



Evaluation of *Codiaeum Variegatum Brilliantissima* - Zanzibar as an Inhibitor of Mild Steel Corrosion in 1 M HCl Using Gravimetric Method

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ABSTRACT

Evaluation of the Corrosion inhibition effect of aqueous extract of *Codiaeum Variegatum Brilliantissima* - Zanzibar (Wire Croton) on mild steel corrosion in an environment of 1 M HCl base stock solution was studied using gravimetric measurement. The work evaluated the effect of immersion time and concentration of inhibitor on the performance of crude extract of CVB-WC as an inhibitor of mild steel corrosion in 1 M HCl environment. The result showed that the surface coverage was close to unity indicating an effective inhibitor with a high inhibition efficiency of 96 – 99%. It is also established that CVB-WC inhibition action is dependent on concentration and time of immersion. Furthermore, the weight loss results are in consonance with the established facts that CVB-WC has compounds that contain –OH, –HN₂, –COOH, –COOC₂H₅, functional groups and heteroatoms, N, O, P that provide the linking bridge to physisorption and chemisorption adsorption processes.

KEYWORDS

Codiaeum Variegatum Brilliantissima – Zanzibar, Weight loss, Adsorption Mechanism, HCl, Mild-Steel, Heteroatoms, functional groups.

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1. INTRODUCTION

The search for cheap and environmentally friendly inhibitors is still ongoing because of corrosion and the associated challenges imposed on infrastructure. The increased cost of production, infrastructure damage, environmental pollution, and loss of human and animal life caused by the effect of corrosion has been a challenge to metals such as mild steel and carbon steel that is widely used in many applications such as construction, petroleum production and refining, chemical

processing, marine applications and others (Fouda *et al.*, 2017; Izionworu *et al.*, 2020b). When metals corrode, they become waste with some level of reusable property as they are usually no longer useful for the intended purpose (Izionworu & Akpa, 2014). Inorganic and organic inhibitors have been used to control corrosion. (Chauhan *et al.*, 2021). However, despite the successes recorded by the use of inorganic and organic inhibitors in corrosion control in all applications of metal such as acid pickling on well tubing, acidizing process, tank descaling, and industrial cleaning, there are issues around their suitability and health implications (Finsgar & Jackson, 2014). They have been found to be toxic and carcinogenic (Subramanian *et al.*, 2000), and the cost of synthesizing organic inhibitors is very high (Umoren & Solomon, 2014).

These environmental, health and economic challenges gave rise to the increase in the search for cheap, convenient, safe and environmentally-friendly corrosion inhibitors (Izionworu *et al.*, 2021a) from natural organic substances that are usually biodegradable in nature to be used as corrosion inhibitors. Reports on different plant extract used as corrosion inhibitors in metal corrosion control in different environments abound and the list includes low-grade leaves of plants, honey, gram flour, onion, roots of plants, shells of plant fruits, potato, gelatin, flowers, plant seeds and gums (Akalezi *et al.*, 2016; Ambrish *et al.*, 2010; Jiyaul Haque *et al.*, 2021; Manimegalai & Manjula, 2015). These various insect and plants products have been reported in different research reports as suitable inhibitors of corrosion of carbon steel, mild steel, nickel sheets and aluminum sheets in Hydrochloric acidic environment (Amitha & Basu, 2011). Few researchers though have evaluated or

reported on organic inhibitors developed by aqueous extraction.

The investigation of *Codiaeum variegatum* Brilliantissima - Zanzibar (Wire Croton - WC), (CVB-WC) as a corrosion inhibitor with good efficiency is justified by the fact that plant extracts contain several phytochemical compounds that have similar molecular and electronic structures that look like the molecules of conventionally synthesized organic inhibitors. These compounds contain groups such as -OH, -HN₂, -COOH, -COOC₂H₅ and heteroatoms, N, O, P, etc. (Jiyaul *et al.*, 2021; Izionworu *et al.*, 2020a; Izionworu *et al.*, 2020c). Available literature shows that CVB-WC as seen from Fourier Transform Infrared (FTIR) and Gas Chromatography – Mass Spectrometry (GC-MS) indicates the presence of functional groups such as aromatics, aliphatic, amines (such as Alkaloid) and aldehydes. The chemical structures of these compounds contain heteroatoms like Nitrogen (N), Sulphur (S), oxygen (O) and conjugated bonds that adsorb on mild steel surfaces (Izionworu *et al.*, 2021a; Izionworu *et al.*, 2021b). Also, the potentiodynamic polarization curve parameters shows that CVB-WC acts as a dual type inhibitor since the corrosion potential was not up to 85 mV when compared to the E_{corr} of blank corrodent solution, but an E_{corr} difference of 15.8 mV was reported for CVB-WC inhibitor at 16% volume content and a difference of 18.4 mV when 67% of CVB-WC inhibitor was used in the corrodent solution. An inhibition efficiency (IE%) of 94.9 and 96.1 % for 16 and 67% volume concentrations respectively has also been reported (Izionworu *et al.*, 2021a)

It is therefore the desire of these researchers to evaluate the potency of CVB-WC as a corrosion inhibitor and demonstrate the possibility of production of this inhibitor using minimal energy requirement. Weight loss measurement was used for this study and the effect of immersion time and concentration of the inhibitor were considered.

2.0 MATERIALS AND METHODS

2.1 Plants

The leaves of the plant *Codiaeum variegatum* Brilliantissima - Zanzibar (Wire Croton - WC),

(CVB-WC), Figure 1, were washed under running water to remove all dirt and then air dried in a shade to conserve their active ingredients. The leaves were grounded to fine powder and sieved with a 75 μ and 15g of the plant powder was soaked in 2000 ml of cold distilled water for 48 hours following standard methods and as reported by Ambrish *et al.* (2010). The solutions containing CVB-WC were filtered thrice using Whatman filter paper and distilled water was added to increase the filtrate up to 2000 ml. The solution was preserved as stock solution and used as an inhibitor. The volume concentrations of the extract used were 73.6, 147.2, 220.8, 294.4 and 368 mg/L. All experiments were carried out at 27 \pm 1 °C and in aerated static solutions.

The concentration of the plant extract in the filtrate was determined following the method employed by Izionworu *et al.* (2020a) and using the expression

$$P_{ex} = W_{p1} - (W_{p3} - W_{p2}) \quad (1)$$

Where P_{ex} is the weight (g) of the extract in the 2000 ml double distilled water, W_{p1}, original weight of the plant dissolved in the double distilled water, W_{p3} is the weight of the crude plant residue plus the weight of Whatman filter paper of 110 mm diameter and W_{p2} as weight of filter paper alone.



Plate 1: *Codiaeum variegatum* Brilliantissima - Zanzibar (Wire Croton)

2.2 Metal Steel Specimen and preparation

Mild steel specimen with the percentage composition of C = 0.05%, Si = 0.03, Mn = 0.6, P = 0.35 and the remaining Fe. The density of the mild steel used was (7.86g/cm³). The mild steel metal for weight Loss analysis was mechanically



press-cut to form coupons with a dimension of 3cm x 3cm x 0.14cm. Each coupon was first scrubbed with emery paper (#200 to #1000), degreased with acetone and then dried with ethanol and stored in a desiccator before use (Arukalam *et al.*, 2016; Izionworu *et al.*, 2020; Oguzie *et al.*, 2010).

2.3 Reagents Used

33% Hydrochloric acid, 98% Ethanol and 98% Acetone, all of analytical grade, acquired from Sinopharm Chemical Reagent Co., Ltd were used without further purification and distilled water. 1 M HCl base stock concentration used was prepared using standard acid dilution procedure.

2.4 Gravimetric Measurement

The gravimetric (weight loss) measurement was carried out using standard methods and as reported by Izionworu *et al.* (2020a) and Oguzie *et al.* (2010) to determine the corrosion rate (C_r), surface coverage (θ) and inhibition efficiency (IE%) using the Equations (2), (3) and (4) respectively.

$$C_r (\text{mmy}^{-1}) = \left(\frac{87,600\Delta W}{\rho At} \right) \quad (2)$$

Where: ΔW is the average negative change in weight (g), ρ is the mild steel density (g/cm^3), A is the surface area of the coupon and t is the immersion time and 87,600 is the conversion constant from cm h^{-1} to mm y^{-1} .

$$\theta = \left(1 - \frac{C_r^{ihb}}{C_r^{blk}} \right) \quad (3)$$

$$\text{IE\%} = \left(1 - \frac{C_r^{ihb}}{C_r^{blk}} \right) \times 100 \quad (4)$$

Where θ is the surface coverage, C_r^{blk} is the coupon corrosion rate in the blank corrodent solution, C_r^{ihb} is the rate of the coupon corrosion in the presence of inhibitor in the corrodent solution. IE% is the inhibitor efficiency.

Ambient temperature (27 ± 1) test was conducted and thereafter the study in a thermostatic water bath that was maintained for temperature test at 313, 323, 333 and 343K. The ambient temperature test was (was) monitored at 24 hours' interval for a period of 120 hours i.e., 5 days as reported by Farag *et al.* (2015) and Arukalam *et al.* (2014). Some researchers have carried out the gravimetric

test in lesser durations between 3 to 10 hours (Wang *et al.*, 2011; Amrita *et al.*, 2015; Solomon *et al.*, 2010; Ashassi-Sorkhabi *et al.*, 2006). The weight loss measurement was done using Adam AAA 260LE analytical balance.

3.0 RESULTS AND DISCUSSION

3.1 Gravimetric Measurement of Codiaeum Variegatum Brilliantissima – Zanzibar - Wire Croton (CVB-WC)

3.1.1 Effect of Inhibitor Concentration and Immersion Time on CVB-WC

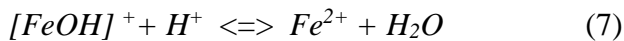
Table 1 gives the calculated results of the surface coverage (θ) of the inhibitor in mild steel corrosion in the corrodent solution of 1 M HCL with different concentrations of CVB-WC at ambient temperature for five days.

The rates of corrosion of the tested coupons were plotted against time of exposure in the absence or presence of different concentrations of CVB-WC as seen in Figures 2. The plot of Figure 2 shows that the tested coupons rate of corrosion in uninhibited 1 M HCL corrodent (blank solution) recorded the highest rate of corrosion when compared with the values of rates of corrosion of the tested coupons shown in Table 1 for corrodent solution with CVB-WC as inhibitor. This is indicative of the effectiveness of CVB-WC as an inhibitor in a 1 M HCL corrosive environment and can be attributed to the existence of polar constituents such alkaloids – Isoquinoline type (Felipe *et al.*, 2013), and Tannin found in CVB-WC (Izionworu *et al.*, 2021a). It is also observed that the plots are not linear and this can be traced to the existence of mill scale on the surface of the mild steel surface which is in agreement with the observation reported by Solomon *et al.* (2010).

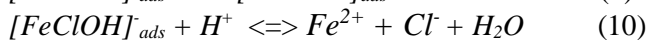
The plot of the blank solution seen in Figure 2 shows that there was gradual drop in the corrosion rate of the coupon. This observation is due likely to the result of iron dissolution in the 1 M HCL solution the iron is immersed in (Oguzie *et al.*, 2007) during the investigation. Iron dissolution takes place through two mechanism – aqueous solution and aqueous solution containing chloride

ion (Cl^-) as seen in Equations (5) to (10) (Noor & Al-Moubaraki, 2008).

Aqueous Solution Mechanism:



Aqueous Solution and Chloride ion (Cl^-) Mechanism:



Where ads is bound species.

The two mechanisms can proceed simultaneously. The formation of these complexes and their accumulation reduces the strength of the acid. Also, the accumulation of oxidation layer on the mild steel surface over time in the solution results in passivity (that is the continuous formation of oxidation layer film on the mild steel surface leading to reduced electrochemical reaction and hence prevention of further corrosion). These two concepts accounts for the reduced corrosion rate that is observed and this is in agreement with the report of Osarolube *et al.* (2008).

Figure 3 graphically represents the variation of mild steel corrosion rate with time of immersion with only different concentrations of CVB-WC extracts in 1 M HCL. All concentrations of CVB-WC used as inhibitor shows a steady reduction in the rate of corrosion of the tested coupons after initial rise after the first day, except for the lowest concentration which had a sharp decrease between the first and third day and thereafter continued decreasing. The gradual drop in corrosion rate of the mild steel for all tested concentrations of the CVB-WC can be credited to rise in adsorption and stability of the particles of CVB-WC on the surface of the tested metal coupons forming a protective coating or film. This situation is corroborated by Saedah (2013).

The inhibition efficiency result presented in Figure 4 graphically shows that within the 5 days' period

of immersion the IE% was stable and this was as a result of decreased metal dissolution. This decrease in metal dissolution may be due to the development of Fe-WC complex film and possible multiple layers of Fe-WC complex on the surface of the mild steel coupons. The more the concentration of CVB-WC the better the complex formed. However, a minimal drop in IE% is observed on the third day likely because of the weakening effect of the weak interspaced Van der Waal force of attraction between the Fe-WC complex formed (Arukalam *et al.*, 2016; Umoren *et al.*, 2014). The overall inhibition efficiency can be related to the presence of Tannin, Phenol and Saponin as established by the FTIR and GC-MS results (Iziorworu *et al.*, 2021b) supported by the reports of Pedroza-Perinan *et al.* (2016).

3.2 Proposed Mechanism of Adsorption for CVB-WC,

The mechanism of inhibitor action on inhibition of mild steel corrosion in hydrochloric acid environment is basically what can be referred to as a "Partico-molecular" adsorption process since there are nano particles and molecules of the phytochemical compounds of the inhibitor, CVB-WC. The negative values of the free energy of adsorption in the tested cold aqueous extract of CVB-WC indicate that the adsorption reaction is chemically feasible hence the inhibited under evaluation inhibits the corrosion of mild steel in HCl environment by the following precepts:

Mild steel in HCl environment results in a charged mild steel surface with empty d-orbitals.

- i) Inhibitor particles contain compounds – phytate, phenyl ring, aromatics such as alkaloid, tannin, flavonoid, reported in the FT-IR and GC-MS characterization (Iziorworu *et al.*, 2021b) with heteroatoms (N, O, P and possibly S), that have lone pair electrons. The heteroatoms get easily protonated in HCl media and ready to bond. There are also delocalized π electrons on the double bonds of phenyl ring, phytate and aromatic compounds present in CVB-WC.

Table 1: Calculated Values of Surface Coverage (Θ) for Tested Coupons Corrosion 1 M HCL with and without Different Concentrations of CVB-WC Extract

CVB-WC System CONC. (mg/L) or	Degree of Surface Coverage (Θ)				
	DAY 1	DAY 2	DAY 3	DAY 4	DAY 5
BLANK	-	-	-	-	-
73.60	0.96	0.96	0.97	0.97	0.97
147.20	0.97	0.97	0.98	(0.97	0.97
220.80	0.98	0.98	0.98	0.98	0.98
294.40	0.99	0.98	0.98	0.98	0.98
368	0.99	0.99	0.99	0.99	0.99

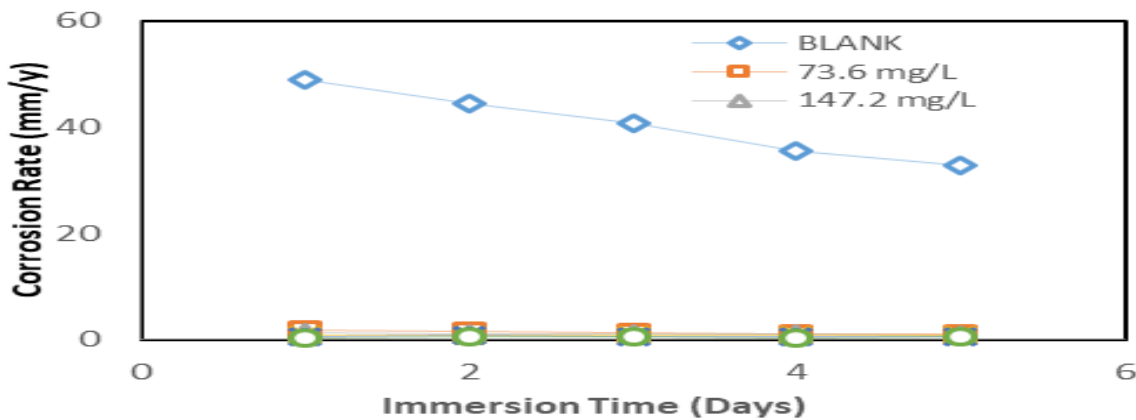


Figure 2: Variation of the Tested Coupons Corrosion Rate against Immersion Time in HCl with and with Different Concentrations of CVB-WC in 1 M HCl base solution

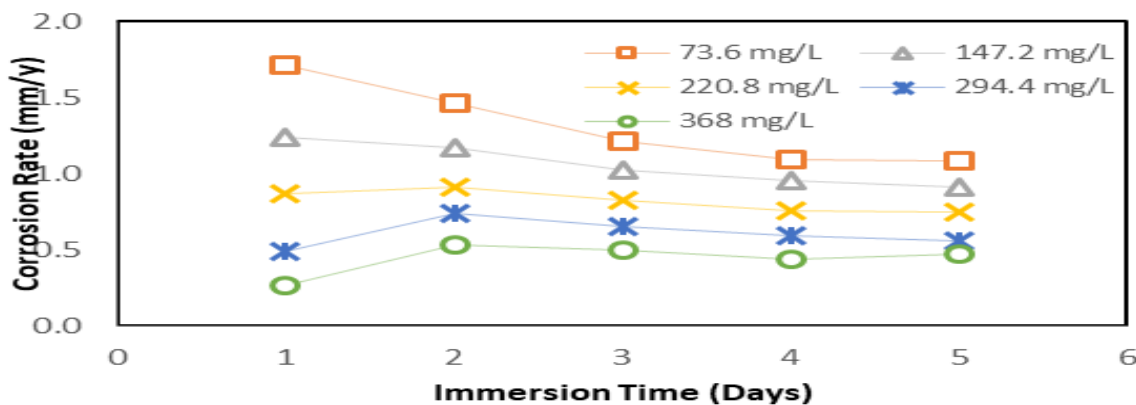


Figure 3: Variation of the Tested Coupon Corrosion Rate against Immersion Time in 1 M HCl with Different Concentrations of CVB-WC Extracts in 1 M HCl base solution

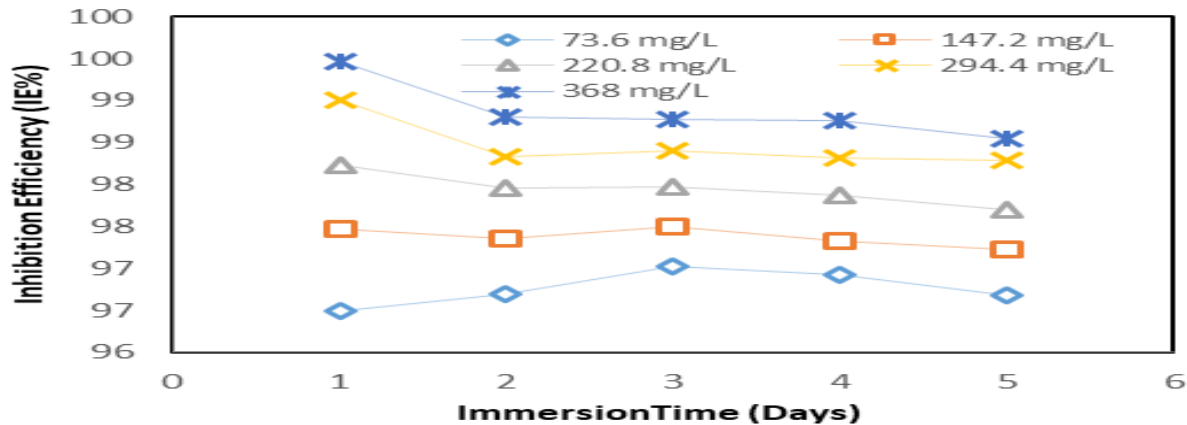


Figure 4: Variation of Inhibition Efficiency of Different Concentrations of CVB-WC Extracts with Time for the Tested Coupons Corrosion in 1 M HCL

- ii) The inhibitors also have neutral molecules. The mechanism of adsorption of the tested inhibitors follows one or more of the following paths which have been corroborated by Oguzie *et al.* (2007); Yadev *et al.* (2015); and Wang *et al.* (2011). Electrostatic attraction between the charged inhibitor “partico-molecules” and charged d orbital of the mild steel surface. Interaction between the lone pair electrons of the heteroatoms (N, O), delocalized π electrons present in the inhibitor and already charged mild steel surface. A combination of electrostatic attraction of charged “portico-molecules” and interaction of lone pair electrons in the case of chemisorption and physisorption adsorption.

The inhibitor’s lone pair electron resulting from heteroatoms N, O, P, and free delocalized π electrons in the double bonds of the phenyl ring and aromatics get adsorbed on the mild steel surface preventing corrosion through the following process:

Anodic reaction: Prevention of, or reduced dissolution of mild steel by the adsorption of delocalized π electrons of the double bonds of phenyl ring and aromatics and the lone pair electrons of the heteroatoms, N, P and O attached to them in the anodic site of mild steel.

Cathodic Reaction: Prevention of, or reduced evolution of hydrogen in the cathodic site in the acidic media by lone pair electrons of the heteroatoms, N, P and O that get adsorbed on the mild steel.

Protonated molecule attraction and reduction in mild steel surface charge density: The charge density on the mild steel surface that has chloride ions attract protonated molecules of the inhibitors forming a compactly or loosely packed double layer of Chloride ion + CVB-WC. The reduction in the current density of the of the electrolyte is an indication of the effect of this electrostatic attraction (physisorption) that paves the way for chemisorption. This agrees with the reports of Wang *et al.* (2011).

Following the mechanism described above it is safe to say that the tested inhibitors reduce corrosion of mild steel in 1 M HCl solution by adsorption of their molecules on the mild steel surface, decreasing anodic and cathodic reactions. Their presence in the solution also decreases the diffusion rate of the reactants to the surface of the mild steel, and reduce the corrosion potential (E_{corr}) and current density (I_{corr}). The adsorption of these molecules on the mild steel surface possibly takes place when they interact with vacant d-orbitals of an iron atom (Rani & Basu, 2012). There is also synergy of the nano portico-molecules.

4.0 CONCLUSION



The decrease in corrosion rate and high inhibition efficiency in the experimental use of the crude extracts CVB-WC indicates that the CVB-WC is a good inhibitors of Mild Steel corrosion in acidic environment. The results revealed that the corrosion rate of mild steel in 1 M HCL decreased with increasing concentration of the tested crude extract proving that the tested crude extract inhibitor is concentration dependent.

As a function of time of immersion, the inhibition efficiency did not show significant difference under the experimental condition implying that particles of aqueous extracts of CVB-WC formed a stable film on the metal surface after the first day of immersion with a possible degradation of the particles in the "Partico-molecular" component of the crude extract.

The study also revealed that the adsorption of the cold extracts of CVB-WC on mild steel surface obeyed Langmuir adsorption isotherm. With a correlation coefficient that is close to unity.

The values of inhibition efficiency calculated from weight loss and potentiodynamic polarization curves reported by Izionworu *et al.* (2021b) are in agreement that the inhibitors get adsorbed through physisorption and chemisorption processes. The polarization curves showed that cold aqueous extract of CVB-WC is both cathodic and anodic corrosion hence they behave mostly as mixed-type inhibitor in hydrochloric acid (Izionworu *et al.*, 2021a).

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