which it began to increase as exposure time increased from day four to eight and subsequently corrosion rates began to decline with exposure time. The reasons stated for the trends observed in the acidic medium with Benzamide as an inhibitor also applies here.
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Figure 4 compares the corrosion rates obtained at different concentrations of the inhibitors on different days. It was observed from the Figure that on day two (2), corrosion rate at 2 g/L of Neem leaves extract is higher compared to the corrosion rate at the same concentration of Benzamide. This could likely be due to the low concentration of Neem leaves extract used. The corrosion rates at 4 g/L, 6 g/L, 8 g/L and 10 g/L of Neem leaves extract were lower compared to the corrosion rates at the same concentrations of Benzamide. On day four (4), six (6) and ten (10), corrosion rates at 2 g/L, 4 g/L 6 g/L, 8 g/L and 10 g/L of Neem leaves extract were lower than the corrosion rates obtained at the same concentrations of Benzamide. On day eight (8) of exposure, corrosion rates at 2 g/L, 4 g/L and 6 g/L of Neem leaves extract were higher compared to the corrosion rates obtained at the same concentrations of Benzamide. On the same day, corrosion rate at 8 g/L and 10 g/L of Neem leaves extract were lower than the corrosion rates at the same concentrations of Benzamide. Low corrosion rate is an indication of good corrosion inhibition performance.

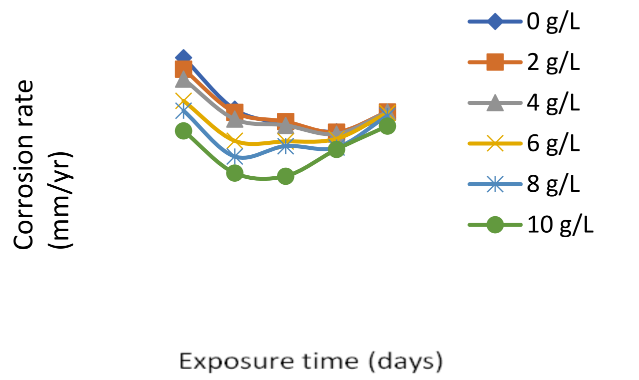

Fig 2 Effect of exposure time on the corrosion rate of mild steel in 0.5 M H_2SO_4 acid solution with Benzamide as an inhibitor.

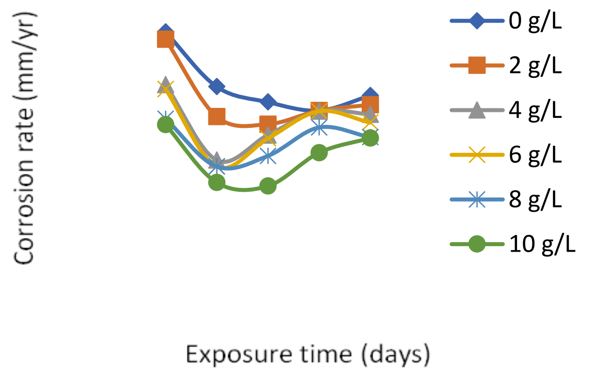

Fig 3 Effect of exposure time on the corrosion rate of mild steel in 0.5 M H_2SO_4 acid solution with Neem leaves extract as an inhibitor.

Journal of Newviews in Engineering and Technology (JNET)
Vol 3, Issue 3, September 2021
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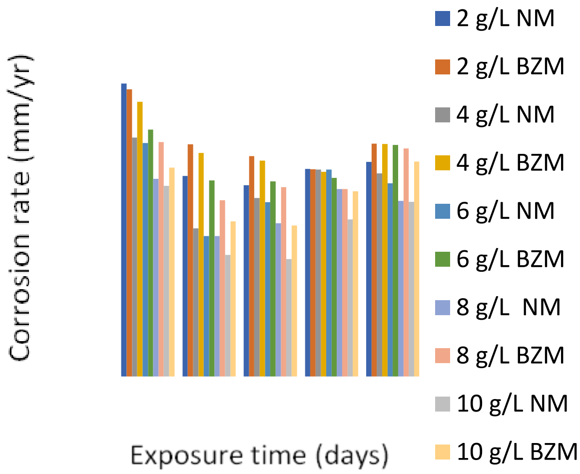

Fig 4 Comparison of the corrosion rates of mild steel in 0.5 M H_2SO_4 solution with different concentrations of Neem leaves extract and Benzamide on different days.

3.2 Effect of Concentration on Corrosion Rate

Presented in Figure 5 is the effect of concentration of Benzamide on the corrosion rate of mild steel in 0.5 M tetraoxosulphate (VI) acid solution. The Figure revealed that the corrosion rates decreased as the concentration of Benzamide increased from 0.0 g/L to 10 g/L except on the sixth day. On day six (6), corrosion rate increased as concentration increased from 0.0 g/L to 2.0 g/L, probably due to the low concentration of the inhibitor after which it began to decline with increased concentration. As the concentration of the inhibitor (Benzamide) is increased, there is a reduction in the surface area of the metal available for direct attack (Ugi, 2014) by the acid. Consequently, corrosion reaction is greatly reduced. The structure of Benzamide revealed the presence of a heteroatom (O) and aromatic ring system. The free electron pairs on the heteroatom form bonds with electrons on the mild steel surface (Rani & Basu, 2011). Therefore, a barrier is created between the metal and the corrodent which led to low corrosion rates as inhibitor concentration is increased from 0.0 g/L to 10 g/L. In addition, the π electrons present in the aromatic ring system present in the structure of Benzamide increases adsorption and consequently improve the inhibition efficiency of the inhibitors (Ali *et al*., 2003). It was also observed from Figure 6 that the corrosion rates decreased with increase in the concentration of the Neem leaves extract. The decrease in corrosion with increased concentration of Neem leaves extract could also be due to the adsorption of phytochemical compounds present in the Neem (*Azadirachta indica)* leaves on the mild steel surface (Madufor *et al., 2012*). The phytochemical analysis of Neem (*Azadirachta indica)* revealed the presence of tannins, flavonoids, saponins, anthraquinone, cardiotonics glycosides and alkaloids (Galeane *et al*., 2017). Sharma et al. (2015) reported that the main chemical compounds found in Neem are nimbidin, nimbin, meliantriol, azadirachtin and salannin. Elachi *et al*. (2016) reported the presence of cyanogenic glycosides, flavonoids, saponins, alkaloids phenols, tannins and oxalates in various concentrations in Neem leaves extract. These compounds have complicated molecular structures, large molecular weights, oxygen, sulphur and nitrogen atoms incorporated in their structures (Okafor *et al., 2010*) as shown in Figure 7. The adsorption of these compounds on the metal surface through the lone pairs of electrons present on their oxygen, sulphur and nitrogen atoms created a barrier (Okafor *et al.*, 2010) between the metal and the corrodent. Consequently, corrosion rate is reduced. However due to the complex chemical composition of *Azadirachta indica*, it is difficult to attribute the inhibitive effect to any particular compound. However, the combined effects of these compounds and other components present in the extracts might have played a part in the inhibition process (Madufor *et al.*, 2012).

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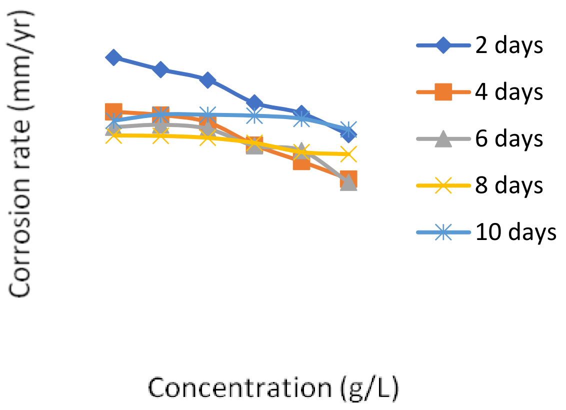

Fig 5 Effect of concentration on the corrosion rate of mild steel in 0.5 M H_2SO_4 acid solution with Benzamide as an inhibitor.

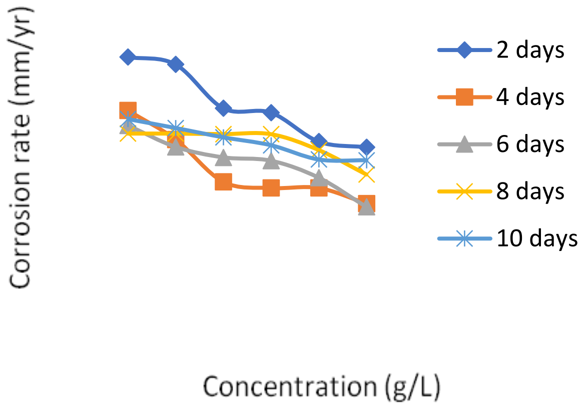

Fig 6 Effect of concentration on the corrosion rate of mild steel in 0.5 M H_2SO_4 acid solution with Neem leaves extract as an inhibitor.

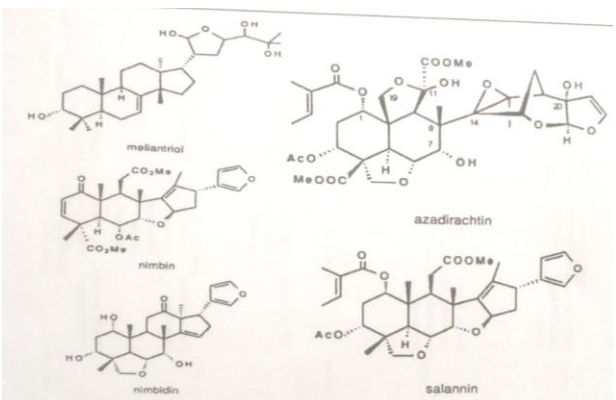

Fig 7 Chemical compounds in *Azadirachta indica* (Sharma *et al*., 2015)

The group statistics test was used to compare the corrosion rates of mild steel in the acidic medium with Neem leaves extract and Benzamide as inhibitors and the result is presented in Table 1.

The mean corrosion rate of mild steel in the acidic environment with Benzamide and Neem leaves extract as inhibitors were 74.06 mm/day and 66.29 mm/day respectively. Therefore, the corrosion rate of mild steel in the acidic environment with Neem leaves extract as an inhibitor is lower compared to the corrosion rate of mild steel in the acidic medium with Benzamide as an inhibitor. This implies that Neem leaves extract inhibited the corrosion of mild steel better than Benzamide in the acidic medium.

The analysis of variance test was further used to determine whether there is any significant difference between the mean corrosion rate of mild steel in 0.5 M tetraoxosulphate (VI) acid solution at p = 0.05 probability level. The calculated value of p is 0.025 (column 6). This value is greater than 0.05, therefore there is a significant difference between the mean corrosion rates of mild steel in the acidic medium with Benzamide and Neem leaves extract as inhibitors at p = 0.05 probability level. This is to further confirm that Neem leaves extract mitigated the corrosion of mild steel better than Benzamide in the acidic medium.

Table 1: Comparison of corrosion rates in 0.5 M H_2SO_4 acid solution with Benzamide and Neem leaves extract as inhibitors using group statistics test

Inhibitor	N	Mean	Std. Deviation	Std. error Mean
Benzamde	30	74.06	11.38	2.08
Neem	36	66.29	14.56	2.68

Journal of Newviews in Engineering and Technology (JNET)
Vol 3, Issue 3, September 2021
Available online at http://www.rsujnet.org/index.php/publications/2021-edition
e- ISSN: 2795-2215

Table 2: Comparison of corrosion rates of mild steel in 0.5 M H_2SO_4 acid solution with Benzamide and Neem leaves extract as inhibitors using analysis of variance test

	Sum of Squares	df	Mean Square	F	Sig
Between groups	905.102	1	905.102	5.3	0.025
Within groups	9899.630	58	170.683		
Total	10804.73	59			

Note: Sig = 0.025, Significant at probability level, p = 0.05

3.3. Effect of Concentration on Inhibition Efficiency

Presented in Figures 8 and 9 are the results of the effect of concentration on inhibition efficiencies of Neem leaves extract and Benzamide in 0.5 M tetraoxosulphate (VI) acid solution. Figure 8 revealed that the inhibition efficiency of Neem leaves extract increased with concentration on day two, six, eight and ten. This could be attributed to the reasons already stated in section 3.2. On day four, inhibition efficiency increased as concentration increased from 0.0 g/L to 6.0 g/L after which it declined slightly as concentration is further increased to 8.0 g/L and subsequently it increased with concentration. The maximum inhibition efficiency of Neem leaves extract was 48.56 % at 10.0 g/L.

Presented in Figure 9 is the effect of concentration on the inhibition efficiency of Benzamide in 0.5 M tetraoxosulphate (VI) acid solution. The Figure revealed that, the inhibition efficiency of Benzamide increased with concentration on day two, four, six and eight. On day ten, inhibition efficiency remained steady as concentration increased from 0.0 g/L to 8.0 g/L after which it began to increase with concentration. The maximum inhibition efficiency of Benzamide was 34.28 % at concentration of 10 g/L.

Figure 10 compares the inhibition efficiencies of the inhibitors at different concentrations on different days. The Figure revealed that on day two, at concentration of 2 g/L, the inhibition efficiency of Neem leaves extract was lower compared to Benzamide at the same concentration. Also, Neem leaves extract was more efficient as an inhibitor in the acidic medium compared to Benzamide at concentrations of 4 g/L, 6 g/L, 8 g/L and 10 g/L. It was also observed from the Figure that Neem leaves extract was more efficient as an inhibitor compared to Benzamide at all concentrations on day four (4), six (6) and ten (10). On day eight (8) of exposure, Benzamide offered superior inhibition efficiency compared to Neem leaves extract at concentrations of 2 g/L, 4 g/L, 6 g/L and 8 g/L. At concentration of 10 g/L, Neem leaves extract was more efficient as an inhibitor compared to Benzamide probably due to the high concentration used.

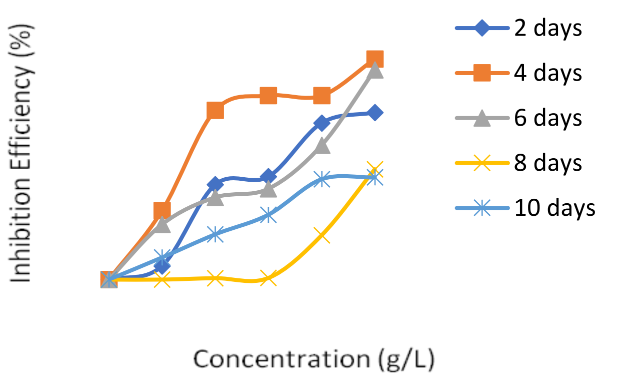

Fig 8 Effect of concentration on the inhibition efficiency of Neem leaves extract in 0.5 M H_2SO_4 acid solution.

Journal of Newviews in Engineering and Technology (JNET)
Vol 3, Issue 3, September 2021
Available online at http://www.rsujnet.org/index.php/publications/2021-edition
e- ISSN: 2795-2215

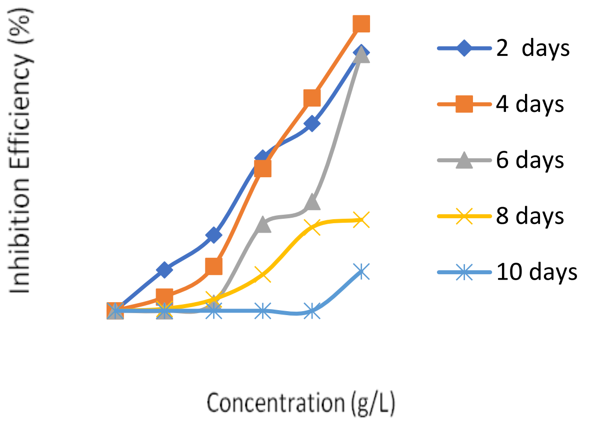

Fig 9 Effect of concentration on the inhibition efficiency of Benzamide in 0.5 M H_2SO_4 acid solution.

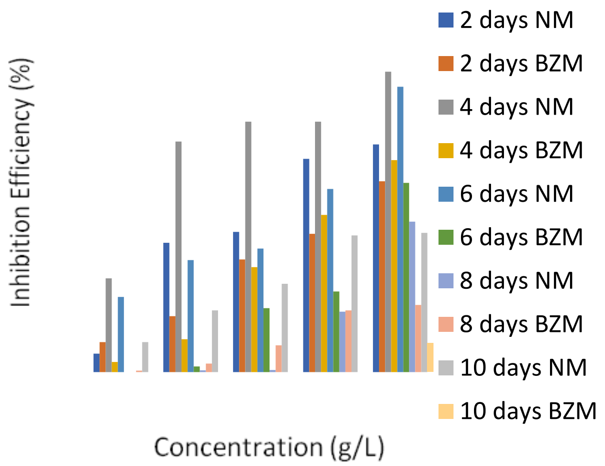

Fig 10 Comparison of the inhibition efficiencies of Neem leaves extract and Benzamide in 0.5 M H_2SO_4 solution.

The group statistics test was used to compare the inhibition efficiencies of the inhibitors in 0.5 M tetraoxosulphate (VI) acid medium and the result is presented in Table 3. The mean inhibition efficiencies of Neem leaves extract and Benzamide are 21.37 % and 10.21 % respectively in 0.5 M tetraoxosulphate (VI) acid. The inhibition efficiency of Neem leaves extract is higher compared to Benzamide in 0.5 M H_2SO_4 solution. Therefore, Neem leaves extract was more efficient as an inhibitor compared to Benzamide in the acid solution.

The analysis of variance was also used to determine whether there is any significant difference in the inhibition efficiencies of Neem leaves extract and Benzamide in 0.5 M H_2SO_4 solution at $p = 0.05$ probability level. The result is presented in Table 4. The calculated value of p is 0.004 (column 6). This value is less than 0.05, therefore there is a significant difference in the inhibition efficiencies of the inhibitors at $p = 0.05$ probability level. This further confirms the superior performance of Neem leaves extract.

Table 3: Comparison of the inhibition efficiencies of Neem leaves extract and Benzamide in 0.5 M H_2SO_4 acid solution using group statistics test.

Inhibitor	N	Mean	Std.deviation	Std. Error Mean
Neem	25	21.37	14.76	2.95
Benzamide	25	10.21	10.99	2.20

Table 4: Comparison of the inhibition efficiencies of Neem leaves extract and Benzamide in 0.5 M H_2SO_4 acid solution using one way analysis of variance test.

	Sum of Squares	Df	Mean Square	F	Sig
Between groups	1556.095	1	1556.10	9.19	0.004
Within groups	8126.858	48	169.310		
Total	9682.952	49			

Note: Sig = 0.004, Significant at probability level, p = 0.05

4.0 CONCLUSION

Neem leaves extract offered better corrosion inhibition performance in the acid medium compared to Benzamide. The corrosion rate of mild steel in the acidic environment with Neem leaves extract as an inhibitor is lower compared to the corrosion rate of mild steel in the acidic medium with Benzamide as an inhibitor. The mean corrosion rate of mid steel in the acidic environment with Benzamide and Neem leaves extract as inhibitors were 74.06 mm/day and 66.29 mm/day respectively. The inhibition efficiency of Neem leaves extract is higher compared to Benzamide in the acidic medium. The mean inhibition efficiency of Neem leaves extract and Benzamide were 21.37 % and 10.21 % respectively in 0.5 M H_2SO_4 acid solution.

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