

Inhibition Performance of Biochar extract of *Manihot esculenta* tuber peel for Corrosion Inhibition of mild steel in Hydrochloric acid solution

Olumide James Oluwole^{1*}, Donald Ngozi Anyiam^{1,2}, Yemisi Deborah Balogun¹, Oluwatobiloba Pelumi Oluwole³, Iyanuoluwa Esther Awomade⁴

¹Department of Industrial Chemistry, Faculty of Physical Sciences, University of Ilorin, P.M.B1515, Ilorin, Kwara State, Nigeria

²Department of Pharmaceutical Technology, School of Science and Technology, Moshood Abiola Polytechnic, P.M.B 2210, Abeokuta, Ogun State, Nigeria

³Department of Environmental Sciences, COMSATS University Islamabad, Abbottabad Campus, 22060, KPK, Pakistan.

⁴Department of Environmental Management and Toxicology, Federal University of Agriculture, P.M.B 2240, Abeokuta, Ogun State, Nigeria.

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Abstract: The weight loss (gravimetric method) and potentiodynamic polarization (electrochemical method) techniques were used to measure the corrosion rates of mild steel in 1 M HCl in the presence of char extract made from *Manihot esculenta* peel (MECE) as an inhibitor. The outcome demonstrated that extract has inhibitory effects at all temperatures. Adsorption is the mechanism of inhibition, and the Langmuir isotherm best fits the data. Potentiodynamic polarization and electrochemical impedance were used to calculate the activation energies, enthalpies, entropies, and entropies of the dissolution process as well as the free energies for the adsorption process. Important data regarding the inhibitory behavior of the char extract of *Manihot esculenta* peel was gathered using the basic thermodynamic functions.

Keywords: *Manihot esculenta* peel Biochar Extract, Langmuir isotherm, Tafel plot, Corrosion.

1. INTRODUCTION

The expense of corrosion and its economic influence is estimated to be 2.5 trillion US dollars, which is equivalent to 3.4 % of the global Gross Domestic Product (GDP). By using available corrosion mitigation practices, it is estimated that savings of between 15 to 35 % of the cost of corrosion could be actualized; i.e., between 375 to 875 billion dollars can be recovered annually on a global basis. One of the methods of reducing its adverse effect is the use of corrosion green inhibitors which contains phytochemicals such as flavonoids, terpenoids etc [1].

Researchers have long recognized the corrosion-inhibiting properties of organic molecules (phytochemicals) including heteroatoms with high electron densities, such as N, S and O. But because the majority of synthetic organic inhibitors are hazardous, they pose a number of risks when released into different water outlets like streams and the like. This is currently a worry for the majority of chemical suppliers and operators who must adhere to the expanding rules and regulations that decrease the impact of the chemicals on nature, humans, aquatic life, and other animals sharing our planet. This has led to research and an increase in demand for natural products, which would have a higher likelihood of being nontoxic and environmentally friendly [2-8].

Peel from cassava tubers, a waste product has long been fed to livestock (ruminant animals) in most regions of Nigeria and other parts of West Africa. Cassava peels often contain a higher proportion of cyanogenic glucosides than the pulp, which makes them unsuitable for use in animal feed [8,9]. However, it is deemed safe to use this agricultural waste as a preferred adsorbent since the peel also includes cyanide detoxifying enzymes (cyanoalanine synthase) that effectively keep cyanide at a safe concentration. Additionally, it contains polysaccharides like starch and holocellulose, which are sugars in the form of sugars. Pectin, cellulose, and amino acids are all rich in carboxyl, hydroxyl and amino groups. Researchers have discussed the use of green corrosion inhibitors, such as *Toona sinensis* leaf extract, Water hyacinth extract, and *Citrus sinensis* [9,10].

Although biochar, an intermediate product used in the manufacturing of activated carbon, is frequently employed in the treatment of water, the control of soil acidity pH, and other applications, its potential for the prevention of corrosion has not yet been fully realized.

In this research investigation, weight loss and electrochemical method measurements were used to examine *Manihot esculenta* biochar extract's potential as a natural inhibitor to stop the corrosion of mild steel in 1 M HCl.

2. MATERIALS AND METHOD

Materials

Chemical Reagents

1.0 M HCl (35% AR Grade), methanol, acetone, distilled water.

Equipments

Muffle Furnace (Lenton Thermal), Oven (Precision 16EG), Microbalance (Mettler Toledo, AL 204), Sieve mesh (150µm), Elemental Analyzer CHNO-S (Thermo Finnigan FlashEA 1220 Series) were used.

Methods

Preparation of Extract

Cassava (*Manihot esculenta*) tuber peel changed into accrued form was obtained from Garri Village in Tanke, Ilorin, Nigeria. The peel was sundried, grinded and sieved. 200 g of the sieved sample turned into heated sample in a muffle furnace set at 350⁰ C in which gentle circulation of N₂ gas passes through it at the rate of 500mL/min to supply *Manihot esculenta* peel char. The extract turned into organized whilst 10 g of the char that is the intermediate of activated carbon was refluxed for 2 hours with the aid of a means of electrical heating changed into refluxed while methanol was used as solvent. The char extract acquired is used to prepare inhibitor concentration within the range 20 – 200 ppm used in weight-loss method and electrochemical study [11].

Preparation of Mild Steel Specimen

The rectangular specimen of 5 x 3 x 0.16 cm and surface area of 1 cm² used for weight loss and electrochemical measurements had the following compositions: 0.04 wt.% (P), 0.33 wt.% (Si), 0.02 wt.% (Al), 0.10 wt.% (Mn), 0.30 wt.% (C), 0.06 wt.% (S) and the remainder iron (Fe). The mild steel test coupons were mechanically polished using emery paper, cleaned with acetone, washed with distilled water and finally dried in dry air before every experiment [12].

Preparation of Test Solution

The test solutions were prepared by the dilution of analytical grade 37 % HCl with distilled water. The dilution was done using the formula below

$$M_1V_1 = M_2V_2$$

1

Where M₁ and M₂ are the initial and final concentrations of the HCl solution and V₁ and V₂ are the initial and final volumes of HCl solution respectively [12].

Gravimetric method

The weight-loss measurements have been carried out at particular time durations of 24 h at 303K the usage of analytical stability. Each run become done in a beaker containing 100ml of 1 M HCl. A clean weighed square metal electrode (3 X 5 X 0.16 cm) was absolutely immersed at inclined role inside the vessel. After the exposure time, the electrode was withdrawn, rinsed with doubly distilled water, washed with acetone, dried and washed [10-12].

From the initial weight (W_0) and final weights (W_1) of the specimens, the weight loss (W) was calculated using the relation $W = W_0 - W_1$

The inhibition efficiency was calculated using the equation:

$$\text{Inhibition efficiency (I.E\%)} = \frac{W_0 - W_1}{W_0} \times 100 \quad 2$$

Where W_0 is the weight loss without inhibitor and W_1 is the weight loss in the presence of inhibitor.

Proximate Analysis of Char

By heating the sample in an oven (Precision 16EG) under predetermined conditions using ASTM method, proximate analysis of *Manihot esculenta* peel biochar was performed to assess its physicochemical parameters, such as moisture content, ash content, volatile matter, and fixed carbon content [13, 14].

Moisture Content Determination

Manihot esculenta peel char was correctly weighed at 5 g before and after being heated in an oven at 180 °C using an analytical microbalance (Mettler Toledo, AL 204). The equation below was used to calculate the biochar's moisture content: [13-15]

$$\% \text{ Moisture Content} = \frac{W_0 - W_1}{W_0} \times 100 \quad (2)$$

Where W_0 is the weight before heating in the oven and W_1 is the weight after heating in oven

Fixed Carbon Content Determination

The fixed carbon content was calculated with equation below:

$$\% \text{ Carbon content of mixed biochar} = 100 - (\% \text{ moisture content} + \% \text{ ash content} + \% \text{ volatile matter}) \quad (3)$$

Elemental Analysis of Biochar

Ultimate analysis of *Manihot esculenta* peel char to determine its elemental composition was performed using Elemental Analyzer CHNO-S (Thermo Finnigan FlashEA 1220 Series).

Electrochemical Measurement

A 3 electrodes cellular gadget containing working electrode (moderate metallic coupon) of a 1cm^2 exposure area, saturated calomel electrode as a reference and a platinum electrode as auxiliary were used. Potentiodynamic polarimetry experiments had been completed at room temperature ($25 \pm 2^\circ\text{C}$) in 1M HCl electrolyte solution with and without extract of inhibitor using VERSASTAT 400 complete DC voltammetry. The working electrode had been allowed to corrode freely and its open circuit polarimetry (OCP) was recorded as a function of time throughout 3h, the time necessary to attain a quasi-stationary value for the open-circuit potential. The steady-state OCP corresponds to the corrosion potential (E_{corr}) of the working electrode. The anodic and cathodic polarization curves have been recorded by means of a consistent sweep rate of 20mVmin^{-1} . The Tafel data information had been analyzed with the usage of graphing and analyzing impedance software, version EC-LabV9.98 [12,13].

The inhibition efficiency from the electrochemical method was calculated using;

$$\text{Inhibition efficiency (I.E\%)} = \left(1 - \frac{E_{\text{corr}'}}{E_{\text{corr}}}\right) \times 100 \quad 3$$

$$\text{Inhibition efficiency (I.E\%)} = \left(1 - \frac{E_{\text{corr}'}}{E_{\text{corr}}}\right) \times 100\% \frac{I_{\text{corr}'}}{I_{\text{corr}}} \times 100 \quad 4$$

Where E'_{corr} = corrosion electropotential with inhibitor

E_{corr} = corrosion electropotential without inhibitor

I'_{corr} = corrosion current density with inhibitor

I_{corr} = corrosion current density without inhibitor

3. RESULTS AND DISCUSSION

Physicochemical properties of Biochar

The result of the determination of properties such as moisture content, volatile matter, ash content and carbon content of *Manihot esculenta* Char extract (MECE) is shown in the table 1 and 2 below.

Table 1: Result of Proximate Analysis of *Manihot esculenta* peel Biochar

Properties/Biochar	Moisture Content (%)	Volatile Matter (%)	Ash content (%)	Carbon Content (%)
<i>Manihot esculenta</i> Biochar	10.90	41.23	9.53	38.34

From the table above, the carbon content of the biochar was 38.34 % indicating high maturity of the biochar while the volatile matter was 41.23% which implies that the biochar contains various compounds which contains heteroatoms that enable it adhere to the surface of the mild steel thereby inhibiting the coupon from corroding [14-16].

Table 2: Result of Ultimate Analysis of *Manihot esculenta* peel Biochar

Sample/Elemental content (%)	Carbon (C)	Hydrogen	Nitrogen	Oxygen	Sulphur
<i>Manihot esculenta</i> Biochar	53.42	6.24	2.58	35.46	0.09

The ultimate analysis's findings showed that char contains 53.42% carbon, 6.24% hydrogen, 2.58% nitrogen, 35.46% oxygen, and 0.09% sulphur, confirming that it contains heteroatoms that qualify it as an anti-corrosion agent which can prevent mild steel from corroding. [14-17].

Effect of Concentration

The inhibition efficiency response of corrosion studies based on *Manihot esculenta* char extract (MECE) toward mild steel in an acidic medium using weight-loss method is shown in Figure 1 above. The inhibition efficiency increases alongside the inhibitor concentration resulting from adsorption of char extract molecules on the mild steel with optimum inhibition efficiency at 84% which is illustrated in the figure below [18-20].

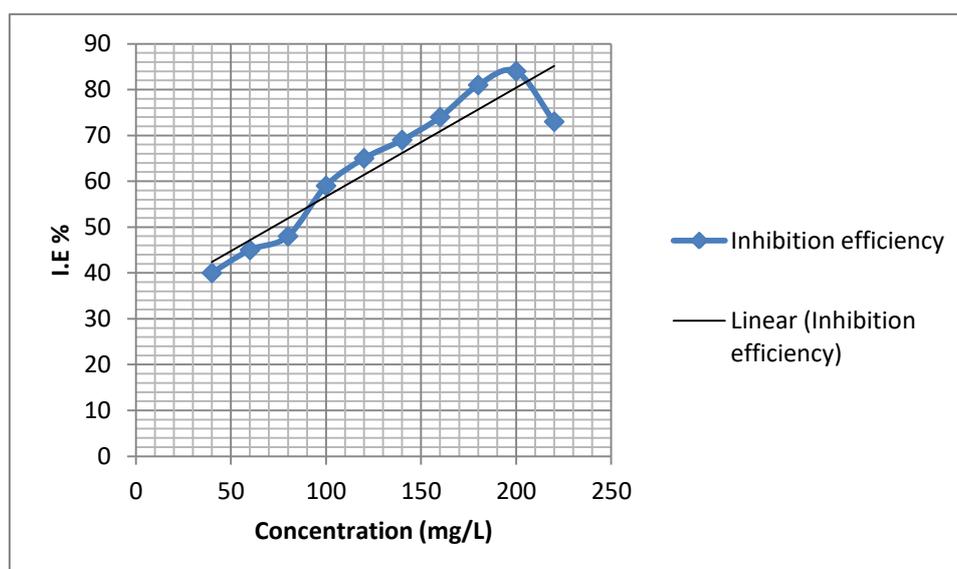


Figure 1: A plot of Inhibition efficiency with concentration for MECE adsorption on mild steel at room temperature

Table 1: Weight loss data obtained from the immersion time of mild steel in 1M HCl in several concentrations of MECE inhibitors for 24h at room temperatures (303K)

Inhibitor	Conc (mg/L)	W.L (mg)	C.R (mgcm ⁻² h ⁻¹)	Surface Coverage (Θ)	I.E%
<i>Manihot esculenta</i> char extract (MECE)	0	756.3	2.1009	-	-
	20	490.0	1.3611	0.3521	35.21
	40	458.2	1.2728	0.3942	39.42
	60	437.8	1.2161	0.4211	42.11
	80	375.4	1.0426	0.4963	49.63
	100	351.4	0.9761	0.5354	53.54
	120	311.8	0.8661	0.5877	58.77
	140	286.4	0.7956	0.6213	62.13
	160	216.2	0.6006	0.7142	71.42
	180	160.0	0.4444	0.7884	78.84
	200	111.7	0.3103	0.8523	85.23

WL= Weight Loss I.E = Inhibition Efficiency, C.R = Corrosion rate, MECE = *Manihot esculenta* Char Extract

The impact of concentration of inhibitor on inhibition efficiency and corrosion rate were examined using inhibitor concentration range of 20mg/L to 200mg/L, for MECE inhibitors for 24 h at 303K. The results were portrayed in Table 1 above.

The inhibition efficiency escalated progressively from 35.32 to 85.23% for MECE as concentration increased from 0mg/L to 200mg/L. The progressive increment of inhibition efficiency with inhibitor concentration is believed to be attributed to the adsorption of *Manihot esculenta* char extracts inhibitor molecules on the metal/solution interface which increased with increased inhibitor concentration [20-24].

As inhibitor concentration increased, the fraction of the mild steel surface obscured by the inhibitor constituents increased thereby forming a barrier against the interaction of the corrosive 1M HCl media with iron surface [22-25].

Also, the corrosion rate of the mild steel in 1M HCl decreased with increase in the concentration of the inhibitors at the studied immersion time of 24 h. The corrosion rate decreased progressively from 2.1009 mgcm⁻²h⁻¹ to 0.3103 mgcm⁻²h⁻¹ for MECE. The decrease in corrosion rate with increase in inhibitor concentration is also attributable to increase in the number of adsorption of the inhibitor components on the surface of mild steel which makes a hedge for mass transfer and prevent further corrosion [8, 20, 25-26].

Adsorption Isotherm

The performance of *Manihot esculenta* char extract MECE as a successful inhibitor was mainly due its ability to adsorb on the surface of mild steel. The mechanism of corrosion inhibition has been determined depending on its ability. The data obtained from weight-loss measurements best fit in to adsorption isotherm with its correlation coefficient close to 1 which is shown in the equation below [8,20, 25-26].

$$\frac{C}{\theta} = \frac{1}{K_{ads}} + C \quad 5$$

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

A plot of $\frac{C}{\theta}$ vs C gives a straight line. Thus, the plots $\frac{C}{\theta}$ vs C with yielded straight lines at several temperatures. The K_{ads} obtained from the Langmuir plot in figure 2 below is used to calculate free energy of adsorption (ΔG_{ads}^0) adopting the equation below [22-27].

$$\Delta G_{ads}^0 = -RT \ln(55.5 K_{ads}) \quad 6$$

Where 55.5 is the molar concentration of water in molL⁻¹, R is molar gas constant and T is the absolute temperature.

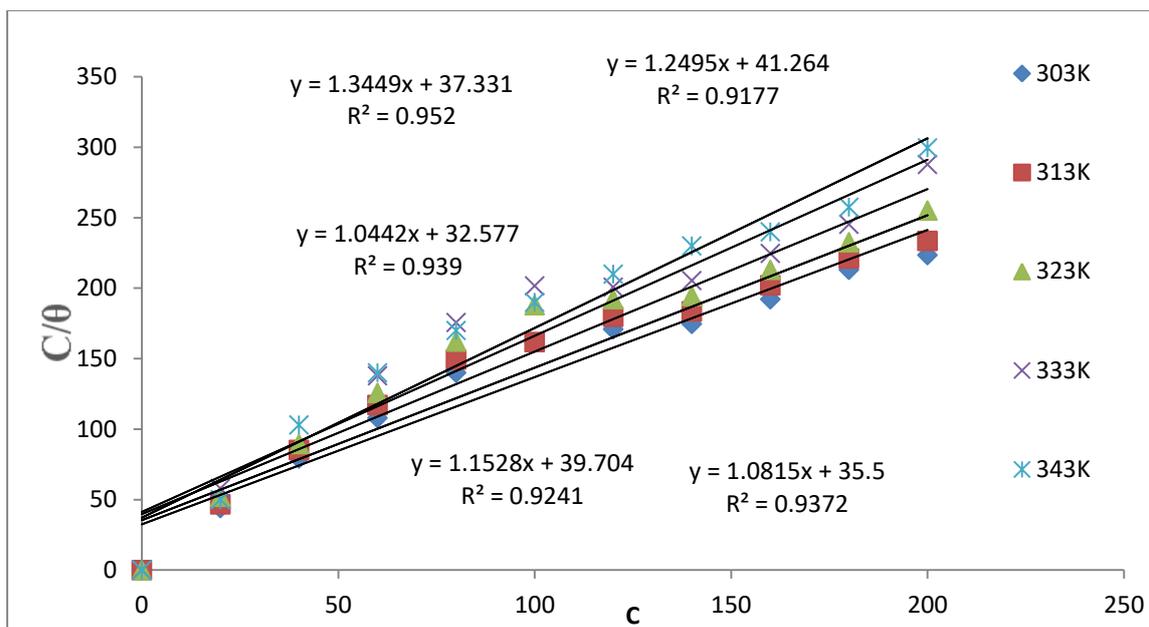


Figure 2: Langmuir Isotherm of MECE adsorption onto mild steel in 1M HCl at various temperatures.

The adsorption equilibrium constant (K_{ads}) and adsorption thermodynamic parameters such as free energy (ΔG_{ads}) obtained from the plots in figure 2 and equation 2 respectively were summarized in table 2.

Table 2: Langmuir isotherm and Free Energy change parameters for MECE on Mild steel in 1M HCl obtained at different temperatures.

Temperature K	R^2	K_{ads} (L/mg)	Slope(m)	ΔG_{ads} (KJ/mol)
303	0.9390	0.031	0.9763	-13.67
313	0.9372	0.028	1.0043	-11.47
323	0.9241	0.025	0.9823	-8.79
333	0.9177	0.024	1.0003	-7.94
343	0.9520	0.021	0.9991	-0.44

From the data in Table 2 above, K_{ads} value decrease with increase in temperature from 303K to 343K, ΔG_{ads} values were beneath -20KJ/mol which indicated that the interaction between the molecules of the char extract and the mild steel was physical adsorption (physisorption). The negative values of ΔG_{ads} showed that the adsorption process was feasible and it correlated with what was reported elsewhere [22-29].

Adsorption Thermodynamic parameters

The adsorption equilibrium constant values (K_{ads}) at different temperatures calculated from the Langmuir plots using equation (4) were used to calculate standard enthalpy of adsorption (ΔH_{ads}^0) and entropy of adsorption (ΔS_{ads}^0) using the integrated Van't Hoff equation: [9,10,23,27].

$$\ln K_{ads} = \frac{-\Delta H_{ads}}{RT} + \frac{\Delta S_{ads}}{R} + \ln \frac{1}{55.5}$$

4

A plot of $\ln K_{ads}$ against $1/T$ gives a straight line with slope equals to $-\Delta H_{ads}^0/R$ and intercept of $(\Delta S_{ads}^0/R + \ln 1/55.5)$ was obtained in figure 3 below.

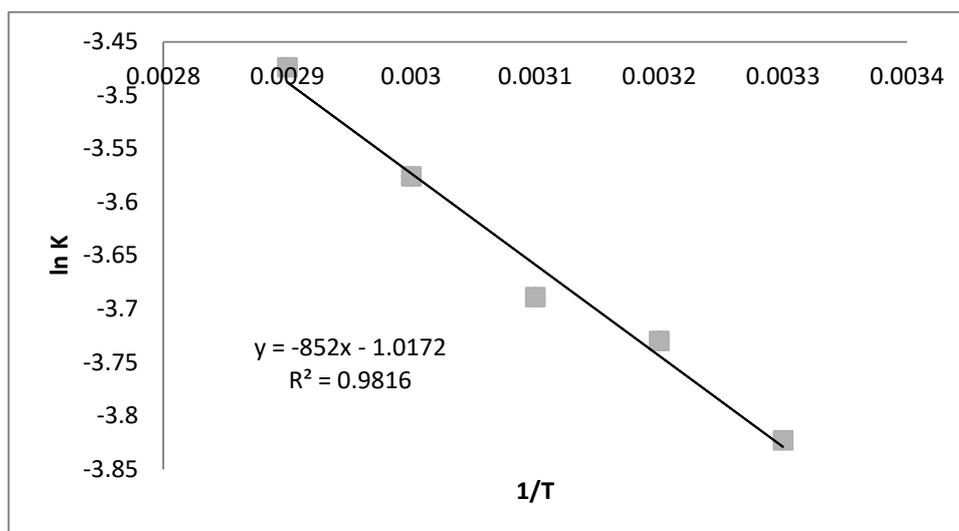


Figure 3: Van't Hoff plot for MECE adsorption onto mild steel in 1M HCl

The slope obtained was used to determine the value of enthalpy of adsorption (ΔH_{ads}) while the intercept obtained from $1/T$ axis is used to determine entropy of adsorption (ΔS_{ads}) while the correlation coefficient for the extracts is 0.9816. The enthalpy of adsorption (ΔH_{ads}) for MECE was 7.08 KJmol^{-1} which shows that the adsorption process on the mild steel in the presence of the inhibitors is endothermic while the entropy of adsorption for MECE was $24.94 \text{ Jmol}^{-1}\text{K}^{-1}$ which implies that the adsorption process is accompanied by increase in randomness of extract molecule on the interface of the coupon and the adsorption process of MECE onto the specimen surface was via chemisorption as reported elsewhere [7,10,13].

Electrochemical Analysis of Mild steel

The corrosion rates of mild steel in aggressive solutions can be obtained using potentiodynamic polarisation curve was thus applied to examine the effect of char extract concentration. The percentages of inhibition efficiency and values of associated electrochemical parameters were highlighted in the Table below. Investigation of the obtained data disclosed that the I_{corr} values decrease steadily in the presence of *Manihot esculenta* char extract (MECE) with increasing inhibitor concentration. The E_{corr} values tilted to more positive potentials in the presence of inhibitor concentration [8,12,24-29].

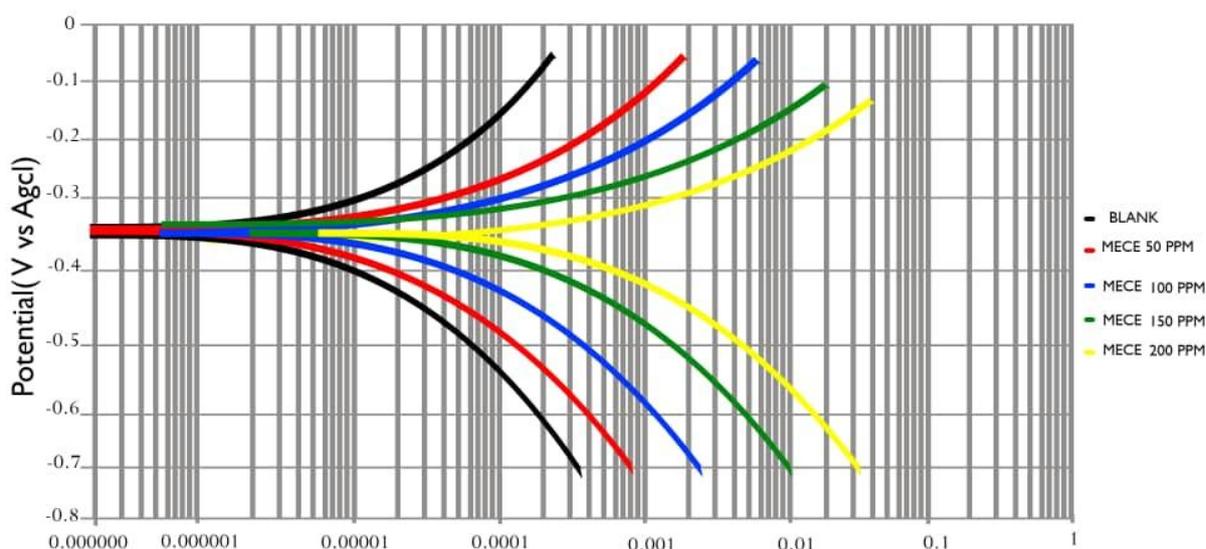


Figure 4: Potentiodynamic polarimetry sweep curve of mild steel immersed in 1M HCl in the presence and absence of *Manihot esculenta* char extract (MECE) at room temperature

The inhibition of mild steel cassava peel char extract on mild steel in 1M HCl was monitored by measuring the cathodic and anodic behavior change of coupon which is equivalent to hydrogen reduction and metal oxidation respectively and the sweep is conducted at the rate of 2mV/s [26,28].

Table 3: Electrochemical parameters obtained from the electrochemical studies of *Manihot esculenta* char extracts (MECE) at 303K.

Conc(mg/L)	$E_{corr}(mv)$	$I_{corr}(\mu A/cm^2)$	β_a	β_c	C.R (mmpy)	I.E %
Blank	-472.78	20.349	155.463	268.462	14.339	NA
50	-413.05	20.296	140.868	250.288	10.626	25.89
100	-480.05	20.264	90.314	179.282	8.362	41.54
150	-468.07	8.813	97.408	172.158	6.210	56.69
200	-457.27	4.029	67.958	164.084	2.839	80.20

Figure 4 and table 3 showed that the cathodic and anodic reactions are reduced as the concentration of the inhibitor increases by decreasing the polarization of the current densities of both side of the curve. Corrosion rate decreased steadily from 14.339 mmpy to 2.839 mmpy as the concentration of the MECE inhibitor increased from 50 mg/L to 200 mg/L while the inhibition efficiency increased from 25.89% to 80.20%. The cathodic and the anodic slopes were altered showing reduction in metal dissolution and decrease in Hydrogen reaction dissolution confirmed that MECE is a mixed inhibitor [27,30-34].

4. CONCLUSION

The inhibitory efficacy of mild steel in 1M HCl rose as the concentration of *Manihot esculenta* biochar extract increased to reach a maximum steady state concentration, as demonstrated by gravimetric method and electrochemical techniques. With the aforementioned procedures, higher inhibition efficiencies were achieved under test conditions and while employing special treat rates. The extract adsorbs to the steel in an acidic media according to the Langmuir adsorption isotherm model, and the reaction is spontaneous based on the negative value of the adsorption free energy. The entropy and enthalpy of adsorption demonstrate a rise in endothermic randomness of extract molecules on mild steel. The physico-chemical and comprehensive examination of *Manihot esculenta* peel biochar reveals that it contains heteroatoms in the form of volatile substances, which are in charge of the extracts' suppression of mild steel in acidic environments. This demonstrates that biochar, despite its many applications, has anti-corrosion properties.

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