THE POTENTIAL OF *Centrosema pubescens* LEAF EXTRACT IN THE CONTROL OF CORROSION OF MILD STEEL IN ACIDIC MEDIUM

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Abstract: The influence of *Centrosema pubescens leaf extract* on electrochemical corrosion behavior of mild steel, exposed to stimulated acidic medium, was investigated. The results of the phytochemical screening revealed the presence of secondary metabolites such as flavonoid, tannins, anthraquinones, terpenoids, alkaloids, phenols and saponnins. The FTIR also revealed aromatics and functional groups such as R-OH, C=O, C-O and C-H which are attributes of good inhibitors. The performance of the extracts was evaluated using gravimetric method of monitoring corrosion rate in different concentrations of 0.1, 0.2, 0.3, 0.4 and 0.5 w/v. *Centrosema pubescens* gave an inhibition efficiency of 74.91%. This could probably be due to the presence of secondary metabolite and other functional groups revealed by both the phytochemical screening and FTIR spectrum. Thermodynamics data generated suggested co-adsorption through a combination of both physisorption and chemisorption mode of interaction of the inhibitor. The negative free energy indicates that adsorption is spontaneous. The adsorption obeys both Langmuir and Freundlich adsorption isotherms. The degree of linearity is close to unity which indicates strong adsorption of the extracts on the mild steel. Efforts were made to propose a suitable mechanism for the inhibition. The results revealed that *Centrosema pubescens* could serve as an eco-friendly, alternative to sodium chromate, in the protection of mild steel.

Keywords: *Centrosema pubescens*, leaf extract, corrosion, mild steel, acidic medium

Introduction

Mild steel have become relevant to industries due to its properties such as hardness, malleability, ductile, weld ability and most importantly, it is relatively cheap. Even though, mild steel possesses all the properties mentioned above, it is still faced with the problem of galvanic and crevice corrosion especially when used in aggressive environments such as acidic solution. The areas of applications in the industries include the following: acid pickling, industrial acid cleaning, acid descaling and oil well cleaning etc. (Fedare *et al.*, 2016).

Corrosion can be viewed as the degradation or deterioration/destructive attack of material usually metals by reaction with its environment (Adejo *et al.,* 2010). Scientifically, corrosion can be defined as the simultaneous transfer of mass and charge across a metal/solution interface. The damage caused by corrosion can lead to the shutdown of plant, waste of valuable resources, loss or contamination of products, reduction in efficiency, loss of life, and inhibition of technological progress (Popoola *et al*., 2013; Cao *et al.*, 2017).

However, scientists have employed methods such as the use of resistance material, coating, cathodic protection, expensive overdesign and the use of inhibitors to control corrosion process. The use of inhibitors has been well documented as an effective method of protecting metallic materials from corrosion (Ijuo *et al*., 2018). Corrosion inhibitors are substances that in low concentration and in aggressive environment can control or minimize the level of destruction of metals by corrosion. Corrosion inhibition is a surface process which involves the adsorption of the inhibitors on the surface of the metal (Ijuo *et al*., 2018). In an attempt to solve these problems of corrosion, inhibitors such as chromates, silicates, and organic amines are commonly used to control corrosion. As a result, environmental chemists do not encourage these types of inhibitors because they are found to be toxic, costly, and environmentally unfriendly; hence their use is strictly regulated and often prohibited. Researchers are now looking for cheap, eco-friendly; non-toxic inhibitors such as plant extract (Samsath and Jamal, 2017). These plant extracts must contain or possess polar atoms such as nitrogen, sulphur, oxygen and phosphorus which enable them to adhere to the surface of the metal (Karthija *et al.*, 2015). Extracts of various plants have been used for this purpose (Nutan *et al.*, 2012). In this research extract of *Centrosema pubescens* was used for the control of corrosion of mild steel in acidic medium.

Materials and Methods

Collection of samples

Centrosema pubescens leaves were collected from Shango area of Minna, Niger State, Nigeria. The leaves was separated from the stems, washed with distilled water and dried at room temperature in the laboratory. The size of the dried leaves was reduced with a blender machine.

Preparation of plant extract

Fine powder of *Centrosema pubescens* (Cp) (450 g) was transferred into 2000 cm^3 conical flask and enough quantity of ethanol was added, covered with stopper and left to stand for 48 hrs. After which it was filtered using filter paper and the filtrate was concentrated. The stock solution of the extracts was used in preparing different concentrations, by dissolving 0.0, 0.1, 0.2, 0.3, 0.4, 0.5 g of the extracts in 1 cm^3 of 5.0 mol/dm^3 HCl.

Preparation of mild steel

Dimensions of mild steel coupon: 4.0 cm by 2.0 cm by 0.2 cm was used. The coupons were initially polished with different size of emery sand paper, washed with distilled water, degreased with ethanol and the surface was activated in 10% HNO_3.

Corrosion measurement by gravimetric method

The corrosion measurement by gravimetric method was carried out on the pre-weighed coupons. The coupons were washed with distilled water, degreased with ethanol and the surface was activated in 10% HNO_3. The concentration of the inhibitor was varied from 0.0. 0.1, 0.2 to 0.5 g of the extracts in 100 cm^3 of 5.0 mol/dm^3 HCl. The pre-weighed coupons (w_1) were suspended by the aid of a thread into 250 cm^3 beaker containing a particular concentration. This was done for all the various concentration and allowed to stand for three hours. After which the coupons were removed from the solution containing the acidic solution with and without inhibitor, rinsed with distilled water, ethanol and dried in acetone and then re-weighed (w_2). The average weight loss for each concentration was calculated. The degree of surface coverage area (θ), corrosion rate and inhibition efficiency

(IE%) for all the concentration was calculated. This experiment was carried out at room temperature.

$$I_E\% = [1-\frac{W}{W_1}] \times 100 \quad (1)$$

$$\theta = [1-\frac{W}{W_1}] \quad (2)$$

Where: w_1 and w are the values of the average weight losses without and with inhibitor, respectively; as calculated by Abd-El-Nabey *et al.* (2013).

The corrosion rate of the mild steel was also calculated using the method reported by Kavitha *et al.,* (2014).

$$CR = \frac{\Delta W \times 87.6}{DAT} \quad (3)$$

$\Delta W = W_1 - W_2$; W_1= pre-weigh; W_2= value of weight after 3hrs; D= density; A= Area of the mild steel; T = Time of exposure.

Thermal stability

The pre-weighed coupons were immersed in 100 cm^3 of the various concentrations of the inhibitors/blank solution ranging from 0.1 to 0.5 g/100cm^3 in 5 mol/dm^3 HCl and maintained at 303, 313, and 323 K in a thermostatic water bath for 3 h, in replicates. This was retrieved, rinsed in distilled water, degreased in ethanol, dried in acetone and reweighed. The results obtained were fitted into different isotherms (such as Langmuir and Freundlich) and the thermodynamic parameters (such as activation energy, enthalpy change, entropy, and free energy) were calculated (Udom *et al.*, 2017).

Surface morphology

The surface morphology of the corroded uninhibited and inhibited coupons was examined with Scanning Electron Microscopy. The FTIR was also carried out using the *Centrosema pubescens* powder.

Phytochemical screening

Phytochemical screenings were performed using standard procedure as reported by Rosaline *et al.* (2012).

Results and Discussion

Phytochemical constituents

Phytochemical screening of *Centrosema pubescens* leaf extract using qualitative methods revealed the presence of tannins, flavonoids, saponins, terpenoids, steroids, alkaloids and anthraquinone, in *Centrosema pubescens* while the quantitative phytochemical analysis revealed that tannins,(4.45), Alkaloid (0.034), total phenol (14.39), flavonoid (2.85), and saponin (0.65) for *Centrosema pubescens* as shown in Table 1. The chemical structure of the phyto-constituents, contained electron rich bond or hetero atoms that facilitate their electron donating ability; hence the inhibition of the corrosion of mild steel by ethanolic extracts of the plant is attributed to the phyto-constituent of the extract. As reported by Nutan *et al.* (2012), compounds with π bonds generally exhibit good inhibitive properties due to interaction of π orbital with the surface of metal. The possession of π-electrons or suitable functional groups may facilitate the transfer of charge from the inhibitor's molecule to the charged metal surface (physical adsorption) or transfer of electron from the inhibitor's molecule to the vacant-orbital of the metal (chemical adsorption). Mshelia *et al.* (2017) in a study implicated tanins as a corrosion inhibitor. The inhibitive properties of tannins have been attributed to the polyphenolic fraction of the tannins moieties, which ensures effective protection of the metal surfaces.

Flavonoid has anti-oxidant activity, anti-allergic, anti-cancer, anti-inflammatory and anti-viral activities. Al-qudah (2011) established in his study, about 92% inhibitory efficiency of different types of flavonoids. Terpenoids and other constituents also possess functional groups which are capable of chelating with metallic ions and thus facilitating strong coordination on the metal surface (Rodriquez-Torres *et al.*, 2018). Nitrogen compounds such as amines, amino acids and quaternary ammonium salts exert their best efficiencies in preventing corrosion of iron and steel in HCl solutions while they are less effective in H_2SO_4 (Loto *et al.*, 2012).

Table 1: Phyto-Chemical Screening Values (mg/g) of *Centrosema pubescens* leave extract

Phytochemical Test	Qualitative analysis	Quantitative analysis (mg/g)
Tannins	++	4.98
Flavonoid	++	2.34
Saponins	++	0.45
Terpenoid	+	N.D
Steroid	++	N.D
Alkaloid	+	0.020
Anthraquinone	+	N.D
Phenol	+	10.16

++ = strongly positive; + = positive ND – not Determined

Table 2: Gravimetric analysis of *Centrosema pubescens*

Concentration (W/V)	Mass Loss (mg)	IE%	Surface Area (θ)	Corrosion Rate (mm/yr)
0.0	267.0	-----	--------	6.2073
0.1	150.0	48.820	0.4882	3.4872
0.2	133.0	50.187	0.5019	3.0920
0.3	117.0	56.180	0.5618	2.7201
0.4	100.0	62.547	0.6255	2.3248
0.5	67.0	74.906	0.7491	1.5576

Gravimetric analysis of Centrosema pubescens

Weight loss, percentage inhibition efficiency, corrosion rate and surface coverage in 5.0 mol/dm^3 HCl solution with different inhibitors of leaves extract are given in Table 2. It can be seen from Table 2 that the inhibition efficiency of the inhibitor increases with increasing concentration of inhibitor. The maximum inhibition efficiency (74.91%) was obtained for *Centrosema pubescens* (Cp) at an inhibitor concentration of 0.5 w/v in 5.0 mol/dm^3 HCl solution for leaves extract. The variation of percentage inhibition efficiency (IE %) with inhibitor concentration is shown in Fig. 1, for leaves extract 5 mol/dm^3 HCl solution. Variation of percentage inhibition efficiency (IE %) with the concentration of inhibitor indicated that the inhibition efficiency increases with increasing inhibitor concentration and the rate of corrosion also decreases with corresponding decrease in weight loss. From Table 2, it is clear that the surface coverage increases with increasing concentration of inhibitor due to the adsorption of the phyto constituents (Kenneth and Sunday, 2012; Omotoyinbo *et al*., 2013; Abdulwahab *et al*., 2012).

Thermal stability result

To assess the thermal stability or the effect of temperature on corrosion and corrosion inhibitive process, weight loss experiments were performed at 10 K intervals in the temperature range 303 to 323 K in uninhibited acid (5 mol/dm^3 HCl) and in inhibitor solutions containing different concentrations of *Centrosema pubescens* leaf extract calculated (Udom *et al.*, 2017; Fig. 2).

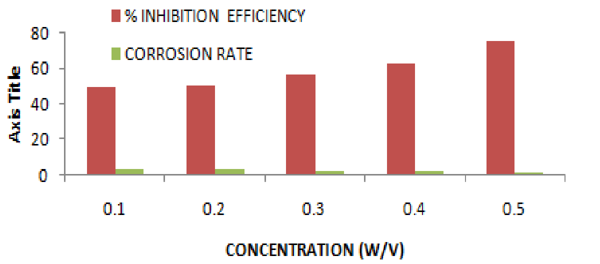

Fig. 1: Variation of surface coverage area, percentage inhibition efficiency (IE%) and corrosion rate with inhibitor concentration in 5 mol/dm^3 HCl

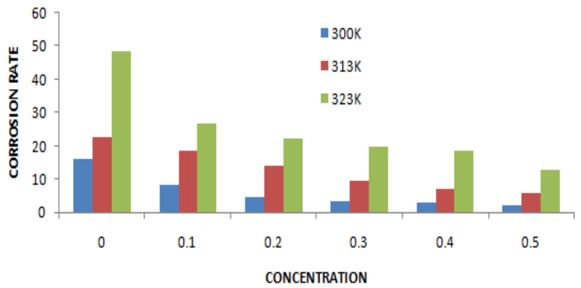

Fig. 2: Variations of corrosion rate with extract concentration and temperature

Effect of temperature on corrosion rate (thermal stability)

Corrosion rate of mild steel increased with increase in temperature, this is as result of desorption of the extract on the surface of the mild steel but decreases with increase in the concentration of the extracts (Fig 2). There are similar observation reported in the literatures (Ating *et al.,* 2010; Vijayalakshmi *et al.,* 2011; Ismail *et al.,* 2011; Okafor *et al.,* 2012; Fadare *et al.,* 2016). The rate of corrosion of mild steel increased as a result of increase in the average kinetic energy of the reacting molecules. However, the corrosion rate is much decreased for higher concentration of inhibited acid solution than the solution with low concentration of inhibitors as seen in Fig. 2. The decrease in the corrosion rate can be attributed to mitigating effect of the plant extract on the corrosion rate of the mild steel.

Thermodynamics studies

The value of activation energy for uninhibited aqueous is 68.52 kJ/mol. The activation energy for *Centrosema pubescens* extract ranges from 74.19 to 100.37 kJ/mol with increasing inhibitor concentration of the extract. From Table 3, it is found that activation energy values of the inhibitor acidic solutions are higher than the uninhibited acidic solutions. It shows that the values of activated energy enhance with an increase in the inhibitor concentrations from 0.1w/v to 0.5w/v is due to the deceleration of the corrosion rate of the mild steel.

Table 3: Variation of thermodynamics parameters for mild steel in 5 mol/dm^3 HCl with inhibitor concentration and temperature

Concn (w/v)	Ea (KJ/mol)	ΔH_{ads} (KJ/mol)	ΔG_{ads} (KJ/mol) 30°C	30°C
0.0 (Blank)	68.52	-----	------	------
0.1	74.19	-110.89	-1.79	-1.79
0.2	71.89	-57.20	-1.67	-1.67
0.3	79.90	-179.51	-1.64	-1.64
0.4	86.46	-260.02	-1.65	-1.65
0.5	100.37	-432.20	-1.69	-1.69

The result (Fig. 2) suggests that corrosion inhibition by *Centrosema pubescens* extract is brought about by the increasing its activation energy. There is an increase in activation energy as a result of adsorption of the constituents on the mild steel surface making a barrier for mass and charge transfer. According to Manimegalai and Manjula (2015), the threshold for physical adsorption is <80 kJ/mol and >80 kJ/mol for chemical adsorption. This means that adsorption of the extracts on the mild steel surface is physisorption and chemisorptions from the result (Fig. 2) obtained. The higher activation energy implies a slow dissolution of the mild steel (Okafor *et al.,* 2010). Fig. 3 also shows the result of the calculated heat of adsorption, ΔH_{ads} of ethanolic extract of *Centrosemapubescens* on the surface of mild steel. The values of ΔH_{ads} were negative and ranged from -110.89 to -432.20 kJ/mol indicating that the adsorption of the extracts is exothermic (Adeniji and Akindehinde, 2018). The negative value of ΔG_{ads} is a suggestion that the adsorption of *Centrosema pubescens* leaf extracts onto the metal surface is a spontaneous process and the adsorbed layer is stable. Usually in the adsorption of free energy involved in a physical process, ΔG_{ads} is$< -40KJ/mol$ (Stephen *et al.,* 2014).

Adsorption consideration

The Values of adsorption parameters deduced from Langmuir adsorption plot as seen in Fig. 3, the values of degree of linearity (R^2) was found to be close to unity indicating strong adherence of the adsorption of the extracts on the metal surface. This fits into Langmuir adsorption isotherm.

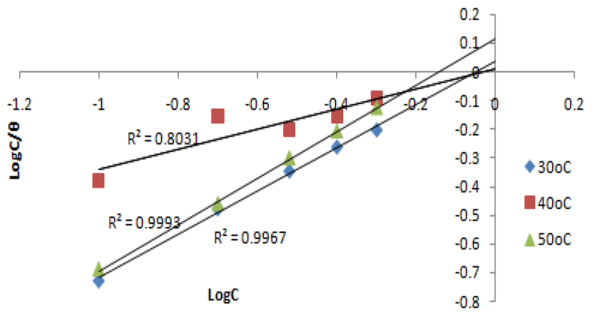

Fig. 3: Langmuir isotherm of *Centrosemapubescens* extracts on mild steel in 5 mol/dm^3 HCl at difference temperature

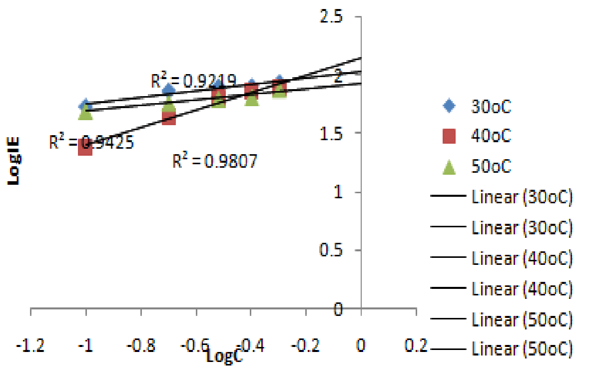

Fig. 4: *Centrosema pubescens* Freundlich isotherm extracts on mild steel in 5 mol/dm^3 HCl at difference temperature

Freundlich Isotherm

Freundlich adsorption isotherms is commonly used to describe the adsorption characteristics for the heterogeneous surface (Ituen *et al.,* 2017). Figure 4 revealed Freundlich's adsorption isotherm of *Centrosema pubescens* extracts on the surface of the mild steel and is given by the equation 4 and 5 (Sharma and Sharma, 1999).

$$\frac{x}{m} = Kc^{1/n} \quad (4)$$

$$\mathrm{Log}\frac{x}{m} = logK + \frac{1}{n}logC \quad (5)$$

The fraction $\frac{x}{m}$ in equation 4 has been found to be approximate to the inhibition efficiency of the inhibitor, K and n are constant. Slope equal to $\frac{1}{n}$ and intercept = log K. Therefore, from equation 4, a plot of log inhibition efficiency (IE%) versus log C produces a straight line that obeyed Freundlich adsorption isotherm.

Scanning electron microscopy

Surface of the mild steel strip used for the study were examined using scanning electron microscope with 500× magnifications. SEM micrographs of the mild steel after immersion in 5 mol/dm^3 HCl solution in the absence and presence of inhibitors (*Centrosema pubescens*) are presented in Plates 1 and 2. Surface analysis using scanning electron microscope provided more information on the level of attack as well as inhibition strength of both extract on the surface of the mild steel. Plate 1 shows the micrograph of the micrograph of the mild steel immersed in 5 mol/dm^3 HCl solution in the absence of inhibitor for 5 h. The surface of the treated coupon shows etching composed of white and dark areas (Fouda *et al*., 2014; Fadare *et al*., 2016); the white areas represent the pearlite (mixture of ferrite and cementite (Fe_3C) in a lamellar form). It indicate clear disarrangement in the surface formation of the mild steel due to high metal dissolution rate and more active site available for corrosion (Ji *et al*., 2012; Jonsirani *et al*., 2012; Fadare *et al*., 2016).

The micrograph of the mild steel immersed in 5 mol/dm^3 HCl solution containing 0.1 w/v SO (Plate 2) shows that the surfaces were smoother when compared to the one without inhibitors. The plates show less extensive attack in the presence of the extract than in the uninhibited acid. This implies that the extracts components absorbed on the surface forming protective film over the surface of the mild steel thereby decreasing the dissolution in acidic medium. The protective film acts as a barrier between the steel and the aggressive environment and thus retards the corrosion reactions (Loto *et al*., 2011). This is in line with various studies on plant extracts (Abdulwahab *et al*, 2012; Noyel *et al*., 2015; Fadare *et al*., 2016).

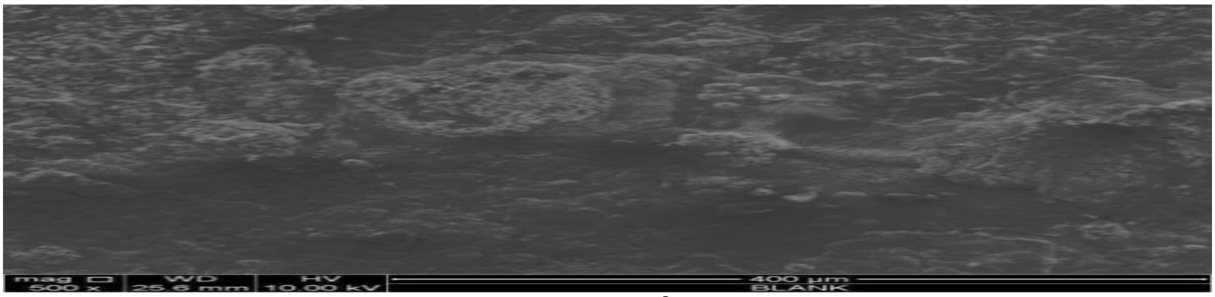

Plate 1: SEM photograph of the mild steel treated with 5 mol/dm^{3}HCl for 5h without inhibitor

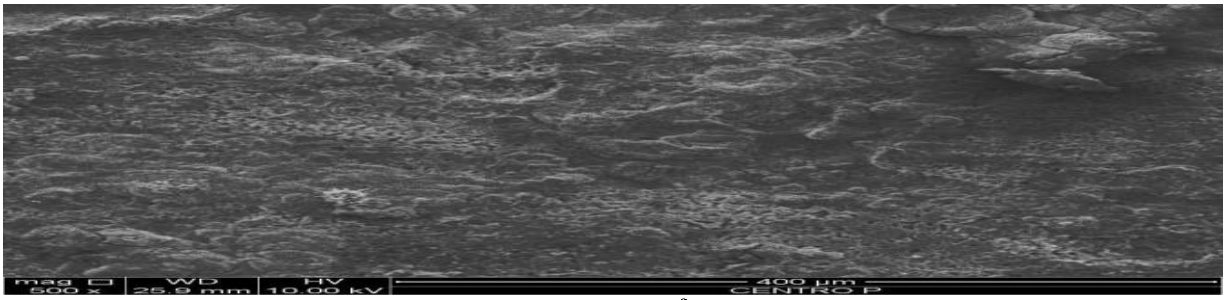

Plate 2: SEM photograph of the mild steel treated with 5mol/dm^3 HCl for 5h in the presence of 0.1%w/v of extract of *Centrosema pubescens* **as inhibitor**

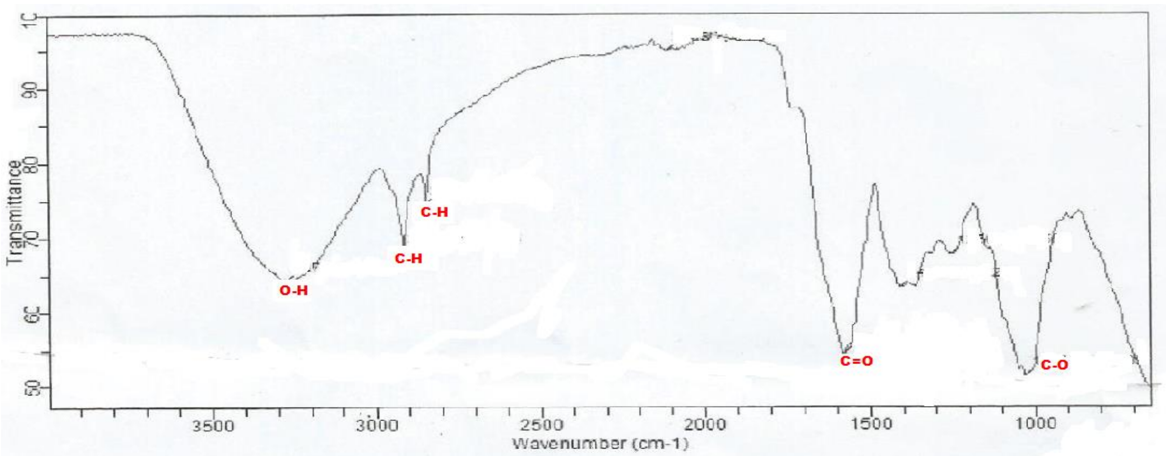

Fig. 5: FTIR Spectrum of *Centrosema pubescens*

FTIR analysis

From Fig. 5, the FTIR for *Centrosema pubescens* shows strong absorptions at 3272.6 cm^{-1} for OH stretching mode. The absorption at 2918.5 cm^{-1} is aromatic C-H band stretching mode. The peak at 1580.4 cm^{-1} for *Centrosema pubescens* correspond to C=O stretching mode. The peaks at 1408.9 and 1256.1 cm^{-1} indicate the presence of aryl OH. Finally, the absorption at 1028.7 cm^{-1} shows the stretching mode of C-O.

Mechanism of inhibition

The corrosion inhibition action of *Centrosema pubescens* and other natural plants inhibitor in acidic solution major constituents exist in either of neutral molecules or as protonated molecules (cations). *Centrosema pubescens* may be adsorbing on the surface by one and/or more, via electrostatic interaction of protonated molecules with already adsorbed chloride ions, interaction between unshared electron pairs of hetero atoms and vacant d orbital of iron surface atoms, donor-acceptor interactions between the p-electrons of aromatic ring and vacant d-orbital of surface iron atoms. It is usually not possible to assign a single general mechanism of action to inhibitors, because the mechanism may change with experimental conditions. Thus, the predominant mechanism of action of inhibitor in acidic solutions may vary with factors such as concentration of the extracts, the nature of the anion of the acid, the presence of other species in the solution, the extent of reaction to form secondary inhibitors and the nature of the metal (Albrakaty *et al*., 2018).

In the present situation two modes of adsorption could be considered: Firstly, the neutral *Centrosema pubescens* may adsorb onto the metal surface via the chemisorption mechanism, involving the displacement of water molecules from the metal surface and sharing electrons between the o-atom and Fe

$$Fe + H_2O \rightarrow Fe.H_2O_{ads} \quad (6)$$

$$Fe.H_2O_{ads} + CP \rightarrow FeCP_{ads}{}^{-} + H^{+} + CP \quad (7)$$

$$Fe.H_2O_{ads} + CP \rightarrow FeCP_{ads} + H_2O \quad (8)$$

The molecules can also adsorb on the surface of the metal on the basis of donor-acceptor interactions between π −electrons of aromatic ring and the vacant d-orbitals of Fe. Secondly, the protonated *Centrosema pubescens* may adsorb through electrostatic interactions between the positively charged molecules and negatively charged metal surface. In other words, there may be a synergy between Cl^{-} and *Centrosema pubescens*, which improves the inhibitive ability of the inhibitor.

When protonated *Centrosema pubescens* is adsorbed on the surface, a coordinate bond may be formed by partial transference of electrons from pair atoms (O-atoms) to the metal surface. In addition, owing to lone – pair electrons of O – atom in *Centrosema pubescens* or protonated *Centrosema pubescens* may combine with freshly generated Fe^{2+} ions on steel surface forming metal inhibitor complexes.

$$Fe \rightarrow Fe^{2+} + 2e^{-} \quad (9)$$

$$CP + Fe^{2+} \leftrightarrow [CP - Fe]^{2+} \quad (10)$$

$$[CPH_x]^{x+} + Fe^{2+} \leftrightarrow [CPH_x - Fe]^{(2+X)^{+}} \quad (11)$$

Similar type of mechanism was also proposed by Leelavathi and Rajalakshm (2013). These complexes might be adsorbed onto the mild steel surface by a weak force of attraction called van der Waals' force to form a protective film to prevent the metal from corrosion.

Conclusion

The results obtained from the present study showed that *Centrosema pubescens* is a good inhibitor and acted as a mixed type inhibitor in 5M HCl. The negative free energy of adsorption indicates strong and spontaneous adsorption of *Centrosema pubescens* on the mild steel surface. The increase in the activation value to the increase in concentration of the extract shows that corrosion rate decrease signifies the protection of the metal by the inhibitor. FTIR spectra clearly reveal the functional group responsible for the inhibitory ability of *Centrosema pubescens* SEM micrograph revealed that corrosion inhibition is due to the adsorption of *Centrosema pubescens* on the mild steel.

Conflict of Interest

The authors declare that there is no conflict of interest related to this study.

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