

Corrosion Inhibition of Mild Steel in Simulated Seawater by *Nymphae Pubscens* Leaf Extracts (NLE)

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Abstract: The corrosion inhibitory behaviour of *Nymphae Pubescens* (water lily) leaf extract (NLE) was studied using weight loss technique. NLE concentrations of 1%, 2%, 3%, 4% and 5% v/v were used at temperatures 303, 313, 323 and 333K. Functional groups present in NLE, probably responsible for the inhibition were analyzed using FTIR. Inhibition efficiency was found to increase with increasing inhibitor concentration. Temperature affected the corrosion inhibition of mild steel by NLE as corrosion rate increased with increase in temperature and a subsequent decrease in the inhibition efficiency. The highest inhibition efficiency of 85.91 % was obtained at 303K with inhibitor concentration 5%v/v and the least inhibition efficiency of 24.51% at 333K with inhibitor concentration 1%v/v. Even at 313K the highest concentration of NLE (5% v/v) gave inhibition efficiency of 77.36%. The surface morphology from scan electron microscopy examination (SEM) confirms this trend. The adsorption of the inhibitor was more suited with the Temkin adsorption isotherm and thermodynamic data calculated are suggestive of physical adsorption of the inhibitor molecules on the mild steel surface.

Keywords: Sea water; Mild Steel; *Nymphae Pubescens*; Weight loss method; Corrosion rate, inhibition efficiency.

1. INTRODUCTION

Corrosion is the gradual destruction of materials by chemical reaction with their environment. In the most common use of the word, this means electrochemical oxidation of metal in reaction with an oxidant such as oxygen (Olawale, *et al.*, 2016). Corrosion is a major problem in desalination plants as well as other industrial applications. In parts of the world with short supply of fresh water, there is an increasing use of sea water for fresh water production (Bardal.E, 2004). Most metal structures used in sea water (ships, oil platforms, piers, pipelines, etc.) are traditionally made of mild low-carbon and low alloy steels as well as copper based alloys. The salt content in the water causes both corrosion and abrasion in piping, tanks and process equipment. Protecting these valuable assets from the effects of exposure to seawater is a critical challenge for corrosion engineers in these regions.

Over the years, there have been reports on the corrosion of mild steel and its alloys in sea water environments (Malik *et al.*, 1999, Melchers and Jeffery, 2005, Moller *et al.*, 2006 and Aramide, 2009). The use of inhibitors is one of the most practical methods for protecting metallic corrosion, especially in hostile environment like seawater (Bakirhan *et al.*, 2016). Plant extracts are a rich source of naturally synthesized chemical compounds, readily available low-cost and eco-friendly, and can be obtained through simple extraction processes with low cost as well as biodegradable (Eletre, 2007). Although many synthetic compounds show good corrosion inhibition ability, the search for more non toxic environmental friendly inhibitors are the focus now in metallic corrosion prevention. Literature records many plant extracts as effective corrosion inhibitors for iron or steel as well as copper and its alloys in aggressive corrosion media (Orubite and Oforka 2004, Abiola *et al.*, al 2007, Nnabuk 2009, Kalada and James 2011, Rajendran and Karthikeyan, 2012, Hamdy and El-Gendy, 2013, , Bammou *et al.*, .2014) . Some of these plants are common aquatic weeds that have found use as corrosion inhibitors. *Nymphae Pubscens* (water lily) is an aquatic weed that grows in fresh water environment of Bonny Island. The plant is a native to the temperate and tropical parts of the world. Most species of water lilies have rounded variously notched, waxy-coated leaves on long stalks that contain many air spaces and float in quiet freshwater habitats (Encyclopedia Britannica, 2017). From the

photochemical investigation of NLE it is worthy to note that the leaves of this aquatic weed contains hetro-atoms (N, S and O) and the availability of π electrons in the aromatic system which are inherent in its complex mixture of glycosides, saponins, alkaloids, terpenes, tannins, phenolic substances and flavonoids (Khaled, 2008) .Not much use of it is made by the locals both for domestic or other purposes.

Table1. *Phytochemical components of *Nymphae Pubescens* leaf extract*

| Substances | Presence |
|--------------------|----------|
| Saponins | ++ |
| Tannins | +++ |
| Flavonoid | - |
| Cardiac Glycosides | + |
| Alkaloids | +++ |
| Anthroquinones | + |
| Steroids | - |
| Triterpenoids | - |

+++ = highly present, ++ = moderately present, - = absent or presence in negligible quantity.

In this present work, corrosion inhibition of mild steel in seawater by *Nymphae Pubescens* (water lily) leaf extract was carried out using the weight loss method.

2. EXPERIMENTAL

2.1. Material Preparation

Mild steel sheets where obtained from the Science/Engineering Workshop, University of Port Harcourt. Each sheet was 1mm in thickness and was mechanically press-cut into rectangular coupon size of 4mm by 3mm.The coupons were polished with emery paper (600 grade), washed with deionized water, degreased with acetone, dried and weighed before experiments. The chemical composition of mild steel used is given below as follows:

Table2. *Shows percentage composition of mild steel used for this research*

| C | Si | Mn | P | S | Ni | Fe |
|------|------|------|-------|-------|-------|-----------|
| 0.28 | 0.22 | 0.73 | 0.015 | 0.006 | 0.007 | Remainder |

2.2. Preparation of Simulated Seawater

Simulated seawater was used for this research rather than natural seawater in order to minimize the biological effects as well as provide a reproducible solution of known composition. The formula of Axelrod Glen S. (2005) was employed in this preparation.

Table2. *Formula for 35.00% artificial seawater*

| SALT | Concentration (mg/l) |
|---|----------------------|
| Sodium chloride (NaCl) | 55 |
| Sodium sulfate (Na ₂ SO ₄) | 10 |
| Potassium chloride (KCl) | 4 |
| Sodium bicarbonate (NaHCO ₃) | 1 |
| Boric acid (H ₃ BO ₃) | 0.010 |
| Magnesium chloride (MgCl ₂ .6H ₂ O) | 32 |
| Calcium chloride (CaCl ₂ .2H ₂ O) | 4 |

Source: *Axelrod Glen S. (2005)*

2.3. Preparation of Plant Extracts

Nymphae Pubescens leaves were plucked from a waterlogged area in Bonny Local Government area of Rivers State and identified at the University of Port Harcourt, Herbarium. Leaves extract of *Nymphae Pubescens* was prepared as described by Olamide *et al.*, (2016).

2.4. Weight Loss Measurement

Mild steel coupons were immersed in five beakers containing various concentrations of the corrosion inhibitor and a sixth beaker without the inhibitor which was used as the control. For the experiment at

303K, the coupons were retrieved after 24 hours. They were thoroughly cleaned and washed with distilled water, degreased with acetone and weighed with an electronic balance. At elevated temperatures, the coupons were retrieved after six hours. The same process of weighing was applied. The difference between the present and previous weights were computed and recorded as the weight loss. The inhibition efficiency (I.E %) was calculated using the following equation :

$$IE\% = \frac{w - w_i}{w} \times 100$$

Where w and w_i are weight loss of steel coupons in the absence and presence of NLE respectively.

The corrosion rate (C.R) in millimeter per year (mmp y^{-1}) was computed using the relation:

$$C.R = \frac{KW}{DAT}$$

Where W is the weight loss resulting from the difference in initial and final specimen weights (mg), A is the coupon surface area (mm^2), D represents the materials density (mg/cm^3), K is the Rate Constant (87.6) and T is the time of exposure (hours).

2.5. Fourier Transform Infrared (FT-IR) Spectroscopy

The functional groups present in NLE and the film formed on the metal surface were determined using the Cary-630 Agilent Fourier transform infra-red spectrophotometer. The analysis was carried out by scanning the sample through a wave number range of 650 to 4000cm^{-1} .

2.6. Surface Morphology

A scanning electron microscope (SEM) model JSM-5600 LV, was used to analyze the morphology of mild steel surface without and with inhibitor added. One piece of mild steel coupon was immersed in seawater solution while the second coupon was immersed in a solution of seawater and 5% v/v of NLE for 24 hours. The coupons were rinsed dried, and subjected to SEM examination.

3. RESULT AND DISCUSSIONS

3.1. Weight Loss Measurement

Table 3. Calculated values of corrosion rate (mmp y^{-1}) and inhibition efficiency (I.E %) for Mild Steel corrosion in seawater in the absence and presence of various concentrations of NLE at different temperatures

| Inhibitor Concentration (v/v%) | Corrosion rate (mmp y^{-1}) $\times 10^3$ | | | | Inhibition Efficiency (I.E %) | | | |
|--------------------------------|--|------|------|------|-------------------------------|-------|-------|-------|
| | 303K | 313K | 323K | 333K | 303K | 313K | 323K | 333K |
| 0 (blank) | 0.34 | 0.37 | 0.41 | 0.45 | - | - | - | - |
| 1 | 0.31 | 0.34 | 0.38 | 0.41 | 30.71 | 28.09 | 24.8 | 24.51 |
| 2 | 0.24 | 0.28 | 0.32 | 0.37 | 33.64 | 30.13 | 29.56 | 27.04 |
| 3 | 0.19 | 0.24 | 0.28 | 0.32 | 54.3 | 51.7 | 50.56 | 48.74 |
| 4 | 0.13 | 0.18 | 0.23 | 0.28 | 69.2 | 66.43 | 66.06 | 61.68 |
| 5 | 0.07 | 0.12 | 0.18 | 0.24 | 85.91 | 84.05 | 79 | 77.36 |

3.2. The Effect of Addition of NLE on the Weight Loss of Mild Steel Coupons in Sea Water

Inhibition efficiency IE (%) and corrosion rate (CR) obtained from weight loss measurements at different concentrations of inhibitor at temperatures 303-333K are summarized in Table 4. It is evident that the extract inhibits the corrosion of mild steel, at all concentrations used (1%-5% v/v). Table 4 also reveals that mild steel coupons were protected to an extent in sea water. The values of corrosion rate obtained for the blank (no inhibitor) was more than when inhibitor was added to the corrosive media. Even at the lowest inhibitor concentration of 1% v/v corrosion rate (0.31 mmp y^{-1}) obtained was lower than the blank (0.34 mmp y^{-1}) at 303K.

This trend has been reported by several researchers confirming the effectiveness of plant extracts as corrosion inhibitors (James and Akaranta. 2009, Orubite and Oforka 2004).

3.3. Effect of Inhibition on Corrosion Rate

Fig.1 below shows the effect of inhibitor concentration on the corrosion rate. As inhibitor concentration increased a corresponding decrease in the corrosion rate occurred. The lowest corrosion

rate was at 0.07 mmp y^{-1} at its highest concentration and its highest corrosion rate of 0.31 mmp y^{-1} at its lowest concentration. The effect of concentration on chemical reaction is an established fact in chemistry. Increase in concentration means more reactant molecules available for a chemical reaction. From the photochemical investigation of NLE, it is worthy to note that the leaves of this aquatic weed contains hetero atoms (N and O) and availability of π electrons in the aromatic system which are inherent in its complex mixture of glycosides, saponins, alkaloids, terpenes, tannins, phenolic substances and flavonoids (Khaled,2008) . Therefore increase in concentration means more of these heteroatoms adsorbed on the surface of the metal and subsequent decrease of corrosion rate. This is in agreement with findings from other works of Subir and Ishita, 2016 and Al-Otaibi et-al, 2012.

3.4. Effect of Inhibitor Concentration and Adsorption Mechanism

Inhibition efficiency plots are to show the effectiveness of an inhibitor in metal protection. This was done for NLE and presented in Figure 2. Inhibition efficiency as observed from the plot, increased with increased inhibitor concentration. The observed inhibition action of the NLE could be attributed to the adsorption of its components on the mild steel surface. They form layer of adsorbed molecules which isolates the metal surface from the aggressive medium leading to a decrease in the corrosion rate and hence a corresponding increase in its inhibition efficiency (James et al., 2009). Plots of surface coverage (θ) expressed the extent of coverage of inhibitor molecules on metal surface. Table 5 below shows surface coverage data obtained for NLE molecules on mild steel coupons immersed in sea water. As inhibitor concentration increased more NLE molecules are available to cover mild steel's surface thus forming a barrier between sea water and the mild steel coupons and subsequent protection of the mild steel coupons. Studies by Hamdy.A and Nour Sh. El-Gendy, (2013) and Aisha Al-Moubaraki *et.al.*, 2015 are in agreement with these findings. It can also be deduced from table 5 that the activation energies (E_a) increase in the inhibited system as the concentration increases. The calculated values of E_a ranges from $0.27 - 1.32 \text{ kJmol}^{-1}$ is less than the threshold value (40 kJmol^{-1}). Such behavior coupled with the increased inhibition efficiency is evident of physical adsorption mechanism as seen in figure 4 below.

Table5. Calculated values of Average Surface Coverage (θ), Activation Energy and Heat of Adsorption for Mild Steel corrosion in seawater in the absence and presence of various concentrations of *Nymphae Pubescens* leaf extracts at different temperatures using weight loss method.

| Inhibitor Concentration (v/v%) | Average Surface Coverage (θ) | | | | Activation Energy (E_a) (kJmol^{-1}) | Heat of Adsorption (Q_{ads}) (kJmol^{-1}) |
|--------------------------------|---------------------------------------|------|------|------|---|--|
| | 303K | 313K | 323K | 333K | | |
| BLANK | - | - | - | - | 0.27 | - |
| 1 | 0.31 | 0.28 | 0.25 | 0.25 | 0.22 | -16.574 |
| 2 | 0.34 | 0.3 | 0.3 | 0.48 | -0.02 | -15.293 |
| 3 | 0.54 | 0.52 | 0.51 | 0.49 | 0.30 | -7.1104 |
| 4 | 0.69 | 0.66 | 0.66 | 0.62 | 0.09 | -1.9297 |
| 5 | 0.86 | 0.84 | 0.79 | 0.77 | 1.32 | 3.56975 |

Another thermodynamic parameter which further describes the adsorption mechanism is the heat of adsorption, Q_{ads} . It is connected to the surface coverage (θ) through the relation:

$$Q_{ads} = 2.303R [\{ \log \{ \theta_2/1 - \theta_2 \} - \log \{ \theta_1/1 - \theta_1 \}]$$

Where, θ_1 and θ_2 are values on the degree of surface coverage at temperatures T_1 and T_2 respectively. The calculated values of Q_{ads} in table 5 show trend of a progressive move from negativity to positivity as the concentration of the inhibitor increases as well as an increase in the inhibition efficiency with temperature.

3.5. Effect of Temperature on Corrosion Rate

Fig. 3 represents the effect of temperature on the corrosion rate. This is also in line with other works (Oloruntoba, 2013 and Adams et-al, 2015) where the corrosion rate of mild steel in sea water increased as temperature increased. The lowest corrosion rate was observed at 303K (0.07 mmp y^{-1}) and highest (0.41 mmp y^{-1}) at 333K. Many metals are used in environments where temperatures differ from room temperature. This is the basis for studying the effect of temperature on the inhibitor efficiency.

The effect of temperature on the corrosion rate of mild steel in seawater with and without the NLE corrosion inhibitor was tested by the weight loss method over a temperature range from 303 to 333 k.

The corresponding data are shown in table 5 above. Also from a close inspection of figure 3, it is clearly seen that the effect of increasing temperature leads to an increase in the corrosion rate with and without NLE albeit to a lesser rate with NLE.

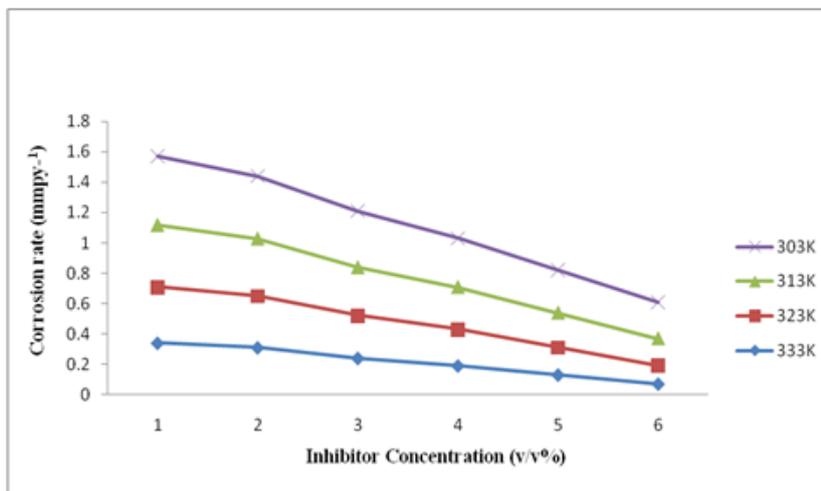


Fig1. Average Corrosion rate (mmp y-1) for Mild steel corrosion in seawatr in the absence and presenc of various concentrations of *Nymphae pubescens* leaf extracts at different temperatures

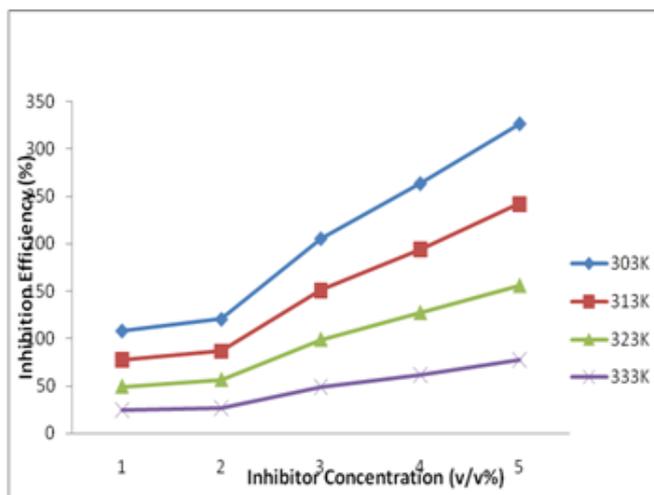


Fig2. Average inhibition efficiency (IE%) for mild steel in seawater in the presence of various concntrations of *Nymphae pubescens* extracts at different temperatures

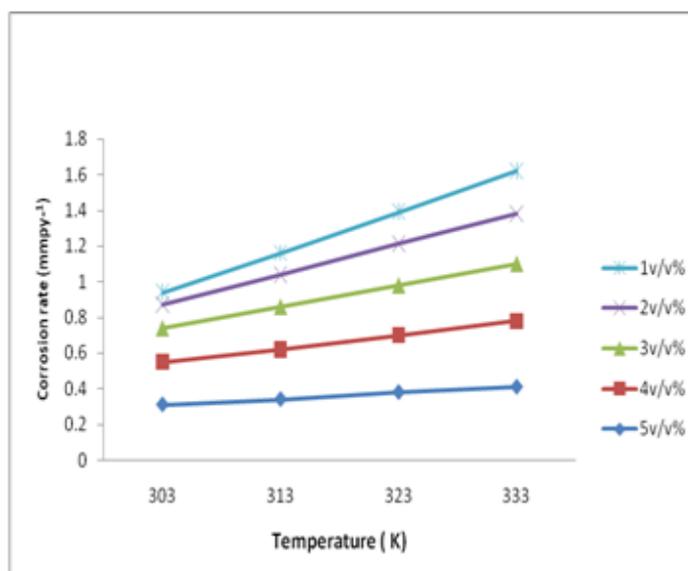


Fig3. Effect of temperature on the corrosion rate of Mild Steel in seawater using NLE

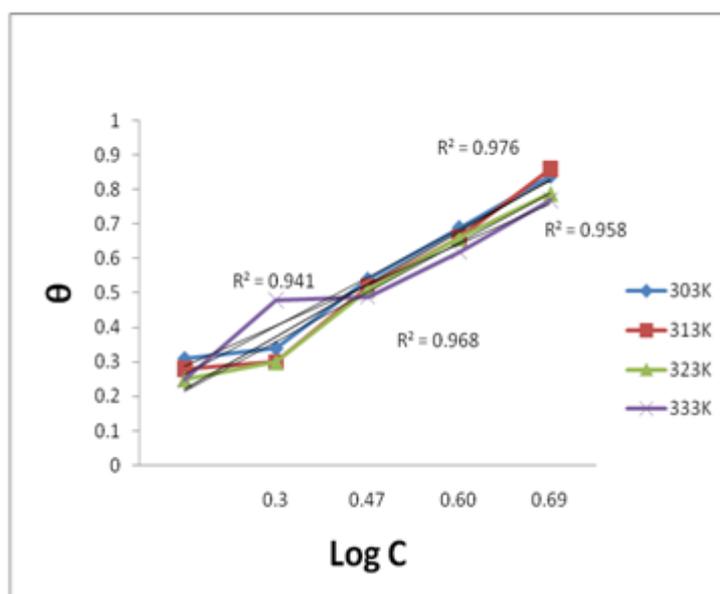


Fig4. Temkin Adsorption Isotherm for corrosion of mild steel in seawater

The effect of temperature on the inhibition efficiency is shown in Fig 2. The inhibition efficiency for the temperatures studied follows the trend $85.91 > 84.05 > 79 > 77.36$ % (303, 313, 323 and 333K) respectively for the inhibitor concentration of 5% v/v. It was generally observed that inhibition efficiency increase with increasing inhibitor concentration but decrease with increasing temperature, indicating an increased rate of dissolution of mild steel coupons in sea water and a partial desorption of the temporary protective film as temperature increases. This further suggests a physisorption mechanism of the inhibitor molecules on the mild steel surface, which is in line with the works of Kalada Hart and A.O. James (2014). The extract showed some reasonable efficiency even at higher temperature. At the highest temperature of 333K an inhibition efficiency of 77.36 % was recorded when 5% v/v extract concentration was used. Most inhibition efficiency record lower percentage at such high temperature (Nnanna *et-al.*, 2014).

3.6. Adsorption Isotherm

Basic information on the interaction between the inhibitors and the metal surface can be provided by the adsorption isotherm. Using the values of the surface coverage, different adsorption isotherms can be used to deal with experimental data. Of all the adsorption isotherms tested the Temkin adsorption gave the best interpretation of interaction between the inhibitors and the metal surface as can be seen in fig.4 above. It is worthy to note that R^2 values were close to unity at both high and low temperatures investigated.

3.7. Analysis of Protective Film

The adsorptive protective film was further confirmed by FT-IR spectra of plant extract and mild steel surface immersed in seawater. The main constituents of NLE are alkaloids, saponins and tannins. These major constituents (hetero atoms) are responsible for the formation of a blanket on the metallic surface. The FT-IR spectrum of the NLE extract is shown in the fig. 5 below.

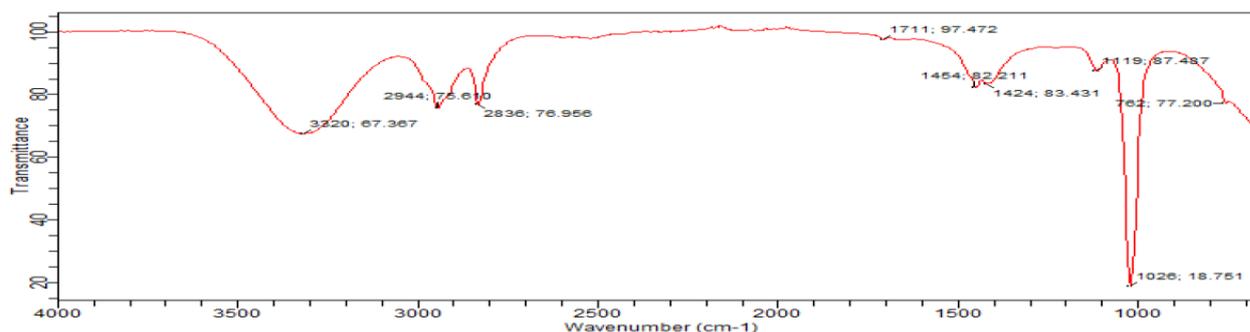


Fig5. FT-IR of *Nymphae Pubescens* leaf extract

The peaks above are in the range of 500-3500 cm^{-1} indicating the presence of similar functional groups which have peaks at 762, 1026, 1711 and 3320 (cm^{-1}).

These peaks show the presence of -C-O stretching, -C-O stretching, -C=O stretching and -OH respectively.

The FT-IR spectrum of NLE is similar to the FT-IR of NLE film on the mild steel surface indicating the presence of similar functional groups. A similar situation was reported by Ngobiri, *et.al.* 2016.

3.8. Surface Morphology Studies

Surface morphology of mild steel specimens in uninhibited and inhibited seawater solution was carried out by immersion in a test solution after 24 hours at 303K. Figures 5 (a) and (b) shows the SEM images of the mild steel in the absence and presence of the inhibitor. A severely corroded surface morphology was observed after the immersion in the uninhibited system due to the corrosive attack by seawater. Corrosion was relatively uniform with no trace of a local attack. With addition of NLE, the corrosion damage was visibly reduced.

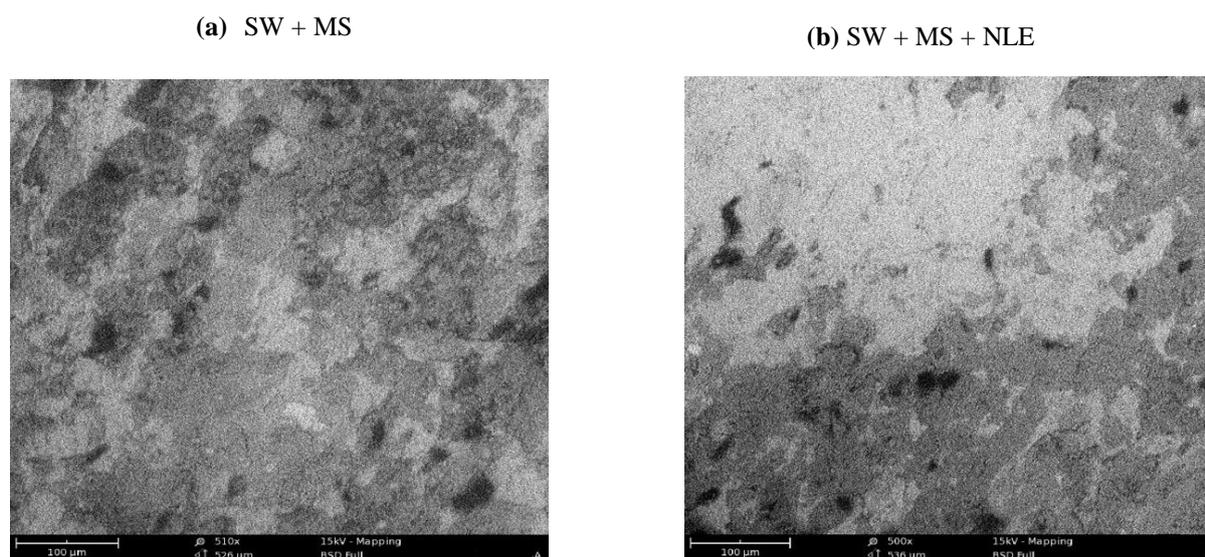


Fig6. Showing SEM micrographs of mild steel corrosion in uninhibited and inhibited seawater

4. CONCLUSION

NLE was found to inhibit corrosion of mild steel in seawater and the inhibition efficiency increased with increasing extract concentration. At the highest extract concentration of 5v/v%, the optimum inhibition efficiency of 85.91% was attained. Although corrosion rate increased as temperature increased with subsequent decrease in inhibition efficiency, NLE is found to have inhibition efficiency of 77.36% even at 333K, the highest temperature studied... The lowest and highest corrosion rate were 0.31 mmp y^{-1} (1v/v%) and 0.07 mmp y^{-1} (5v/v%) respectively at 303K and 0.41 mmp y^{-1} (1v/v%) and 0.24 mmp y^{-1} (5 v/v%) respectively at 333K. While the highest inhibition efficiency of 85.91% and the least inhibition efficiency of 24.51% was at 303K and 333K respectively when (1v/v %) NLE was used. The inhibitor is an adsorptive inhibitor and its adsorption obeys the Temkin's adsorption isotherm. Thermodynamic data calculated and SEM micrographs from uninhibited and inhibited system are also suggestive of adsorption of the inhibitor molecules on the mild steel surface.

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