Inhibition of mild steel corrosion in HCl solution by plant extract of *Biden pilosa*

S.J. Olusegun [1, 3], T. S. Joshua [2], M.O. Bodunrin [1, 4] and S. Aribo [1]

[1]Corrosion and Electrochemical Laboratory, Department of Metallurgical and Materials Engineering, Federal University of Technology, Akure, Nigeria.
[2]Kogi State Polytechnic, Lokoja, Itakpe Campus, Nigeria.
[3]Departamento de Quimica, Universidade Federal de Minas Gerais (UFMG), Belo Horizonte, MG, Brazil
[4]DST-NRF Centre of Excellence in Strong Materials, African Materials Science and Engineering Network (AMSEN), University of Witwatersrand, Johannesburg, South Africa
arewasegun@gmail.com, arewasegun@ufmg.br

Abstract: The viability of using *biden pilosa (BP)* as corrosion inhibitor in hydrochloric acid environment was investigated. Mild steel substrates that were prepared following standard procedures were exposed at varied temperatures (303, 313, 323 and 333K). Compound identification and corrosion inhibition efficiency of the extract were evaluated using gas chromatography (GC), gravimetric analysis and Tafel extrapolation techniques. Furthermore, scanning electron microscopy was used to establish the corrosion inhibition mechanisms. The results obtained from GC analysis show that four active compounds (Oleic acid, Trans-13-Octadecenoic acid, 3-hydroxyproply ester and cis-13-Octadecenal) were present in the extract. Corrosion rates obtained from weight loss and Tafel extrapolation were in accordance with each other. The extract exhibited 97% inhibition efficiency at 303 K but inhibition efficiency decreased with increasing temperature indicating physiosorption as the main adsorption mechanism. The protection of the mild steel substrate in BP extract containing environment was confirmed by SEM analysis.

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1. Introduction

The degradation of metallic materials during various industrial processes is undesirable (Li et al., 2015). Hence, the search for efficient chemical compounds to minimize the influence of corrosive media on the integrity of steel and other metallic materials becomes imperative. Synthetic corrosion inhibitors are often used in oil industries among others, to suppress dissolution of metallic materials which are often caused by the presence of dissolved gasses - carbon dioxide and hydrogen sulfide (Rajeev et al., 2012). More so, the durability of reinforced concrete metallic materials is also being protected from the concrete paste by adding inhibitors (Yohai et al., 2013). However, in recent times, high cost, difficulty in usage, toxicity to human lives and the environment have necessitated a paradigm shift to non-toxic inhibitors (Lebrini et al., 2011). As a result, there has been increasing research interest in the search for efficient, cost effective, sustainable and eco-friendly inhibitors. Plant extracts fall within these categories. These extracts consist of organic compounds which have polar atoms through which they are adsorbed on the surface of metallic materials to form protective films (Rajeev et al., 2012).

Several plants and animal wastes extract have been investigated and proven to be efficient inhibitors that could be used to mitigate the corrosion of ferrous material in sulpuric, hydrochloric, phosphoric acid among others. Li et al., (2014) studied the effectiveness of the extract from bamboo leaves to mitigate the corrosion of cold rolled steel (CRS) and zinc in 0.2 M citric solution using potentiodynamic polarization in addition to weight loss techniques. 90.7 and 81.7 % was reported as the efficiency of 2 g/l of the extract for steel and zinc respectively. Chitosan extracted from snail shells was explored to inhibit the corrosion of carbon steel in acid solutions by Okoronkwo et al., (2015) using gravimetric and thermometric methods. Their results show that the inhibitor has efficiency of 92 % at the concentration of 5 g/l.

More so, mild steel corrosion in 1 M hydrochloric acid was substantially retarded in the presence of green capsicum annuum fruit extract according to the report of Ji et al., (2012). Maximum efficiency of 86 % was obtained for 1400 mg/l of the extract concentration. The extract was affirmed to act as mixed type inhibitor, based on the result from polarization curve. Langmuir and Temkin model was used to ascertain adsorption characteristics of the extract. However, based on their findings, Langmuir model was found to be most appropriate to describe the adsorption mechanism. Alaneme and Olusegun,

(2012) reported that lignin extracted from sun flower could be used during acid pickling to prevent the corrosion of carbon steel in H_2SO_4 solution. Weight loss technique was used to carry out the research work. The authors reported the maximum inhibition efficiency of 78 % with 15 g/L.

Among other plant-based inhibitors that were previously investigated and reported include *Murraya koenigii leaves* (Quraishi et al., 2010), *Vernonia Amygdalina* (Nwabanne and Okafor, 2011), Water hyacinth (Oloruntoba et al., 2012), *Jatropha Curcas* (Olusegun et al., 2013), *Nicotiana tabacum* (Olasehinde et al., 2013), *Aquilaria Crassna* (Helen et al., 2014) Pectin (Fiori-Bimbi et al., 2015), *Psidium Guajava* (Noyel-Victoria et al., 2015), and *Cucurbita maxima* (Anbarasi and Vasudha, 2017).

It is worth mentioning that most of the aforementioned green inhibitors among several other inhibitors are extracts from unwanted plants (weed). This means that the challenges associated with sustainable production of these inhibitors on a commercial scale has been solved to a greater extent. Therefore the search for different sources of green (plant based) inhibitors would continue to attract the interest of researchers.

Biden Piosa an epitome of unwanted plant (weed) in Nigeria. It is readily available in other parts of the world namely America, Africa, China and Japan (Horiuchi and Seyama, 2006)]. The plant is reportedly used for diabetes, dysentery and biological activities in Nigeria (Okoli et al., 2009). It is also used to repel pests in stored grain as well as aphids, ants, beetle, caterpillars, crickets, mites and termites (Kazembe and Nkomo, 2012). It possesses antibiotic and anti-malarial properties (Chang et al., 2007). The presence of functional group like OH in the extract as revealed by the FTIR analysis carried out by our group necessitated the curiosity to study the corrosion inhibition effect of the extract. Organic molecules that have groups like -OH, double or triple bonds or unpaired electrons have been affirmed to interact easily with metals thereby leading to the protection of metals in aggressive medium (El-Etre, 2006). This research aims to examine B*iden pilosa* extract as a potential inhibitor in hydrochloric medium.

2. Materials and method

2.1 Materials, Extraction/test solution preparation

Mild steel of composition (wt %) Fe=98, C=0.13, P=0.0061, Mn=0.82, Cr=0.08 was used for this study. *Biden pilosa* leaves were obtained from Akure, sun dried and pulverized. The extraction was carried out as reported by Alaneme et al., (2016). The concentration of the test solution prepared from the extract was varied from 0.1-0.5 g/L of 1 M HCl.

2.2 Temperature study using weight loss measurement

Temperature study was carried out at the temperature variation of 303, 313, 323 and 333 K. 100 ml of the blank and the prepared extract solutions were poured into respective beakers. Mild steel coupons of known weight were immersed inside each beaker. The beakers were transferred into thermostat controlled water bath and maintained at the aforementioned temperature for 6 hours. The substrate were retrieved after the set time and reweighed.

Corrosion rate was calculated from weight loss using the expression in equation 1 (Fiori-Bimbi et al., 2015).

$$CR = \frac{\Delta W}{At} \quad (1)$$

Where CR is the corrosion rate in $gcm^{-2}h^{-1}$, ΔW is the weight loss in g, A is the area of the coupon in cm^2 and t is the exposure time (h).

Equation 2 and 3 were used to calculate the inhibition efficiency and surface coverage respectively.

$$IE_{wt}\% = \left(1 - \frac{CR_{inh}}{CR_{blank}}\right)100 \quad (2)$$

Where IE_{wt} is the inhibition efficiency from weight loss technique, whereas CR_{inh} and CR_{blank} are the respective corrosion rates of the coupon in the test solution with and without the extract

$$\theta = \left(1 - \frac{CR_{inh}}{CR_{blank}}\right) \quad (3)$$

Where θ is the surface coverage.

Effect of immersion time was also put into consideration. This was carried out for 7 days; meanwhile the samples that were immersed in the various solutions (blank and inhibitor solutions) were retrieved every day, cleaned with organic solvent and weighed. The data obtained for each day were plotted against the exposure time.

2.3 Electrochemical measurements

Potentiodynamic polarization was measured using Autolab PGSTAT 204N at the scan rate of 1.0 mV/s and at a potential initiated at -250 mV to + 250 mV with respect to OCP. After each experiment, the electrolyte and the test sample were replaced. The area of the working electrode is 1 cm^2. Tafel parameters listed in Table 1. $IE_{Icorr}\%$ was calculated using the current densities.

$$IE_{Icorr}\% = \left(\frac{I^{o}_{corr} - I_{corr}}{I^{o}_{corr}}\right)100 \quad (4)$$

Where I^{o}_{corr} and I_{corr} are the corrosion current densities of the blank and extract solution respectively. $IE_{Icorr}\%$ is the inhibition efficiency from the Tafel extrapolation data.

2.4 Characterization and surface analysis

Fourier transform infra-red spectrophotometer (Perkin-Elmer-1600) was used to characterize the extract while JSM 7600F Jeol ultra-high resolution

Scanning Electron Microscope was used to examine the surface topography.

2.5 GC/MS Chromatography

Gas chromatography–mass spectrometry (GC–MS) analysis was carried so as to identify the compounds which are present in BP extract, using Agilent Technologies 5975 series MSD. The experiment was carried out in a wall coated capillary column (BP-1; (30 m x 0.32 x 0.25 µm) at the initial temperature of 60°C, and maintained at this temperature for 2 min. At the end of this period, the oven temperature rose up to 250°C, at the rate of 2°C/min, and maintained for 10 min. The carrier gas was helium at a flow rate of 1 ml/min, split mode, with a ratio of 1:5 and injection volume of 1 μl in a CH_2Cl_2 and the ionization voltage 70 eV.

3 Result and Discussion

3.1 Extract Characterization

Zhang et al., (2007) stated that GC-MS gives detailed qualitative information about organic samples. GC-MS study of ethanolic extract of *Biden pilosa* confirm the presence of four compounds.

The compound identified with highest retention time (75.43 min) is cis-13-Octadecenal while Oleic acid has the lowest retention time (61.42 min). Inhibition process of metallic materials has been attributed to the presence of N, S, O, conjugated double bond in the (Noor, 2009).

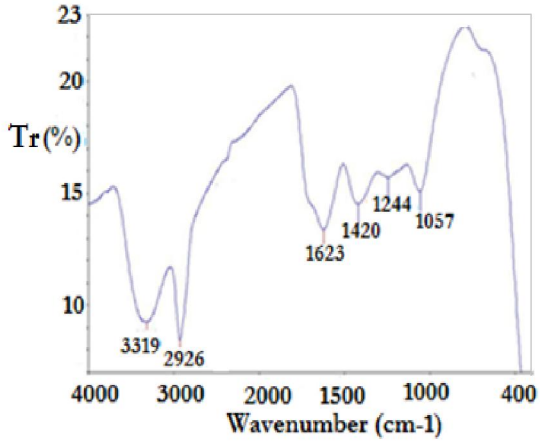

Figure 1. IR spectra of the ethanol extract of *Biden pilosa.*

Figure 1 shows the FTIR spectrum (Transmittance % (Tr) against wavenumber cm^{-1}) of the BP extract at different bands. The broad peaks at 3319 cm^{-1} is due to OH stretching vibration, 2926 and 1623 cm^{-1} bands correspond to –CH and C=C stretching respectively. C-O and OH found to be due to 1244 and 1057. Organic molecules that have these functional groups have been attested to interact easily with metals thereby leading to the inhibition of metals in aggressive medium (El-Etre, 2006).

3.2. Weight loss with respect to time and inhibitor concentration

Figure 2 is the plot of reduction in weight loss with respect to the exposure time at room temperature. The plot indicates that the values of weight loss cumulatively increase with time. However, the addition of the extract reduced the metal dissolution significantly. Corrosion inhibition is believed to occur as a result of the extract constituents being adsorbed on the surface of the coupons, the extract interacted with iron cations and then led to the insoluble complexes being formed as a product on the coupon surface that prevented the rapid corrosion of the mild steel coupons to occur (Zor et al., 1999). At the end of the 7 days exposure time, weight loss was reduced from 0.5975 g (Blank solution) to 0.0832 g (0.5 g/l BP extract) which amount to 86.6 % inhibition efficiency.

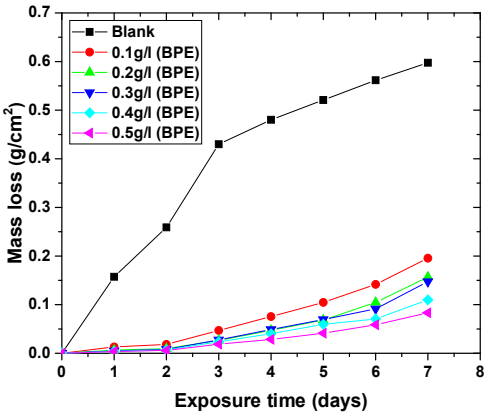

Figure 2. The variation of weight loss of the mild steel coupon

3.3 Tafel extrapolation study

Figure 3 connotes plot of natural logarithm of corrosion current density (log i) in Acm^{-2} against corrosion potential in volts (v) with respect to silver/silver chloride used as reference electrode (Ag/AgCl). The I_{corr} values were progressively decreased as the amount of the inhibitor that was added to the acid solutions was increased. Parameters evaluated from the plot are presented in Table 1.

The value of I_{corr} was 200 µA/cm^2 before inhibitor was introduced into the system; meanwhile, it was reduced to 25.61 µA/cm^2 when 0.5 g of the extract was added. This was due to the constituents of the extract covering the surface of the coupons, thereby retarding the dissolution of the metal into the electrolyte. Cathodic or anodic type inhibitor has been ascribed to inhibition process whenenver the deviation in E_{corr} is greater than 85 mV in presence of the

inhibitor compare to the blank solution (Bammou et al., 2014). The addition of BP extract did not affect the E_{corr} significantly as showed in Table 1, in view of this; the inhibitor can be regarded as mixed-type inhibitor that affects the reaction at the anode and the cathode.

Table 1:

Biden pilosa extract (g/L)	E_{corr} (mv/SCE)	I_{corr} (μA/cm^2)	Ba (mV/decade)	Bc (mV/decade)	IE_{Icor}(%)
Blank	-465	200	91.9	85.6	-
0.1	-458	48.9	74.3	62.6	75.5
0.2	-460	45.6	75.6	73.2	77.2
0.3	-455	37.8	49	46.4	81.1
0.4	-459	34.1	53.4	60.1	83
0.5	-493	25.6	24.6	51.2	87.2

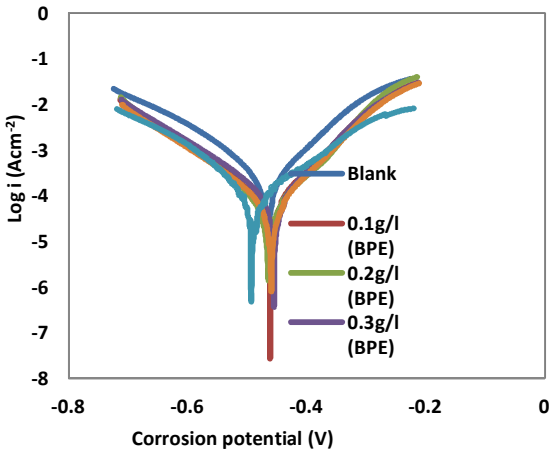

Figure 3. Tafel polarization curves.

3.4 Temperature study

Graphical result presented in Figure 4 shows the influence of temperature on the efficiency of BP extract. Comparing the IE at 303K and 333K for all the extract concentration, there was a noticeable decrease when the temperature of the system was raised.

Whenever efficiency of an inhibitor is reduced as a result of the temperature of the system being raised, the extract is believed to have been physically adsorbed on the coupons (Deyab, 2014). At the moment the temperature of the system was increased, the equilibrium of the reaction is shifted, this resulted to higher desorption of the BP extract from the surface of the coupons than its adsorption (Hmamou et al., 2013). The solubility of adsorbed constituents of BP at higher temperature contributed to its low efficiency.

Activation energy was evaluated by using the Arrhenius equation below, plotting $\log CR$ against reciprocal of temperature.

$$\log CR = \log A - \frac{E_a}{2.303RT} \qquad (5)$$

Where CR is the corrosion rate, R is the gas constant, T is the absolute temperature, A is the pre-exponential factor and E_a is the activation energy.

The slope $(-\frac{E_a}{2.303RT})$ of individual line of blank and different concentration of the extract in Figure 5 was used to evaluate the activation energy E_a

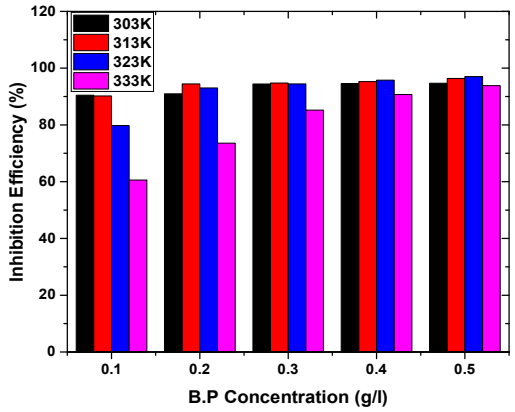

Figure 4. Percentage efficiency of *Biden pilosa* extract with temperature.

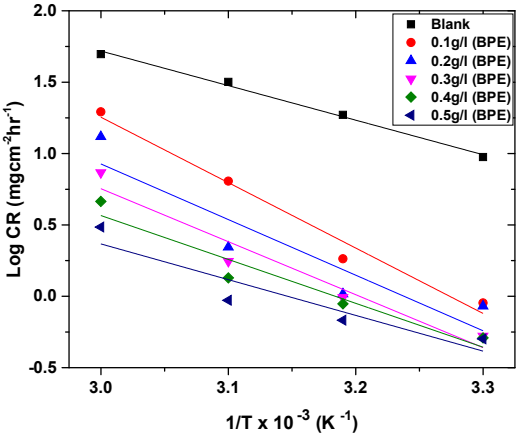

Figure 5. Arrhenius plot for mild steel corrosion

The E_a obtained for the blank solutions is lower than the inhibited solutions this is because the extract

increased the energy needed for the dissolution of the metal to occur in the acid solutions. Chemical reaction process is slower at higher activation energy in view of this the corrosion reaction of the test solution that contained BP extract, was impeded due to the raised in activation energy of the reaction process. Similar observation was also reported by Faustin et al., (2015). BP extract raised the activation energy route between the reacting molecules and the product that is being formed.

The corrosion rates of the coupons in all the test solutions increased with increased in temperature. This is expected because at higher temperature the reacting molecules possess more kinetic energy; however, the presence of the BP extract on the other hand raised the activation energy above that of the blank solution. Therefore the kinetic energy of the molecules (of the test solutions that contained the extract) upon collision is lower than the activation energy needed for the rapid dissolution of the metallic material to take place. This explains the reason for the decreased in the corrosion rates when the extract was introduced into the test solution.

Table 2:

Acid medium	Concentration of *Biden pliosa extract* (g/l)	Activation energy, E_a (kJ mol^{-1})	Enthalpy of activation, ΔH_a ($kJmol^{-1}$)	Entropy of activation, ΔS_a ($Jmol^{-1}$ K^{-1})
1 M HCl	Blank	46.36	43.72	-81.82
	0.1	87.75	85.11	33.46
	0.2	74.58	71.94	-12.29
	0.3	71.08	68.44	-26.12
	0.4	58.86	56.22	-66.39
	0.5	47.93	45.29	-103.01

The E_a values listed in Table 2 decreases as the concentration of the BP in the test solutions increases this result is consistent with what was reported by Soliman et al., (2014). Decrease in activation energy with respect to increase in the concentration of the inhibitor was noted to be as a result of shift in the net corrosion reaction from the portion of the metal surface uncovered by inhibitor to the covered part (Noor, 2009). Physical adsorption has been attributed to corrosion inhibition process in which the activation energy of the test solution that contains the inhibitor is higher than solution without inhibitor (Ebenso et al., 2008).

Enthalpy (ΔH_a) and the entropy of activation (ΔS_a) were evaluated using the transition state equation (Umoren et al., 2014).

$$\frac{\log CR}{T} = \left[\log\left(\frac{R}{nh}\right) + \frac{\Delta S_a}{2.303R}\right] - \frac{\Delta H_a}{2.303RT} \quad (6)$$

Where h is the Planck's constant, n is the Avogadro's number, ΔH_a and ΔS_a are the enthalpy and entropy of the activation respectively. CR, R, and T have been defined in the previous equations. From the plot shown in Figure 6, ΔH_a and ΔS_a were calculated from the slope and intercept respectively.

The values of ΔH_a calculated being positive imply endothermic reaction process of the dissolution of the coupons. However, the addition of the extract raised the values of ΔH_a above the blank solution; this means that the heat that will be needed for the dissolution of the metal to occur will be higher than in the absence of the extract. This gives insight to the reason behind retardation in the corrosion rate values when the extract was added. Similar trend of results was also reported by Alaneme et al., (2015).

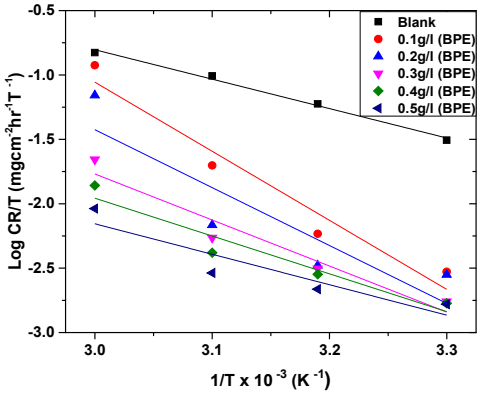

Figure 6. Transition state plot for mild steel corrosion.

In addition, the corresponding values of activation energy (E_a) are greater than that of the Enthalpy (ΔH_a) this observation has been affirmed to be as a result of evolution of gas (hydrogen) during corrosion process which must involve gaseous reaction and associated with a decrease in the total reaction volume. The calculated $E_a - \Delta H_a$ from the data obtained is 2.64kJ/mol, this value is approximately around the average value of RT which is 2.69kJ/mol, and shows that the corrosion reaction process is unimolecular reaction (Noor, 2009).

3.5 Inhibition adsorption study

Corrosion inhibition process had been regarded to occur due to the adsorption of inhibitor on the metallic materials (Karthikaiselvi and Subhashini, 2014), due to this, the data obtained was subjected into various adsorption isotherm models among which; Langmuir isotherm was found to be more applicable.

Equation 7 defines Langmuir isotherm.

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \qquad (7)$$

Where C is the concentration of the inhibitor, K_{ads} is the equilibrium constant of adsorption and θ is the surface coverage.

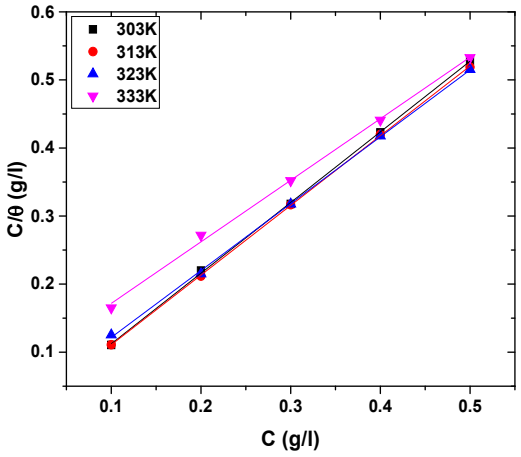

Figure 7. Langmuir adsorption isotherm plot.

Figure 7 shows the plot of $\frac{C}{\theta}$ against C. The parameters obtained from the plot are itemized in Table 3. The values of the slopes being approximately 1 show that the extract molecules occupied a sole adsorption sites and that there is no interaction between them (Yaro et al., 2013).

Table 3:

Temp.	K_{ads}	r^2	Slope
303K	119.84	0.9998	1.038
313K	118.69	0.9999	1.023
323K	42.71	0.9997	0.982
333K	12.320	0.9991	0.904

K_{ads} evaluated from the reciprocal of the intercept, decreased as the temperature of the system was raised. This affirmed that the adsorption of constituents of BP extract on the coupons is a physical process and unstable at higher temperature. Fouda et al., (2014); Olusegun et al., (2016) noted that small K_{ads}value indicates that interactions between inhibitor and metallic material are weak whereas, larger K_{ads}implies strong bond.

3.6 SEM surface examination

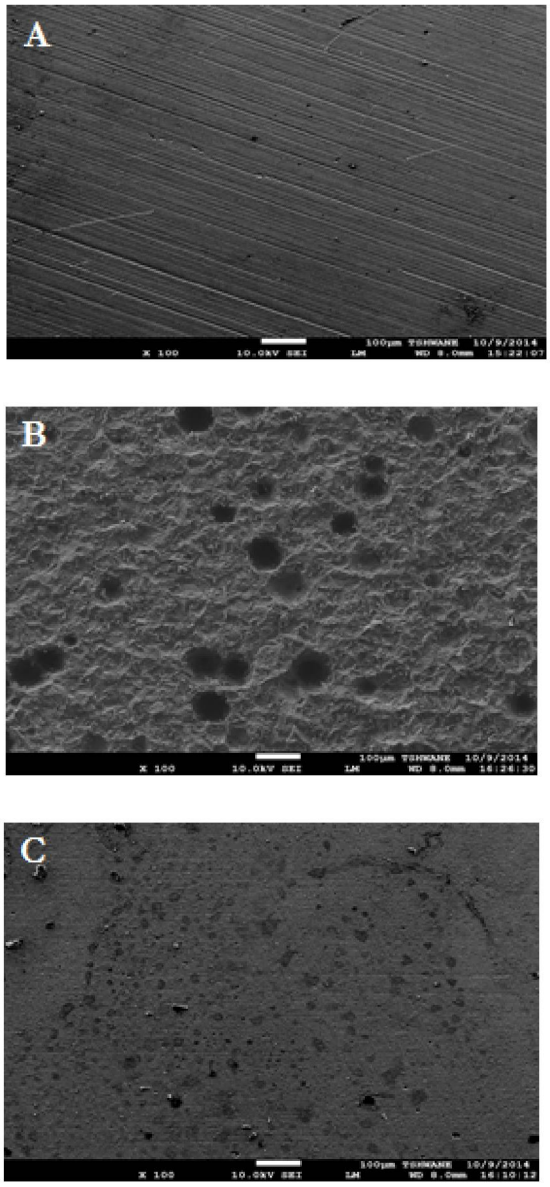

Figure 8. SEM images of the coupons (a) As received (b) Immersed in blank test solutions (c) Immersed in test solution containing the extract.

Presented in Figure 8 are the SEM images of the coupons surface. The potency of the extract to reduce the damage caused by corrosion is more visible from the images. The image of the coupon before it was immersed in the test solution is uniform and smooth with some lines which were as a result of polishing effect. Whereas noticeable pits was observed when it was immersed in the test solution without BP extract. This was the consequence of the aggressive corroding medium. However, image of the coupon immersed in

the test solution containing the extract revealed that the extract remarkably inhibited the corrosion reaction since the surface is comparatively smoother.

The EDX spectra of the surface of the coupons immersed in the test solutions (Blank and extract solutions) show the presence of O which is as a result of formation of iron oxides. However, its elemental composition (in weight) after immersion in the blank solution is as follow: Fe 60.57, O 14.82, C 10.48 and Cl 14.13, meanwhile, in the presence of *Biden pilosa* extract the composition are Fe 85.3, O 5.43, C 8.26 and Cl 1.01. The presence of the extract significantly reduced O and Cl.

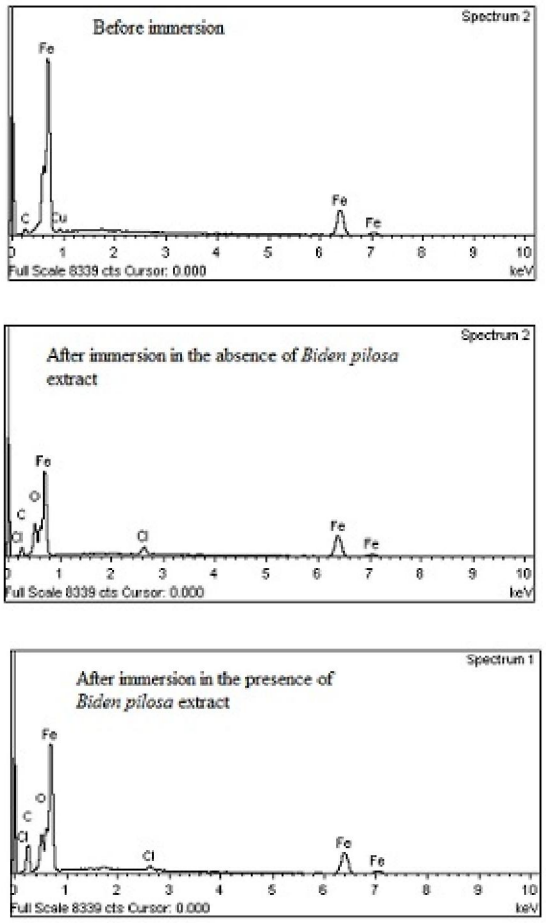

Figure 9. EDX spectra of the coupons.

4. Conclusion

Ethanolic extract of *Biden pilosa* leaves has been studied for corrosion mitigation of mild steel by using gravimetric and Tafel polarization technique.

- The extract exhibits appreciable inhibition potential. Its efficiency is sensitive to change in temperature.
- Results obtained were consistent with physical adsorption process.
- Biden pilosa leaves extract could be regarded as mixed-type inhibitor.
- SEM images revealed the suitability of BP as inhibitor for the protection of engineering metallic from the corroding medium.

Corresponding author:
Olusegun Sunday J.
Departamento de Quimica,
Universidade Federal de Minas Gerais (UFMG),
Belo Horizonte, MG, Brazil.
arewasegun@gmail.com,
arewasegun@ufmg.br

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