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EVALUATION OF INHIBITIVE ABILITY OF CHROMOLAENA ODORATA (CHRISTMAS BUSH) ON MILD STEEL AQUEOUS ACID SOLUTION

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ABSTRACT

The major problem militating against engineering materials is corrosion, which in turn has slowed down industrial growth and the major solution proffered to this menace is the use of inhibitors. In this work, Chromolaena Odorata (Christmas Bush) was used as natural inhibitor to prevent corrosion of mild steel in dilute hydrochloric acid media using electrochemical method (ECM) and Electrochemical Impedance Spectroscopy (EIS). Phytochemical screening was carried out on the Chromolaena Odorata (Christmas Bush). The phytochemical analysis shown that the inhibitor contains the following constituents; steroids, tannins and flavonoids which assisted in the inhibition efficiency of the inhibitors. From the electrochemical analysis, the results showed that corrosion rate increases with higher concentration of inhibitor. Which shows that Christmas bush is a good inhibitor. The electrochemical impedance spectroscopy results showed that both anodic and cathodic reactions are inhibited in the presence of Christmas bush extract on mild steel. The value obtained showed that the corrosion rate did not necessarily decrease with increase in concentration of inhibitors, but a 4_{ml} reveals the highest corrosion resistance thus Christmas bush is a viable inhibitor that can be used to mitigate corrosion of mild steel in dilute hydrochloric acid.

KEYWORDS: Corrosion; Inhibition; Inhibitors; Electrochemical analysis; Concentration

INTRODUCTION

Inhibition is the ability of any liquid to mitigate corrosion. Corrosion control of metals and alloys is an expensive process and industries spend huge amounts to control this problem. It is estimated that corrosion cost in the developed countries such as the U.S. and European Union is about 3–5% of their gross national product (Bhaskaran et al., 2005). By definition, a corrosion inhibitor is a chemical or natural substance that, when added in small concentration to an environment, decreases the corrosion rate (Fiaud, 1994).

There are two types of corrosion inhibitor; artificial and natural mean. In an artificial corrosion inhibition, chemicals are employed to mitigate corrosion of metals but mostly with antecedent shortcoming such as toxic pollution which is very harmful to human. In natural corrosion inhibition, certain natural occurring plants leaves are employed to mitigate corrosion of metals in a corrosive media. Some selected leaves when pulverized and extracted mitigate corrosion of metals in acidic media. Researchers are still researching some leaves to ascertain their inhibitive value and efficiency.

Corrosion damage can be prevented by using various methods such as upgrading materials, blending of production fluids, process control and chemical inhibition (Dudukcu et al., 2004; Galal et al., 2005). Among these methods, the use of corrosion inhibitors (DeSouza & Spinelli, 2009; Raja & Sethuraman, 2008) is one the method to prevent degradation of metal surfaces in corrosive environment. Corrosion inhibitors are chemicals either synthetic or natural which, when added in small amounts to an environment, decrease the rate of attack by the environment on metals. A number of synthetic compounds can be used (Bentiss et al., 2007; Dudukcu et al., 2004; Galal et al., 2005).

Cookey et al., (2018) carried out research on evaluation of the corrosion inhibition potentials of green-tip forest lily (Clivia nobilis) leaves extract on mild steel in acid media. In the study, it was stated that corrosion tests were performed on mild steel to evaluate the effect of concentration of inhibitor, varying immersion period and temperature on the corrosion inhibition properties of Clivia nobilis leaves extract. It was done in H_2SO_4 and HCl acid solutions using weight loss and gasometric methods. From the weight loss results, corrosion rate and degree of surface coverage were evaluated as a function of inhibitor concentration at various immersion times. The result of the study showed that Clivia nobilis leaves extract has corrosion inhibition ability on mild steel in H_2SO_4 and HCl solutions, with inhibition efficiency greater in HCl than in H_2SO_4 solution.

Onwumelu et al., (2018) worked on Inhibition of Mild Steel Corrosion in HCl Solution by Gongronemalatifolium Methanol Extract. Corrosion inhibition effect of methanol extract of Gongronemalatifoliumon mild steel in HCl solution was studied using gasometric methods at 303K, 313K and 323K. The inhibition efficiency was found to increase as the concentration of extracts increased from 0.1%w/v to 0.5%w/v and decreased with increase in temperature. The maximum efficiency of inhibition was found to be 77.17% at 303K. Values of activation energy for the inhibited system were greater than the values obtained for the uninhibited system.

Arockiasamy et al., (2014) carried out research on evaluation of Corrosion Inhibition of Mild Steel in 1 M Hydrochloric Acid Solution by Mollugo cerviana. The inhibiting effect of methanolic extract of Mollugo cerviana plant on the corrosion of mild steel in 1 M HCl solution has been investigated by different techniques like potentiodynamic polarization, electrochemical impedance spectroscopy, and weight loss methods for five different concentrations of plant extract ranging from 25 to 1000 mg/L. The results indicated that the corrosion inhibition efficiency increased on increasing plant extract concentration till 500 mg/L and decreased on further increasing concentration.

MATERIALS AND METHOD

Materials

The materials used for this research were mild steel, Chromolaena Odorata (Christmas bush), ethanol, HCL and equipment used were; Chemical weighing balance, Filter medium, A calibrated cylinder, Pulverizer (electric blender), Bench Vice, and Hack saw.

Preparation of Coupon

The coupon which was mild steel was cut into 20 mm by 10 mm to fit into electrochemical machines. It was rubbed with emery paper, washed in distilled water and rinsed in acetone and allowed to dry in air to ensure proper surface cleanliness of the coupon before immersion. It was attached to the naked part of small wires with the aid of aluminium foil and placed inside the moulding cup. This was allowed the coupon to pass through electrochemical method which was based on Faraday second law of electrolysis which states that “when the same quantity of electricity is passing through different electrolytes, the masses of different ions liberated at the electrodes are directly proportional to their chemical equivalent''. The coupon was tested using Electrochemical Impedance Spectroscopy Method (EIS) and Phytochemical Method which was based on weight loss due to coupon degradation in the said medium.

Plant and Media Preparation

The leaf was washed, and sun dried before pulverization. After which the pulverized Christmas bush was measured using wind weight balance. 1gram of the pulverized leaf was soaked in $10CM^3$ of ethanol in a conical flask. The resulting solution was left for 72 hours, which was later filtered. The filtrate was subjected to evaporation at room temperature to obtain the extract. 1M of concentrated HCL was prepared with distilled water out of which the various HCL media for the coupons was prepared.

***Electrochemical Method and Electrochemical Impendence Spectroscopy Method* (EIS)**

The open-circuit potential (OCP) experiment was performed using an electrochemical machine whose principles of operation were based on second Faraday law of electrolysis. The results presented on the monitor of the equipment were tabulated to determine the inhibition of Christmas bush on mild steel in the aqueous acid medium, using electrochemical method. It has a scanning rate of 0.166mm/s, they have a set initial potential of -200mV to +200mV. The electrochemical instrument consisted of the platinum counter electrode kit, reference electrode (silver chloride, single JXN, pin connector, 14/20 adapter), 5 neck glass RDE cell of 150_{ml}, fixed shaft (non-rotating). E4TQ change RDE tip and E4TQ replacement holder. The set-up or the instrument contained the cell and the three electrodes, counter electrode was the one in charge of voltage measurement, while the reference electrode served as standard for measurement, it was the working electrode and the measurement based on the conductivity of the reference used. It can be silver, platinum or graphite. But the one used in this experiment was platinum. The specimen was connected to the working electrode and was separately dipped inside the solution in the beaker together with the counter and reference electrode before solidification. Two different electrochemical tests were conducted, Linear Polarization Resistance (LPR) and Electrochemical AC Impedance Spectroscopy (EIS). A linear polarization test was carried out by a scan from approximately -10mV to +10mV with respect to working rest potential. The cyclic seep polarization was taken into consideration the open circuit potentials of working electrode. EIS measurements were performed using AC signal amplitude of 20mV peak to peak in the frequency range of 0.1 Hz to 1k Hz. The open circuit potential, corrosion potential, corrosion current and the corrosion rate were displayed on the monitor. The procedure was performed on the blank (solution without inhibitor) and inhibited samples.

Phytochemical and Proximate Analysis

The leaves were air dried pulverized into powdery form. The powdery sample of the plant (Christmas bush) was stored in an air-tight container until ready for use. The Phytochemical screening and proximate analysis of the powdered leaves were done according to standard procedures.

Extraction and Fractionation

The powdered plant materials (750g) were extracted with methanol (4.25l) by maceration at room temperature for 7days. The extract was concentrated to dryness using a rotary evaporator at reduced pressure. The crude methanol extract (17.71g) was dissolved in aqueous methanol (4:1,

100_{ml}. The solution was extracted successively with n-hexane (600_{ml}), chloroform (600_{ml}) and ethyl acetate (500_{ml}). The various fractions were concentrated to dryness and stored in the refrigerator until use.

RESULTS AND DISCUSSION

Table 4.1 shows corrosion rate, corrosion potential, corrosion current, cathodic beta and anodic beta of the coupon for blank, 2_{ml}, 4_{ml}, 6_{ml}, 8_{ml}, and 10_{ml}.

Effect of Inhibitor Concentration on the Corrosion Rate on Mild Steel Using Electrochemical Method

Figure 4.1 shows the corrosion behaviour of mild steel in 0.5M HCL when protected with Christmas bush extracts. From the plot, it was observed that the corrosion rate reduced at the addition of 2_{ml}, the corrosion rate of 4_{ml}, 6_{ml} and 8_{ml} have higher corrosion rate than the blank. The addition of higher concentration of inhibitors did not correspond to reduction in corrosion rate, the shift of the curves of 4_{ml}, 6_{ml}, and 8_{ml} in the fig.4.1 moved to the right indicating higher current density so showing a higher corrosion rate and corrosion value. The sample in inhibited at 2_{ml} was found to be the only concentration of the extraction that inhibit thereby given the last corrosion rate and so the highest corrosion residence in the environment used. At 4_{ml} concentration shows the last corrosion rate with inhibited efficiency of 10%. The corrosion rate is 0.62278mmpy compared with the corrosion rate of blank that is 10.605mmpy. The corrosion rate is 1:3 of blank to 4_{ml} of the sample in 0.5M HCL without inhibitor. The current density of the blank is higher indicating a higher flow of electron and so higher corrosion rate. This agreed with previous work (Arockia et al., 2016; Babatunde et al., 2012; Olaseinde et al., 2014;). The graph has two branches in which the downward is the cathodic and the upward is the anodic. Where the two-curve meet is called the high curve which is the current density, at that point it is assume that the cathodic current is equal to the anodic current if the corrosion rate is high, the current density will be high using the principle of electrolysis (which is the second Faraday law of electrolysis).

Note: *The higher the current density the higher the corrosion rate. The lesser value of corrosion current density the lesser the corrosion rate. The higher the cathodic beta value, the lower the reduction of the hydrogen evolution in the reaction. The higher the anodic beta values the lower anodic dissolution. This shows that the corrosion rate is reduced.*

Effect of Inhibitor Concentration on the Corrosion Rate on Mild Steel Using Electrochemical Impedance Spectroscopy (EIS)

The superimposed electrochemical impedance spectroscopy polarization curves of the corrosion of mild steel as shown in Fig 4.2: The corrosion parameters such as corrosion potential, corrosion current for anodic and cathodic slopes are shown. Polarisation curves indicated that both anodic and cathodic reactions are inhibited in the presence of Christmas bush extract on mild steel. The values obtained shows that the corrosion rate did not necessarily decrease with increase in concentration on inhibitor but at 4_{ml} the highest corrosion resistance. The real impedance (Z_{re}) value of the control specimen is lower than the real impedance value of the inhibited specimen. This shows that the inhibitors inclusion reduces the corrosion rate of the environment on the mild steel. The higher the real impedance values the higher the inhibitor efficiency. In figure 4.3, as frequency increases the real impedance increases which means more resistance depicts beta corrosion. In Figure 4.4, as the frequency increase the phase of impedance (Z) also increases but at slow rate compare to fig 4.3: frequency with real impedance.

Table 4.2 shows the phytochemical analysis of the Christmas leaf constituents, it was discovered that the plant constituent has corrosion resistance of organic agents (saponins, tannins and flavonoids) which helps in the inhibition efficiency of the inhibitor which correlate to what others researchers discovered. (Helen et al., 2014; 2019). Tannins forms complex compounds with Fe (iii) on the metal surface while the presence of saponin and flavonoid gives high metal complex affinity that is responsible for good corrosion inhibitors in performance which is accordance to reseacher report (Sachin et al., 2009). The constituent mentioned above give strong adsorption molecules of the Christmas bush extract film on the surface of mild steel used.

CONCLUSIONS AND RECOMMENDATIONS

The research summary indicates how natural inhibitors can be used to mitigate corrosion in developing nation. Christmas bush as natural occurring plants and in abundance in Nigeria will play vital role in mitigating corrosion than the artificial means of preventing corrosion if employed. Artificial means of preventing corrosion have antecedent effect such as poisoning and huge cost. Green inhibitors as alternatives to the fossil origin toxic corrosion inhibitors is preferred and must be encouraged. Acid extract of Christmas bush leaf acts as good corrosion inhibitor for mild steel in HCL medium and inhibition efficiency increases with inhibitor concentration and maximum inhibitor efficiency was 80.61% at the inhibitor concentration 95ppm. Corrosion inhibition of the Christmas bush was as result of the leaf constituents being adsorbed on the mild steel surface. Polarizations studies shows that is a mixed type inhibiting both cathodic and anodic reactions. Cost effectiveness is a huge advantage of green inhibitors due to renewability of its resources.

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Table 4.1: Electrochemical Method

Name	CORROSION RATE (mmpy)	CORROSION POTENTIAL (mV)	CORROSION CURRENT (ųA)	CATHODIC BETA (mV)	ANODIC BETA (mV)
Blank	10.605	520.378	913.928	145.163	107.276
2ml	0.00046156	144.403	39.77	573.222	368.287
4ml	0.62278	443.517	53.671	302.424	208.859
6ml	0.29676	445.517	25.573	117.555	54.987
8ml	0.79001	485.193	68.083	119.562	84.74
10ml	0.35683	466.101	30.751	88.867	56.102

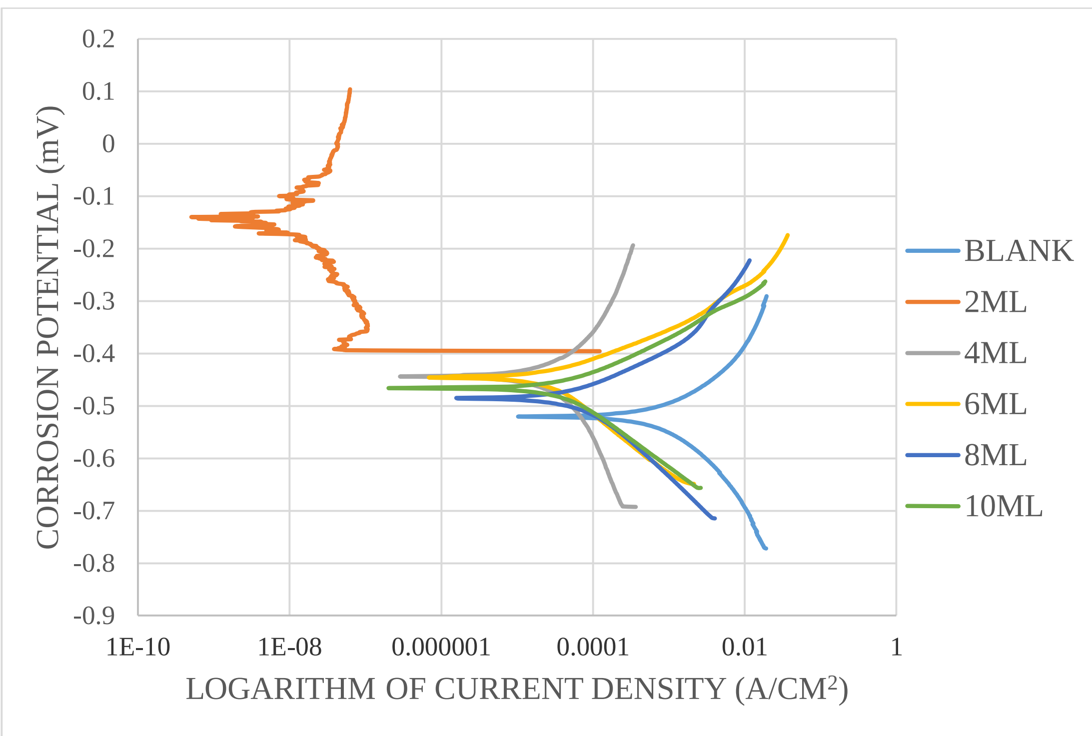

Figure 4.1. Graph of Corrosion Potential (mV) against Logarithm of Current Density (A/CM^2) on mild steel in aqueous acid solution on the extracted leaf of Chromolaena Odorata (Christmas Bush**)**

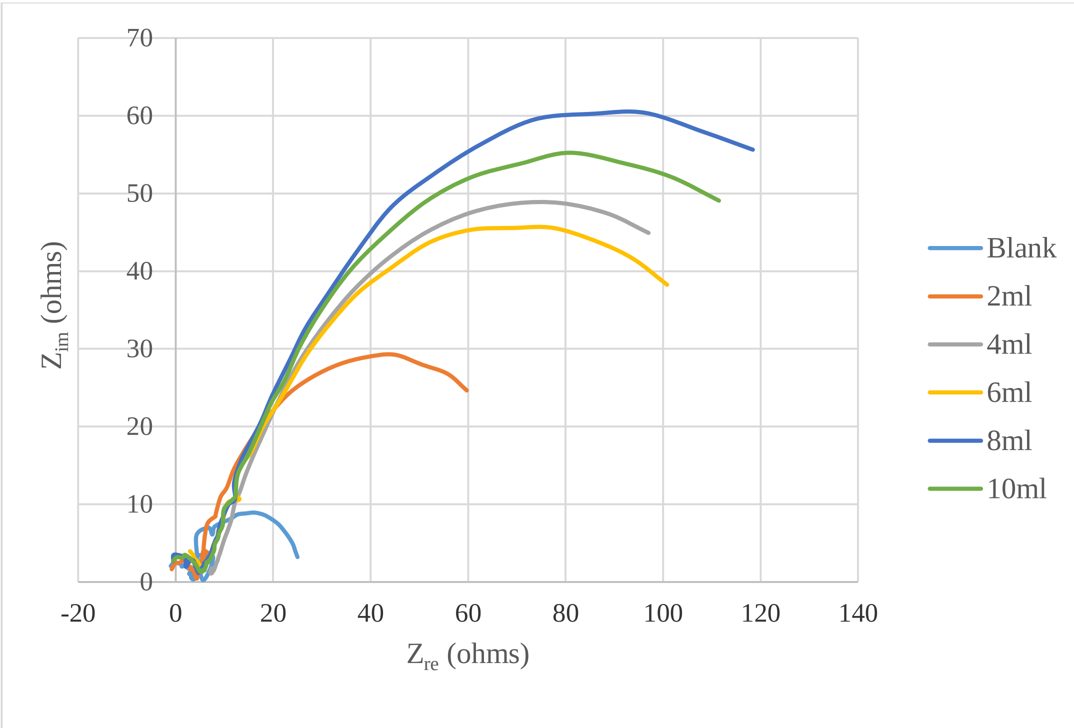

Figure 4.2. Graph of Z_{im} (ohms) against Z_{re} (ohms) on mild steel in aqueous acid solution on the extracted leaf of Chromolaena Odorata (Christmas Bush)

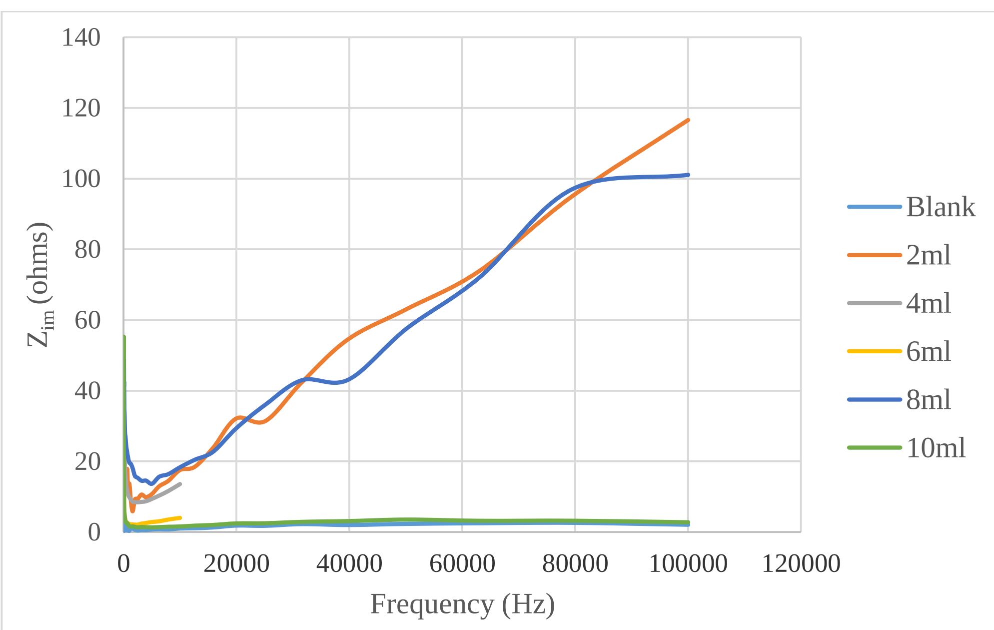

Figure 4.3. Graph of Z_{im} (ohms) against frequency (Hz) on mild steel in aqueous acid solution on the extracted leaf of Chromolaena Odorata (Christmas Bush)

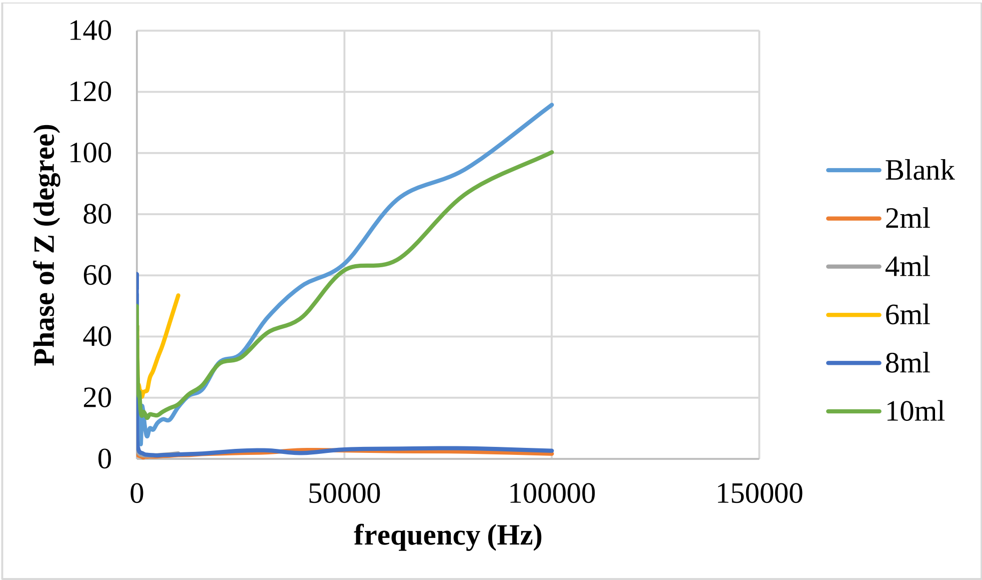

Figure 4.4. Graph of Phase of Z (degree) against frequency (Hz) on mild steel in aqueous acid solution on the extracted leaf of Chromolaena Odorata (Christmas Bush)

Table 4.2: Phytochemical Analysis

TANNIN mg/g		
5	6.029961	6.019144
STERIOD mg/g		
5	3.916991	3.891051
GYCOSIDES mg/g		
TERPENOID mg/g		
ALKALOIOD %		
FLAVONOID mg/g		
5	12.15839	12.12733