

Corrosion Inhibition of Mild Steel C1026 Pipeline in Na₂SO₄ using Green Inhibitors

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ABSTRACT: Mild Steel C1026 is widely utilized as crude oil pipeline material. Interestingly, soil characteristic in Bayelsa State vary from one location to another. This research work is to determine Mild Steel C1026 behaviour in a predominately Sulphate soil environment. In order to determine the disproportionate corrosion behaviour of the Mild Steel (MS) in Bayelsa State, soil samples of three communities in three local government areas of the state were collected for the investigation. Green plant extracts were utilized as corrosion inhibitors in Sodium Sulphate, Na₂SO₄ environment using gravimetric method. The green plant extracts were Scent Leaf (SL) (*Ocimum gratissimum*), Cassava Leaf (CL) (*Manihot Esculenta*) and Neem Leaf (NL) (*Azadirachta Indica*). Parameters investigated were: Weight Loss (g), Corrosion Rate, CR (mpy), Inhibition Efficiency, IE (%), pH, Brinell Hardness Test and Surface Roughness. The plant extracts were observed to exhibit some degree of Inhibition Efficiency. Scent Leaf was 97.58%, Neem Leaf was 97.53%, while Cassava Leaf was 97.42%; Average Corrosion Rate, CR of Scent Leaf was 3.302mpy, Neem Leaf 2.999mpy and Cassava Leaf 2.999mpy; Average Weight Loss of Scent Leaf was 0.085g, Cassava Leaf 0.084g, and Neem Leaf was 0.049g; Brinell Hardness of Control Sample (Unpolished) was 129HK₁₀₀, Control Sample (Polished) was 131HK₁₀₀, MS in Sodium Sulphate was 154HK₁₀₀, Scent Leaf 147HK₁₀₀, Cassava Leaf 147HK₁₀₀, while Neem Leaf was 143HK₁₀₀. Surface Roughness: Sodium Sulphate was 8.377µm, Scent Leaf was 5.621µm, Cassava Leaf 5.779µm, while Neem Leaf was 4.658µm, etc.

KEY WORDS: Bayelsa State, Corrosion, Green inhibitors, Mild Steel, Pipeline.

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I. INTRODUCTION

Bayelsa State is one of the major producers of oil and gas in the Niger Delta region of Nigeria. In 1956, Shell British Petroleum (now Dutch Shell) discovered crude oil in Nigeria at Oloibiri community in Ogbia Local Government Area in Bayelsa State, while in 1958, commercial quantity was produced [1,2]. Pipelines are often used to transport oil and gas underground from the production site to the central processing base. Pipeline being made of mild steel is usually susceptible to corrosion. Corrosion is the steady destruction of a metal by a variety of slow chemical or electrochemical reaction between the metal and its environments. A major cause of pipeline corrosion underground is the nature of the soil [3,4,5]. Thangavelu et al., 2011 [6] stated that corrosion is a tumor on metal due to thermodynamic instability [7]. A viable method of corrosion control is the use of inhibitors. Inhibitor is a substance when added in small concentration decreases the effective corrosion rate. Most common inhibitors used in industry are inorganic compounds predominantly composed of nitrogen, oxygen and sulphur atoms. Inhibitors that contain double or triple bonds play an important role in facilitating the adsorption of compounds onto the metal surface [8,9]. In this investigation, natural green plant extracts like *Ocimum gratissimum*, *Manihot Esculenta* and *Azadirachta Indica* leaves were used as the corrosion inhibitors to investigate the behavioural characteristics of MS C1026 in a predominately sulphate environment. Unlike the inorganic compounds, they are environmentally benign, less costly, readily available and ecologically acceptable [10,11].

II. MATERIALS AND METHODS

The investigation was conducted in accordance with ASTM G13 which is a recommended standard practice for laboratory immersion corrosion testing of materials [11].

Mild Steel grades: C1026 coupons of oil and gas pipeline were used for the investigation throughout as control and inhibited samples. The corrosive medium utilized was Sodium Sulphate, while the corrosion inhibitors were Scent Leaf (*Ocimum gratissimum*), Cassava Leaf (*Manihot Esculenta*) and Neem Leaf (*Azadirachta Indica*). The laboratory investigations were performed in plastic containers and ropes were used to suspend the coupons in the baths. The coupons were weighed with Tare electronic weighing balance before and after immersion in each experiment. The Mild Steel C1026 elemental composition utilized for the work is shown in Table 1. The grade of MS and its elemental composition were determined using OXFORD INSTRUMENTS.

Table 1: Elemental composition of the Mild Steel.

Element	Mn	Ti	As	Cu	Fe
Wt %	0.08	0.17	0.07	0.07	98.88

2.1 Sample Surface Preparation

Polishing of the coupons surfaces were with rough and smooth grit emery papers. They were rinsed in water, degreased in acetone, dried in air and weighed with Tare weighing balance followed by immersing in the test medium for a period of 100 days (2400 Hours).

2.2 Preparation of the Inhibitor Extract

Fresh Leaves of Scent Leaf (*Ocimum gratissimum*), Cassava Leaf (*Manihot Esculenta*) and Neem Leaf (*Azadirachta Indica*) were obtained from Akenfa market and Agudama forest in Bayelsa State. A weight of 31g of each inhibitor leaves were measured and thoroughly pound with mortar and pestle and filtered after adding distilled water with filtration cloth. Thereafter, 150ml each of the leaves extracts were added to 250ml of distilled water with 35g of Sodium Sulphate.

2.3 Preparation of Mild Steel Coupons

Mild Steel samples were cut into 4 x 2cm from a corroded cylindrical pipeline. They were abraded with emery papers, washed with acetone and subjected to the various bath concentrations for a period of 100 days (2400 hours).

2.4 Preparation of Corrosive Media

About 250ml of distilled water was measured with a cylindrical flask and poured into a plastic container containing 35g of Sodium Sulphate.

2.5 Weight - Loss Analysis

The Mild Steel coupons were first abraded with coarse and fine emery papers and degreased with acetone. The initial weights of the coupons were obtained by using Tare weighing balance before they were immersed in the corrosive medium and specific concentrations of inhibitor extracts were added. A control bath was also prepared and coupons were tested for purpose of comparison.

The following formulae were used to compute the rate at which the MS coupons corroded and the inhibitors efficiency of the Leaf-extracts of Scent Leaf, Cassava Leaf and Neem Leaf extracts.

Corrosion Rate, CR = $K \times \text{Loss in Weight (g)/Surface Area} \times \text{Period of immersion (Hours) of the specimen} \times \text{Density of the MS}$ (1)

Where, K is Constant, 87.6 and Density of Mild Steel is 7.9 g/cm³, W₁ is Initial Weight and W₂ is Final Weight of coupons [9,11,12,13].

Corrosion Inhibition Efficiency,

IE (%) = $100 [1 - W_2/W_1]$ % (2)

Where, W₁ is Weight Loss in the Absence of inhibitor and W₂ is Weight Loss in the Presence of inhibitor [6].

2.6 Brinell Hardness Analysis

The Mild Steel coupons were analysed with Mi-Tech MH180 instrument. The tip of the instrument was placed and stroke at three different positions to take the readings and the average readings were taken.

2.7 Surface Roughness Analysis

SRT 6100 Surface Roughness Tester instrument was used to analyse the MS coupons. Three readings were taken for each coupon. The notches of the instrument were placed on the surface of each of the coupons. The reading continued until it clicked to stop. The readings displayed on the glass screen of the instrument were recorded as the surface roughness. The analysis was conducted at Turret Engineering, Port Harcourt Rivers State. Fig. 5 shows the trend of surface roughness of the coupons in the absence and presence of corrosion inhibitors.

2.8 Soil Sample Analysis

Soil samples were collected from three difference locations to characterize the elemental compositions using X-SUPREME OXFORD INSTRUMENT and the pH values using EQUIPMENT MODEL NO: HACH SENSION

1. The locations were: Ayamasa in Ekeremor Local Government Area, Angiama in Sagbama Local Government Area and Yenagoa in Yenagoa Local Government Area all in Bayelsa State. Weight loss and corrosion rate methods were used to examine the soil samples. The coupons were buried in the various soils in plastic containers and were retrieved, washed and weighed at interval of 240 hours (10 days). These were carried out over a period of 2400 hours (100 days). Table 7 shows the elemental composition of soil at the different locations. Fig. 1 show the coupons buried in the various soils.



Figure 1: The coupons buried in the various soils of Ayamasa, Angiama and Yenagoa.

2.9 Scanning Electron Microscope (SEM) Analysis

Scanning Electron Microscopy, SEM was used to determine the surface morphology of the various coupons. It was specifically utilized to establish the disparity in surface morphology and microstructure of the inhibited and uninhibited coupons.

III. RESULTS AND DISCUSSION

3.1 Effect of immersion time on weight loss of the coupons for the different inhibitors

Fig. 2 shows the effect of exposure time on weight loss of both inhibited and uninhibited coupons.

It shows that coupons in sodium sulphate without inhibitor had the highest average weight loss of 0.196g, Scent Leaf coupon was 0.085g, Cassava Leaf coupon was 0.084g, while Neem Leaf coupon was 0.049g. The results revealed that the coupon with SL exhibited the highest weight loss compared to the others, while those with NL extract exhibited the least weight loss. The difference in weight loss for each of the different inhibitors may have been due to disparity on adsorption into the MS substrate. As evident from Table 2, each of the inhibitors contain different amounts of the active ingredients. Such differences may have been responsible for different degrees of adsorption and surface coverage leading to differences of corrosion and weight losses exhibited by the coupons.

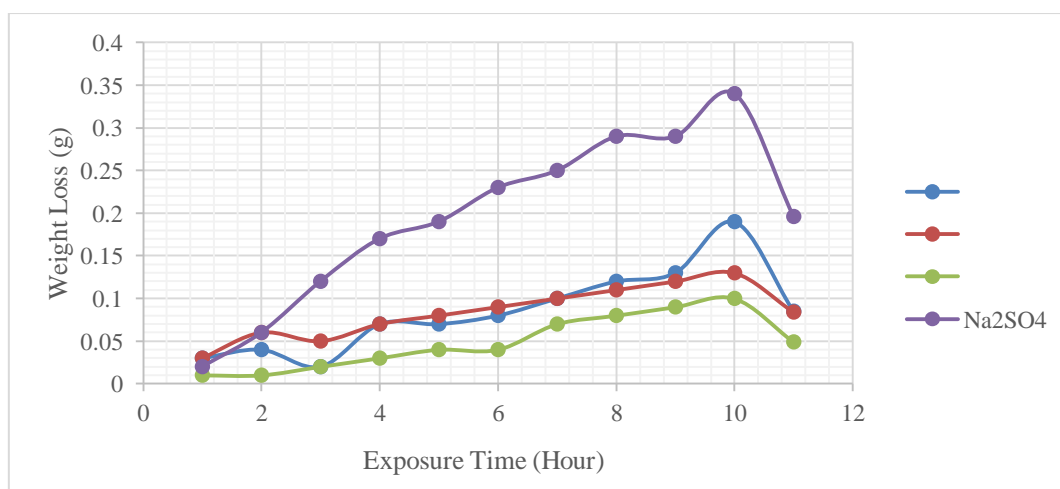


Figure 2: Effect of immersion time on weight loss of the coupons for the different inhibitors in Sodium Sulphate.

3.2 Effect of immersion time on corrosion rate (CR) of the coupons for the different inhibitors

Fig. 3 shows the effect of exposure time on Corrosion Rate of MS in sodium sulphate solution with the different inhibitors. It reveals that when the coupons were subjected to the sodium sulphate and different inhibitors namely: Scent Leaf, Cassava Leaf and Neem Leaf extracts for 100 days (2400 hours), the coupon in Scent Leaf solution had the highest Average CR of 3.302mpy, while Cassava Leaf and Neem Leaf had an Average CR of 2.999mpy respectively. The Cassava Leaf and Neem Leaf had the lowest value of corrosion rate. The average corrosion rate might be due to the factors that influence the efficiency of inhibition, such as temperature, water chemistry, surface condition of the steel, humidity, etc [14]. There was slight fluctuation in the initial stages but corrosion rate decreased with increase in time. The gradient of decrease in corrosion was steep initially up to 4hrs. Thereafter, the rate of change become insignificant and levelled up with overall tendency to increase after 10hrs. these behavioural pattern may be linked to passivation and depassivation not only with time but with other conditions such as temperature, fluctuations, bath composition changes.

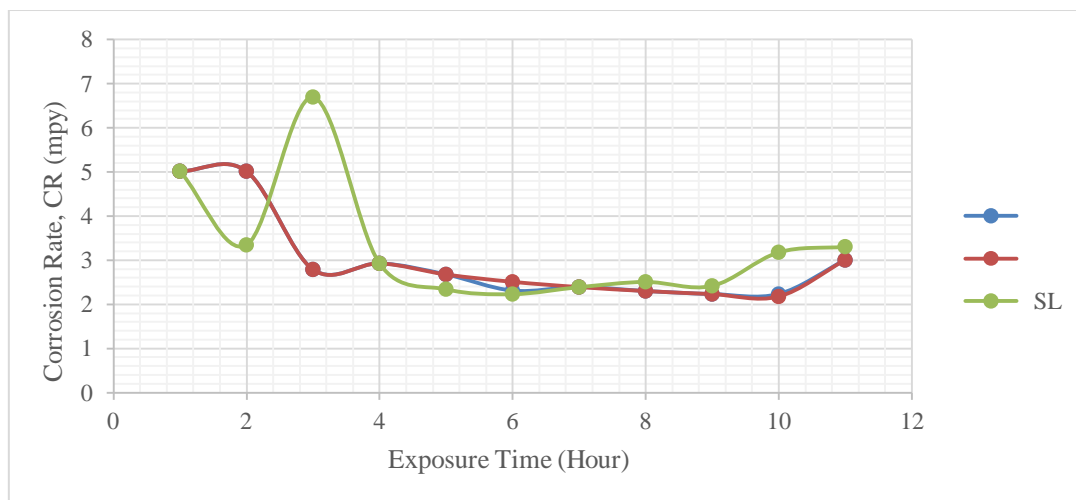


Figure 3: Effect of time on Corrosion Rate of Mild Steel in Sodium Sulphate and inhibitors.

3.3 Effect of immersion time on Inhibition Efficiency (IE) of the coupons for the different inhibitors

Fig. 4 shows the inhibition efficiency of the various inhibitors in sodium sulphate as a function of time. The results reveal that Scent Leaf solution had the highest Average Corrosion Inhibition Efficiency, IE of 97.58%, Neem Leaf was 97.53%, while coupon in Cassava Leaf had the least value of 97.42%. The Scent Leaf was observed to be more efficient, probably, because it also contains high amounts of alkaloid and tannin as shown in Table 2. Loto, 2003 [15] reported that the corrosion inhibition efficiency of some plant extracts have been credited to the presence of tannin in their chemical constituents in several cases [16]. Also correlated with the presence of tannin in the extracts is the bitter taste in the bark and/or leaves of the plants [15]. Neem is bitter in taste. The bitterness is owed to an arrangement of complex compounds called “triterpenes” or more precisely “limonoids” [15,17].

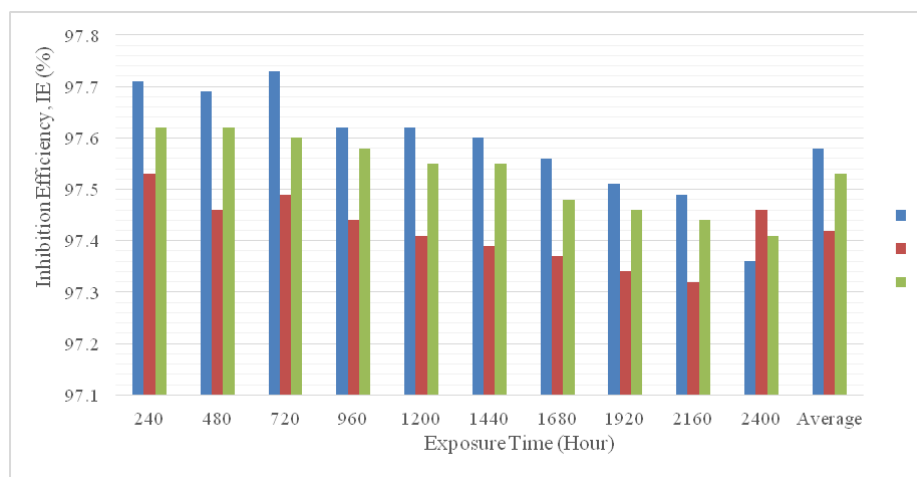


Figure 4: Effect of immersion time on the average inhibition efficiency (IE) with different Inhibitors on mild steel in sodium sulphate medium

Table 2: Qualitative Method Analysis of Scent Leaf, Cassava Leaf and Neem Leaf

Sample	Alkaloid	Tannin	Saponin	Flavonoid	Phenol
Scent Leaf	++	++	-	+	+
Cassava Leaf	+	+++	++	++	++
Neem Leaf	+	+++	+	++	-

KEY

+ = Less, ++ = More, +++ = Abundant and - = None

3.4 Brinell Hardness Test

Fig. 5 shows the hardness values of the coupons investigated with or without inhibitors in the Sodium Sulphate solution. The results reveal that the control sample (unpolished) had the least hardness value of 129HK₁₀₀, Control Sample (Polished) was 131HK₁₀₀, Na₂SO₄ was 154HK₁₀₀, SL and CL had 147HK₁₀₀ respectively, while NL was 143HK₁₀₀. The Control Sample (Polished) exhibited higher value than the Control Sample (Unpolished). This might be due to plastic deformation taking place during polishing of the coupon. While Scent Leaf and Cassava Leaf exhibiting higher values than Neem Leaf might be due to the coupons being adsorbed onto by chemical constituents from the leaf extracts and/or due to compaction of the grain boundaries during polishing.

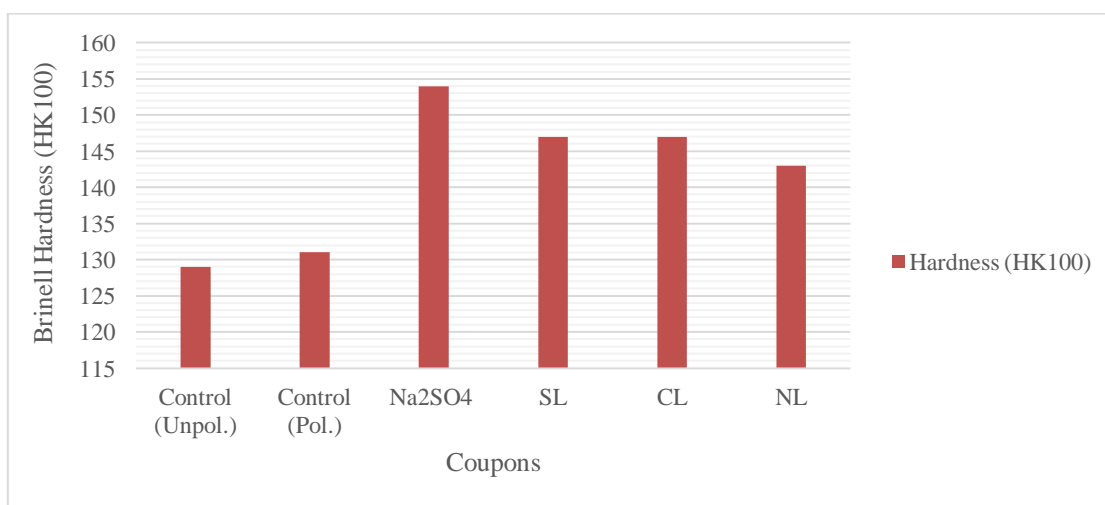


Figure 5: Brinell Hardness Test

3.4 Surface Roughness Analysis

Fig. 6 shows the surface roughness analysis values of the coupons with or without inhibitors. The results reveal that sample Na₂SO₄ had the highest surface roughness value of 8.377µm, Control sample (Pol) had 5.621µm, Control sample (Unpol) had 5.576µm, SL had 5.621µm, CL had 5.779µm, while NL had the least roughness value of 4.658µm. The Na₂SO₄ having the highest surface roughness of 8.377µm might be attributed to much corrosion on the coupon due to absence of inhibitor, while the coupons with inhibitors, it was evident that there might be adsorption of the inhibitor molecules to inhibit corrosion on the metal surface [19].



Figure 6: Surface Roughness Tests

3.5 Bath pH Analysis

Fig. 7 shows that SL, CL, NL, SL/Na₂SO₄, CL/Na₂SO₄ solutions had pH values less than 7 and therefore, they are acidic, while Na₂SO₄ and NL/Na₂SO₄ had pH values greater than 7 which are basic. Pure water is said to be neutral at pH 7, being neither an acid nor a base [18,19]. Pitting and stress corrosion cracking in hostile environments like acidic (HCl and Na₂SO₄) and Halide-Seawater greatly obstruct the application of Mild Steel [10].

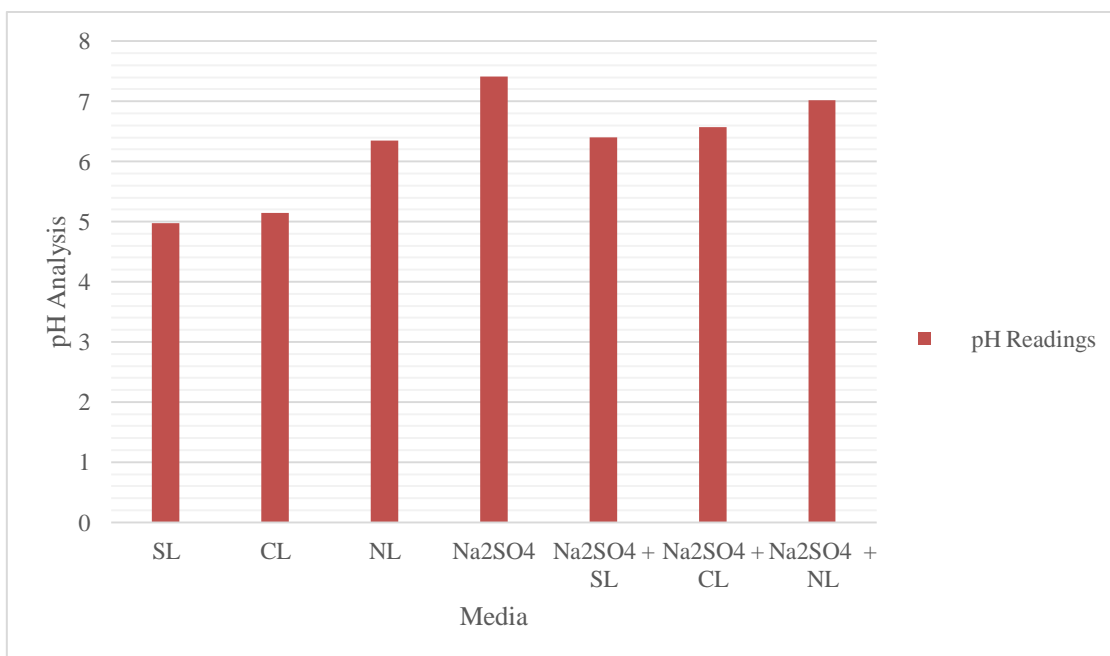


Figure 7: pH Analysis of Bath Concentrations

3.6 Soil pH Analysis

Fig. 8 shows the pH value of Soils from three different locations of Bayelsa State in Nigeria. The pH values of the Soil Samples from the different locations revealed that Ayamasa had 10.89; Angiama had 9.11, while Yenagoa had 7.30 values. The pH values of the various locations are higher than 7 which indicate that they are alkaline. Chlorine, (Cl⁻) ions are major species that cause corrosion. From Table 3 the chloride content of soil at the different locations, Yenagoa had 40.501; Angiama had 39.900, while Ayamasa had 20.379 Cl⁻ ions. It is evident that Yenagoa environment is more predisposed to corrosion than the other environments because it has more chlorine ions which are highly corrosive.

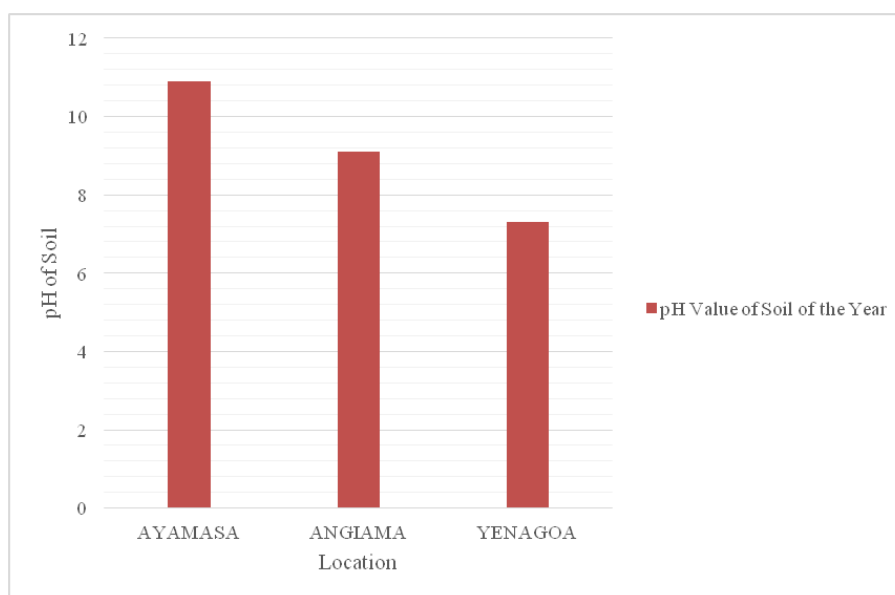


Figure 8: pH values of the soil samples from three locations.

3.7 Elemental Composition of the Soil Samples

Table 3: Elemental composition of the soil samples from different locations.

Element	Concentration (mg/kg) of Soil of the Year		
	AYA	ANG	YEN
Mg	21770.767	2947.920	1784.571
Al	5725.079	7192.359	5146.507
Si	6152.794	6350.295	4958.466
S	168.856	181.051	152.841
Cl	20.379	39.900	40.501
Ca	233.870	342.781	288.045
Ti	1428.817	1559.628	1291.746
Mn	73.123	80.090	43.040
Fe	3654.273	5232.249	3304.503
Co	34.307	45.939	19.969
Mo	9.368	-	-
Sn	3014.131	3184.014	3514.693
Ba	56.446	63.973	-
Cr	-	13.601	-
Zn	-	20.231	-
V	-	-	27.595

KEY: AYA – Ayamasa, ANG – Angiama, YEN – Yenagoa

3.8 Microstructure or Morphological Studies

Fig. 9 a-f shows SEM morphologies of Mild Steel coupons which were exposed for 2400 hours (100 days) in Na₂SO₄ solution uninhibited and inhibited. From the various characterizations conducted on hardness test and surface roughness analysis in Fig. 5 indicated that the coupons in the presence of the inhibitors exerted some passivation. This proves that the extracts might have inhibited corrosion on the Mild Steel through adsorption of the inhibitor molecules on the surface [19].

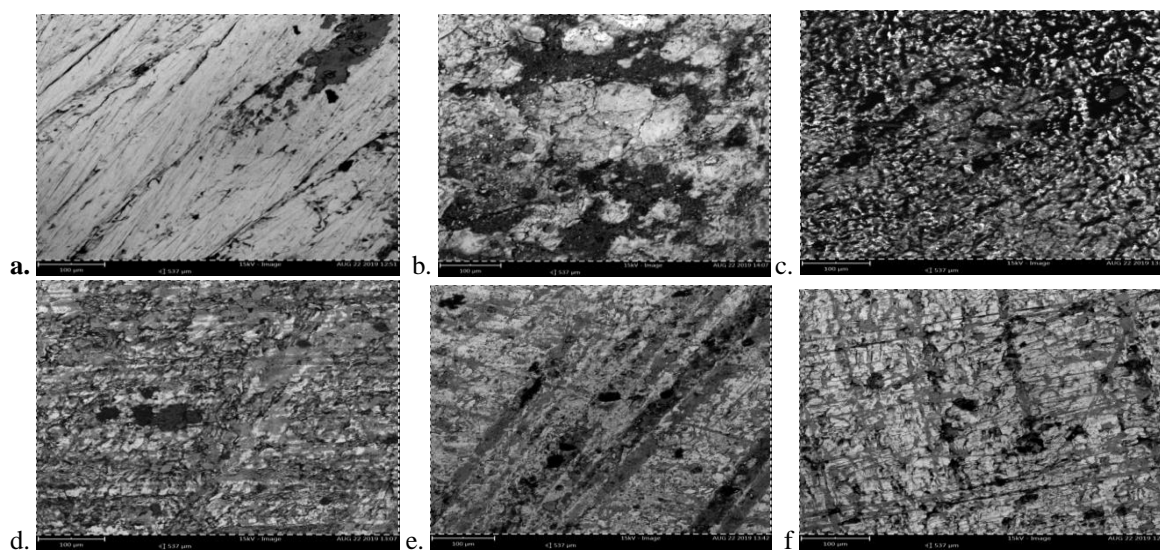


Figure 9: SEM analysis for Mild Steel **a.** Polished **b.** Unpolished **c.** Sodium Sulphate **d.** Scent Leaf **e.** Cassava Leaf **f.** Neem Leaf.

IV. CONCLUSION

Mild steel coupons immersed in the corrosive medium in the presence of inhibitors, the average weight loss revealed that the Scent Leaf solution coupon had the highest weight loss of 0.085g, while Neem Leaf had the least weight loss at about 0.049g.

The corrosion rate was highest in the Scent Leaf solution with an average corrosion rate of 3.302mpy, while Cassava and Neem Leaves exhibited similar inhibition rate at about 2.999mpy.

For Inhibition Efficiency, Scent Leaf solution exhibited the highest average of 97.58%, while Cassava Leaf had the least rate at about 97.42%.

The Brinell harness test revealed that the coupons in the Scent Leaf and Cassava Leaf solutions had the highest value of 147HK₁₀₀ respectively, while the coupon in Neem Leaf solution had the least value at about 143HK₁₀₀.

Surface Roughness analysis shows that Sodium Sulphate coupon in the absence of inhibitors had the highest roughness value of 8.377 μ m, while Cassava Leaf coupon had 5.779 μ m, and Neem Leaf had 4.658 μ m surface roughness respectively.

From the pH analysis, it was observed that Scent Leaf, Cassava Leaf, Neem Leaf, Sodium Sulphate and Scent Leaf, Sodium Sulphate and Cassava Leaf solutions were less than pH 7 which indicated that they are acidic, while Sodium Sulphate and Neem Leaf solutions were greater than pH 7 which indicated that they are basic.

From the soil analysis, it shows that Yenagoa has more Chloride radical than other locations which indicate that it might be more corrosive compared to the others as shown in Table 3.

From the Microstructures, it appears adsorption on the metal surface took place in the presence of inhibitors as compared to the coupons in the absence of inhibitor. This might be confirmed from the results obtained on Weight loss and Corrosion Rate in the absence and presence of inhibitors.

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