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STUDY OF HYPTIS SUAVEOLEN L POIT LEAVES EXTRACT AS CORROSION INHIBITOR ON MILD STEEL IN H2SO4 SOLUTION

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ABSTRACT
In this study, *hyptis suaveolen L poit* (HSLP) leaves extract as corrosion inhibitor on mild steel in H_2SO_4 solution was carried out. Gas chromatography- Mass spectrometer analysis was conducted to identify active components present in the extract. Corrosion inhibition of the plant extract on mild steel in 1M H_2SO_4 was evaluated using weight loss and potentiodynamic polarization methods. The weight loss method results revealed that corrosion rate decreased with increasing inhibitors concentration resulting in inhibitor efficiency of 98 %. The adsorption of *hyptis suaveolen L poit* leaves extract on the metal surface obeys Langmuir adsorption isotherms. Potentiodynamic polarization data indicated that *hyptis suaveolen L poit* leaves extract is a mixed-type corrosion inhibitor, they retarded both cathodic and anodic reactions. All the studied parameters gave good anti-corrosive properties against corrosion of mild steel in the tested solutions.

INTRODUCTION
Corrosion inhibition is one of the most practical methods for protection against corrosion, especially in acidic media. Among numerous inhibitors that have been tested and applied industrially as corrosion inhibitors, those that are non-toxic or low-toxic are now far more strategic than in the recent past. In the 21st century, research in the field of ''green'' or ''eco-friendly'' corrosion inhibitors has been addressed toward the goal of using cheap, effective compounds at low or ''zero'' environmental impact (Deng and Li, 2012).

Many industrial processes and media are usually rich in elemental gases, inorganic salts, and acidic solutions most of which influence corrosion rates and mechanisms (Abu and Owate, 2003; Abiola and Oforka, 2005). Metals including various grades of mild steels, are usually exposed to the action of these gases, bases or acids in industries. Processes in which acids play a very important roles are acid pickling, industrial acid cleaning, cleaning of oil refinery equipment, acid descaling. The exposure of these metals to corrosive acidic media can be most severe but in many cases, corrosion inhibitors are widely used to prevent or reduce the corrosion rates of metallic materials in these acid media. Because of the toxic nature and high cost of some chemicals currently in use, it is necessary to develop environmentally acceptable and less expensive inhibitors. Natural products can be considered as good sources for this purpose. The possible replacement of some expensive chemicals as corrosion inhibitors for metal in acid cleaning process by naturally occurring substances of plant origin has been studied by researchers (Aisha et al., 2010). Natural products of plant origin contain different organic compounds e.g. alkaloids, tannis, pigments, organic and amino acids, and most are known to have inhibitive action. (Abiola, et al 2004). Also, a number of organic compounds are known to be applicable as corrosion inhibitors for mild steel in acidic environment (Umoren, et al 2008).

Such compounds typically contain nitrogen, oxygen or sulphur in conjugated system and function via adsorption of the molecules on the metal surface, creating a barrier to corrodent attack.

According to Sangeetha et al (2011) extracts of plant materials contain a wide variety of organic compounds. Most of them contain heteroatoms such as P, N, S, O. These atoms coordinate with the corroding metal atom (their ions), through their electrons. Hence protective films are formed on the metal surface and hence corrosion is prevented.

Use of naturally occurring substances to inhibit corrosion of metals in acidic (and alkaline) environments has been reported by researchers (Satapathy et al., 2009; Rosliza et al., 2010; Sheeja and Subhashini., 2010; Znini et al, 2012; and Salaghi et al., 2014). However, the use of alcoholic extract of *hyptis sauveolens L poit* extract as a green inhibitor has not been reported elsewhere.

The objective of the present study is therefore, to investigate the inhibitive and adsorption properties of hyptis *sauveolens L poit* extract leaves for the corrosion of mild steel in H_2SO_4.

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Hyptis suaveolens L Poit is a well-known pseudo-cereal plant in the Latin American region and in many regions of Africa including Nigeria. It grows to a height of approximately 2 meters, has branches and long white piliferous stems. Its flowers are purple or white, its leaves oval, wrinkled and pointed and flourishes well in both warm and semi-warm regions (See Plate 1).

Scientific research (Sharma et al, 2013) has shown that HSLP possesses antifertility, anti-inflammatory, and antiplasmodial properties which makes it a good traditional cure by many population in several parts of the world.

MATERIALS AND METHODS
2.1 Preparation of *hyptis sauveolens L poit* leaf extract
Freshly harvested leaves of *hypis sauveolens poit* were washed under running water, air dried and pulverized (Deng and Li, 2012). The pulverized HSLP extract was soaked in 500 ml of methanol in an extraction thimble for three days. The liquid was allowed to drain into an evaporation dish and the extract rinsed finally with SME ethanol. The extracted solution was then evaporated in a hot plate and the residue scrapped from the dish as solid extract.

2.2 Gas chromatography-mass spectrometry (GC-MS)
The HSLP extract was characterized using GC/MS model 6890 Series Hewlet packerd. About 20 ml of the plant extract was analyzed by GC-MS using GC/MS model 6890 Series Hewlet Packerd equipped with flame ionization detection and a CPB capillary fused silica column. The oven temperature of the column was held at 70ºC for 5 min then programmed at 20ºC/min to 250ºC, held for 20 min. Other operating conditions were as follows: carrier gas Helium (99.9%); inlet pressure 76 kPa; injector temperature, 250ºC; and detector temperature, 310ºC.

2.3 Specimen preparation
Mild steel plate having composition as shown in Table 1 was obtained from steel market in Makurdi, Benue State, Nigeria. The steel was cut into coupons, measuring approximately 20 x 20 x 4 mm. The specimens were machined to a smooth finish then mechanically abraded with 120, 220,400, 800, and 1000 grades of emery papers. Polished sample were degreased with acetone, rinsed with distilled water, dried, and weighed on an electronic weighing balance.

Table 1: Basic Information of Mild Steel Sample

Element	C	Si	Mo	P	Cr	Ni	Mo	Al	Cu	Ti	Nb	V
% content	0.27	0.16	0.68	0.014	0.029	0.06	0.02	0.00	0.21	0.00	0.00	0.00

2.4 Solution preparation
An appropriate concentration of 1M H_2SO_4 solution was prepared using triple-distilled water. The concentration range of HSLP leaf extract employed was varied from 0 – 5g/l in equal steps.

2.3 Weight loss measurement
Experiments were performed at 25ºC with different concentrations of *Hyptis suaveolens* leaf extract (ranging from 0 to 5g/l in equal steps). The immersion time for the weight loss was 48 hrs. At the end of each period, the specimens were taken out, washed with distilled water and dried to remove the corrosion products and then weighed accurately. This procedure was repeated for 96 hrs, 144 hrs, 192 hrs and 240 hrs. Weight loss for each of the specimen was recorded as the difference between the final and initial weight of the specimens. The results of the weight loss experiments are the mean of three runs, each with a fresh acid solution.

The average corrosion rate was determined using the relation in equation 1 (Fontana, 1994).

$$\text{Corrosion Rate (mm/yr)} = \frac{87.6\Delta w}{\rho AT} \qquad (1)$$

Where,
Δw = weight loss of mild steel coupon (kg)
ρ = Density of mild steel (kg/cm^3)
A = Surface area of mild steel (cm^2)

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T= immersion time (hrs)

The percentage inhibition efficiency (IE %) and surface coverage (θ) of each concentration was also, calculated using equations (2) and (3) respectively.

$$IE\% = \frac{r_o}{r_{o-r}} \times 100 \qquad (2)$$

And,

$$\theta = \frac{r_o}{r_{o-r}} \qquad (3)$$

Where r_o and r are the corrosion rates in the absence of and presence of inhibitors respectively

The interactions between the extracts and the mild carbon steel surface were examined by Langmuir adsorption isotherm after series of trials using other absorption isotherms such as Temkin, Frumkin and Flory-Huggins for *Hyptis suaveolens* leaf extract. The inhibition efficiency is directly proportional to the fraction of the surface covered by adsorbed molecules (θ). Therefore, each of these adsorption isotherms was tested in terms of their descriptions of the adsorption behavior of extracts on mild steel surface in 1M H_2SO_4 solution. The Langmuir absorption isotherm was carried out by plotting C/θ vs C; where C is the concentration and θ, the surface coverage.

Langmuir adsorption isotherm is mathematically expressed as:

$$\frac{C}{\theta} = C + \frac{1}{K_{ad}} \qquad (4)$$

where, C is the concentration of the inhibitor θ is the fractional surface coverage, and K is the adsorption equilibrium constant.

2.4 Electrochemical measurements

The electrochemical experiment was carried out using AUTOLAB PGSTAT 204N instrument. The experiments were performed using a three-electrode corrosion cell set-up comprising of mild steel as the working electrode (surface area = $0.76cm^2$), saturated silver/silver chloride as reference electrode, and platinum rod as counter electrode (Satapathy et al., 2009). The test electrolyte was 1M solution of H_2SO_4 in the presence and absence of the extract. All electrochemical experiments were conducted at room temperature ($25\pm2°C$) using 100 ml of electrolyte in stationary conditions. The working electrode was immersed in a test solution for 30 minutes until a stable open circuit potential was attained. The working electrodes were prepared by attaching an insulated copper wire to one face of the sample using an aluminum conducting tape, and cold mounting it in resin.

Potentiodynamic polarization measurements were carried out using a scan rate of 1.0 mV/s at a potential initiated at -250mV to + 250mV with respect to open circuit potential (OCP). The above procedure was repeated for each concentration of HSLP. After each experiment, the electrolyte and the test sample were replaced. The linear Tafel of the anodic and cathodic curves were extrapolated to corrosion potential to obtain the corrosion current densities (I_{corr}) and corrosion potential (E_{corr}). Anodic Tafel slope (βa) and cathodic Tafel slopes (βc) were determined from the experimental curve. The IE_{Icor} (%) was calculated using the current densities.

$$IE_{I_{cor}}(\%) = \frac{I^0{}_{corr} - I_{corr}}{I^0_{corr}} \qquad (5)$$

Where, i^0_{corr} and icorr are the corrosion current density values in the absence and presence of inhibitor, respectively.

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RESULTS AND DISCUSSION
3.1 Characterization of *hyptis sauveolens L poit* leaf extract
The phytochemical analysis conducted in this study revealed types, quantity and qualities of the bioactive compounds present in the selected plant materials that are capable of corrosion inhibition. Table 2 revealed the presence of tannins, alkaloids flavonoids, and plytosteriods in the HSLP extract. However, saponins, glycoside, phenols were not detected in the extract and the compounds present were found to be low, moderate and high for plytosteriods and alkaloids, tannins, and flavoids respectively. The presence of these compounds, (alkaloids, flavonoids, and plytosteriods) have been reported to promote the inhibition of mild steel in aggressive acidic media (Alaneme et al, 2015). These findings agreed with the ones reported by several other researchers including Satapathy et al (2009), and Rosliza et al (2010).

Table 2: Phytochemical constituents of Hypis suaveolens Poit leaf extract

Phytochemical constituents	Results
Tannin	++
Alkaloids	+
Plytosteriods	+
Flavonoids	+++
Saponins	-
Glycoside	-
Phenols	-

Key: (-) Not detected, (+) Low, (++) Moderate, (+++) High

3.1.2 Chemical composition of *hyptis suaveolens L poit* leaf extract
Gas chromatography (GC) – mass spectroscopy (MS) spectra of *hyptis suaveolens poit* extract shown in Figure 1 (and Table 3) identified 13 major components, representing about 100% of the compounds in the extract. The high peaks related to low retention times are mainly polar plant compounds while the small peaks may be attributed to compounds present in small quantities as well as degenerated major compounds (Satapathy et al, 2009). Table 3 further showed terpinolene (14.1%), beta-caryophyllene (12.3%), terpinen-4-01 (9.8%), beta-Ocimene (9.2%), 1,8-cinerle (8.7 %) and biclyclogermacrene (7.1%) as the major compounds of the extract while beta-phellandrene (5.6%), eucalptol (5.2%), limonene (5.0%), sabinene (5.0%), beta-pinene (4.5%) and fenchone (4.1%) were shown as the compounds in smaller quantities. These results are similar with the one obtained by Znini et al (2012) for extract of Asteriscus gravveolens utilized for corrosion inhibition of mild steel in sulphuric acidic medium. It is clear from the above that our choice of methanol extract of HSLP is justified for the fact that retention time of majority of compounds is close to each other and it is very difficult to separate them (Satapathy et al, 2009).

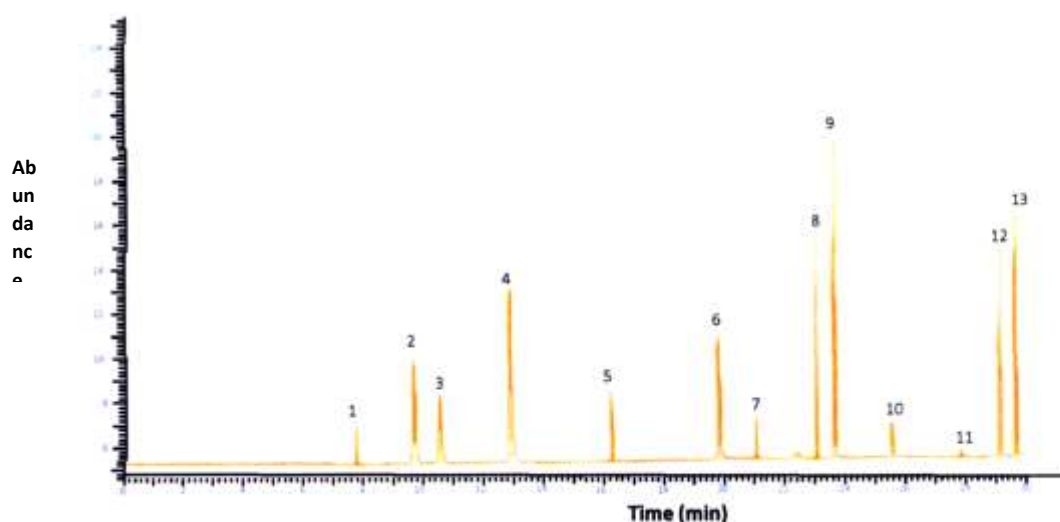

Plate 1: *Hyptis sauveolens L poit* **plant**

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Figure 1: GC-MS of Hypis suaveolen poit leaf extract

Table 3: Chemical composition of Hyptis Sauveolens L Poit

Peak number	Component name	Percentage (%)
1	Sabine	5.0
2	Limonene	5.0
3	Biclyclogermcrene	7.1
4	Beta-phellandrene	5.6
5	1, 8 – Cinerle	8.7
6	Menthe - 2, 4 – diene	9.2
7	Eucalptol	5.2
8	Beta-caryophyllene	12.5
9	Terpinolene	14.1
10	Fenchone	4.1
11	Beta- pinene	4.5
12	Beta- ocimene	9.2
13	Terpinen -4, o1	9.8

3.2 Effect of immersion time and concentration

Corrosion rate (mm/yr) of mild steel exposed to 1M H_2SO_4 at 25°C, as a function of immersion time and as a function of concentration of plant extract is presented in Figures 2 and 3 respectively. It is observed from Figure 2 that corrosion rate of mild steel at all concentrations of the HSLP leaf extract increased steadily with increasing exposure time except at 0 g/l leaf extract where a rapid increase was observed. This is in agreement with Aleneme et al (2015) where it was established that corrosion rate varied linearly with exposure time in uninhibited and inhibited acid solutions. The authors explained further that decrease in inhibition for long periods of exposure can be attributed to the depletion of available inhibitor molecules in the solution due to complex formation between iron and inhibitor molecules. In Figure 3, corrosion rate of mild steel decreased with increasing concentration of HSLP leaf extract. This behavior could be attributed to the increase in adsorption of HSLP at the metal/solution interface on increasing its concentration. The extent of the decrease in corrosion rate was found to depend on the concentration of additive and these findings were in accordance with the works done by Alaneme et al (2015), Rosliza et al (2009), Deng and Li (2011), and Sheeja and Subhashini (2010) while investigating rice husk, natural honey, Ginkgo leaves extract, and grewia tiliaefolia bark extracts respectively as inhibitors. The adsorption of such phytochemical constituents (Table 2) on the metal surface creates a barrier for charge and mass transfer leading to decrease in the interaction between the metal and the corrosive environment. Negm et al (2013) also attributed this adsorption of inhibitor molecules on the metal surface in either physical or chemical adsorption where the metal surface becomes negatively charged by adsorbing the anions on the surface.

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Corrosion rate varied from 0.28- 4.8 mm/yr and decreased with increasing concentrations of extract in 1M H_2SO_4 solution. The inhibition efficiency ranged from 75.9 - 94.2% and increased with increasing concentration to a maximum value of 94.34%, at 10 g/l. This is an indication that the plant extract acted as a good inhibitor for mild steel in 1M H_2SO_4 solution. This behaviour could be due to the fact that adsorption coverage of inhibitors on metal surface increases with the inhibitor concentrations resulting in the blocking of the reaction sites, and protection of the mild steel surface from the attack of the corrosion active ions in the acid medium. That this adsorption was influenced by the nature and surface charge of metal, the type of aggressive electrolyte and the chemical structure or components of the plant extract is reported in Znini et al (2012).
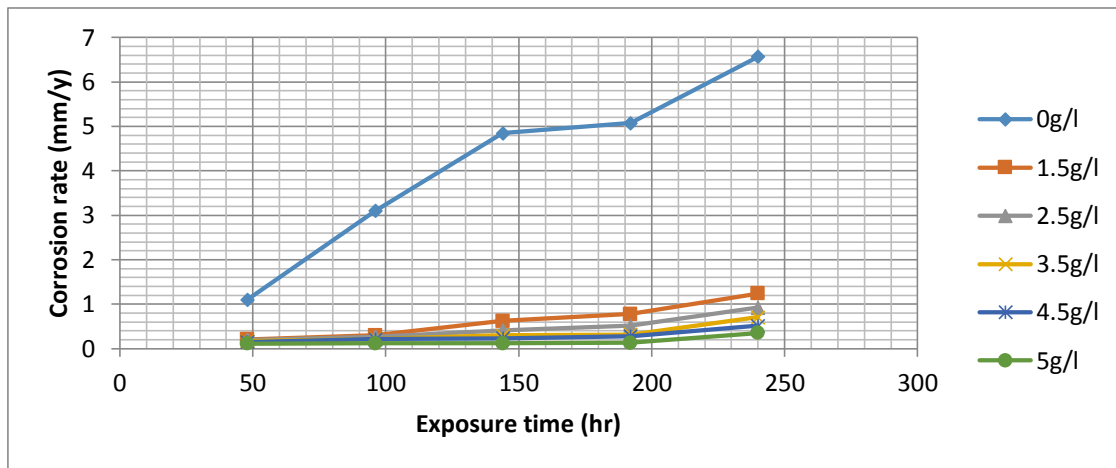

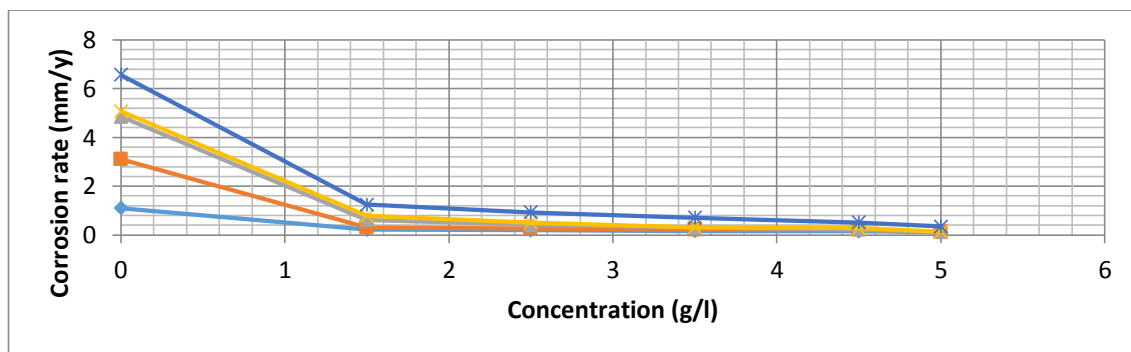*Figure 2: Variation of corrosion rate with exposure time*

Figure 3: Variation of corrosion rate with concentration

Surface coverage θ, of inhibitor are also very useful while explaining the adsorption characteristics. The adsorption of an inhibitor species at a metal/solution interface can be expressed as a place exchanger process between the inhibitor molecules in the aqueous solution and the water molecule on the metallic surface. The interaction between them can be described by adsorption isotherms; the simplest of which is the Langmuir isotherm. It is based on the assumption that all adsorption sites are equivalent and that particle binding occurs independently from nearby sites being occupied or not (Satapathy et al., 2009). The best correlation was obtained for the plot of C/ θ vs C as shown in Figure 4 with slopes around unity, thus suggesting that the adsorption of *hyptis suaceolen L poit* extract on Mild steel surface obeyed Langmuir adsorption isotherm.

The plot of $\frac{c}{\theta}$ against C was linear indicating that the adsorption of the inhibitor on the surface of mild steel is consistent with Langmuir isotherm. It was based on the assumption that the adsorbed molecule decreases the surface area available for the corrosion reactions to occur. The correlation coefficient (R^2) obtained shows that Langmuir isotherm fitted into the experiment with R^2 value of 0.9725 and this value was similar with values of 0.9424 - 0.9934 reported by Alaneme et al (2015).
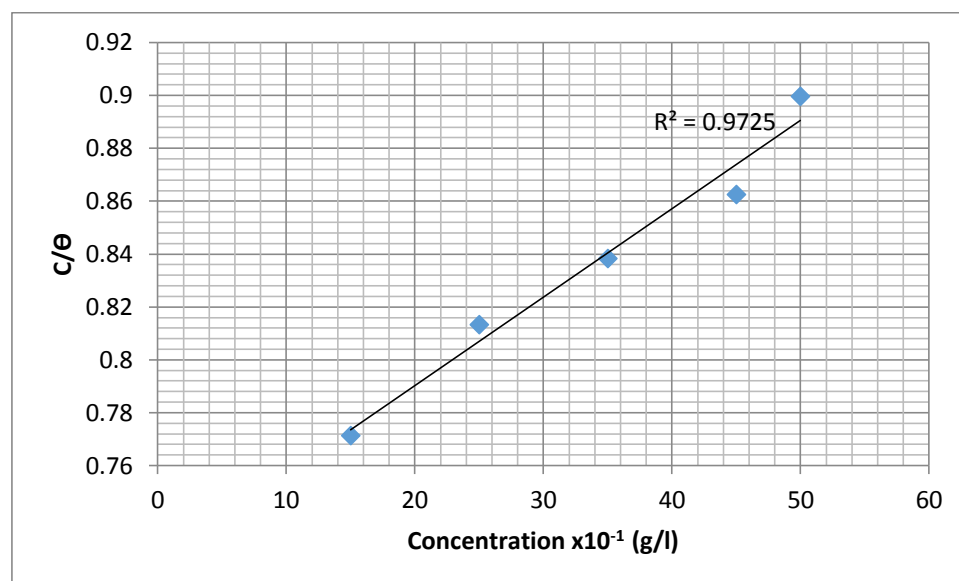
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Figure 4. Langmuir adsorption plots of mild steel immersed in 1 M H_2SO_4 HSLP Extract

3.4 Electrochemical measurements

Figure 5 shows Potentiodynamic anodic and cathodic Tafel polarization curves for mild steel specimens in 1M H_2SO_4 solution in the presence and absence of different concentrations of HSLP extract. The respective electrochemical or kinetic parameters including corrosion current density (Icorr), corrosion potential (Ecorr), cathodic and anodic Tafel slopes (βc, βa) and inhibition efficiency (IE %) are also presented in Table 4. It is clear from the table that addition of HSLP extract decreases corrosion current density. Corrosion current density (i_{corr}) decreases considerably with increasing concentration of extract. This moves the corrosion potential to positive values and βa changes in the presence of extract indicating that inhibitor molecules were pore adsorbed on the anodic sites resulting in an inhibition of the anodic reactions for mild steel in 1M H_2SO_4 solution. It is also clear that the inhibition efficiency of the extract increases with inhibitor concentration. This indicates clearly that HSLP extract acts as a good inhibitor for the corrosion of mild steel in H_2SO_4 media. It is also clear from the table that the presence of HSLP extract the values of corrosion potential E_{corr} are nearly constant. Addition of HSLP extract at all concentrations investigated decreased the anodic and cathodic current densities resulting in significant decline in the i_{corr}. This clearly shows that hyptis suaveolens acted as a mixed type corrosion resistance as reported by Rosliza et al (2009).

A critical look at Figure 5 reveals that the addition of HSLP extract has an inhibitive effect on both anodic and cathodic parts of the polarization curves. The presence of inhibitor results in a marked shift in the cathodic and anodic branches of the polarization curves implying that addition of the inhibitor reduces the mild steel dissolution as well as retards the hydrogen evolution reaction (Salghi et al 2014). Besides, the parallel cathodic Tafel curves in Fig. 5 show that the hydrogen evolution was activation-controlled and the reduction mechanism was not affected by the presence of the inhibitor as reported in Znini et al (2012). In the anodic domain, the polarization curves of mild steel have shown that the addition of HSLP decreases the current density. This results suggest that the inhibitory action depends on the potential of inhibitor and a desorption process appears at high potential. It is obvious that desorption rate of the inhibitor is higher than its adsorption rate in this case as reported in Rosliza et al (2009). Therefor HSLP extract could be said to be a mixed type inhibitor for mild steel in 1 M H_2SO_4 solution.

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Table 4: The electrochemical parameters of mild steel at various concentrations of plant extract.

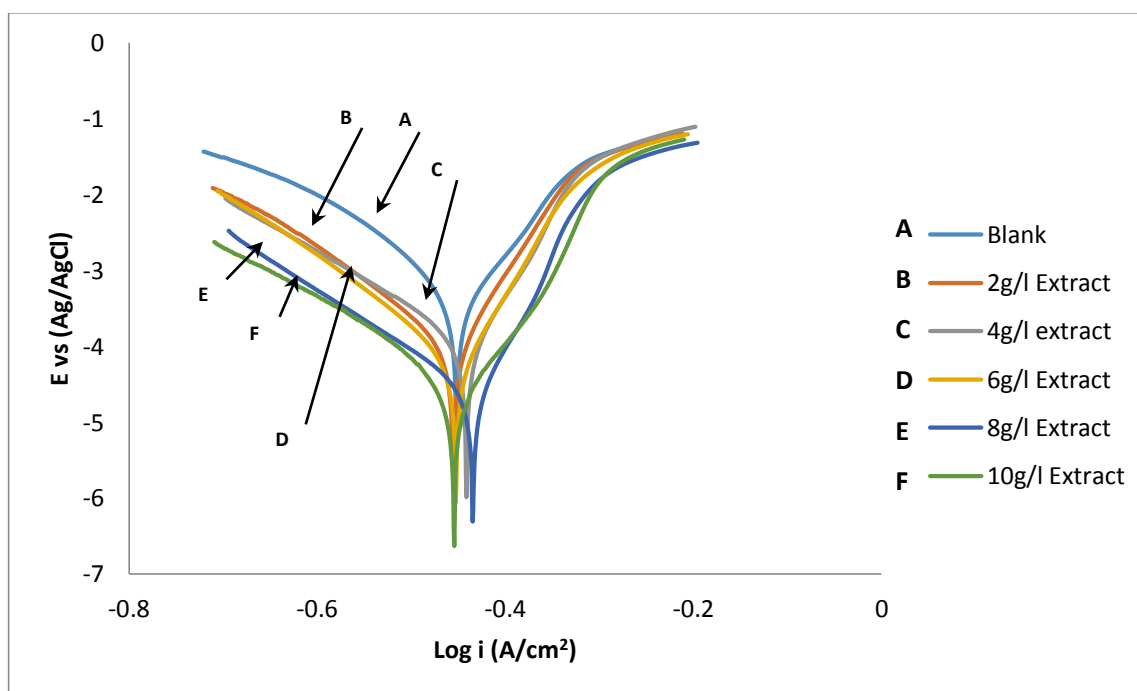| HSLP (g/l)	β_a (mV/decade)	β_c (mV/decade)	$-E_{corr}$ (mv/SCE)	I_{corr} ($\mu A/cm^2$)	Corrosion rate (mmpy)	PR	IE_{Icor}(%)
0	86.5	88.6	452	415	4.82	45.8	
2	106.3	58.7	451	100	1.16	163	75.9
4	109.2	61.3	441	105	1.22	162	74.6
6	90.7	60.1	451	59	0.67	264	85.8
8	138	62.7	433	31.1	0.36	602	92.5
10	87.5	82.6	454	24.2	0.28	762	94.2

Figure 5: Tafel polarization curves for the mild steel in the absence and presence of plant extract.

CONCLUSIONS

The following conclusions were drawn from the investigation of corrosion inhibition of mild steel in 1M H_2SO_4 by *hyptis suaveolens L poit* leaves extract:

1. That *hyptis suaveolens L poit* leaves extract contains compounds such as tanins, flavoids and limonene that are capable of inhibiting corrosion
2. The corrosion rate was found to increase with immersion time but decreased with increasing concentration of the plant extract for both weight loss and electrochemical methods.
3. *Hyptis suaveolens L poit* leaves extract is a mixed type inhibitor by preventing anodic metal dissolution and cathodic hydrogen evolution reaction
4. The adsorption of inhibitor molecules obeyed the Langmuir adsorption isotherms
5. *Hyptis suaveolens L poit* leaves extract can be used as an inhibitor for mild steel in acidic media.

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