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Research Paper

Inhibition effect of *Hibiscus rosa sinensis* Leaves Extract on Corrosion Rate of Mild Steel in HCl Medium

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Abstract: *There is a growing trend to utilize plant extracts compounds as corrosion inhibitor. Corrosion Inhibition of Mild Steel in 2M Hydrochloric acid (HCl) by leaves extract of Hibiscus rosa sinensis has been studied at 35 °C using weight loss measurement and spectrophotometric technique. Results confirmed that the leaves extract of Hibiscus rosa sinensis inhibited the acid induced corrosion of Mild Steel (MS). The inhibition efficiency increases with increases in concentration of the extract. The investigation showed optimal inhibition efficiency of about 87.23% at 1.08%v/v concentration of inhibitor. Spectrophotometric technique was used to evaluate the inhibition efficiency based on ferrous ion concentration. As concentration of inhibitor increase, decreases in concentration of ferrous ion that indicate increase in inhibition efficiency of inhibitor. Inhibitive effect was afforded by adsorption of the extracts' components which was approximated by Freundlich, Langmuir and Temkin adsorption isotherm. Characterization of Hibiscus rosa sinensis leaves extract was carried out using GC-MS technique.*

Keywords: Corrosion Inhibition, *Hibiscus rosa sinensis* leaves extract, Mild steel, Spectrophotometry.

Introduction

The meaning of the word corrosion is the substance usually a metal or its alloys which change in its properties because of a reaction with its environment. Normally it specifically applies to metals. Corrosion causes enormous losses which rise yearly with the increased using of metals industrial development. The accepted concept of the corrosion is that it is a result of an electrochemical reaction taking place on the surface of the metal where the metal is converted in to metal oxides or other corrosion products with some metals[1-4].

The study of the corrosion behavior of iron, iron alloys, and steel in corrosive media has continued to attract considerable attention because of the many important applications of the metal. The corrosion of Mild Steel is of such interest because Mild Steel is widely used as a constructional material in many industries due to its excellent mechanical properties and low cost. Acids are widely used for the pickling, cleaning, descaling, and so on of iron and steel[5-8].

Corrosion inhibitors are generally used in these processes to control the dissolution of the metal. Most of the well-known acid corrosion inhibitors are organic compounds containing nitrogen, sulfur, and oxygen atoms. Interest has grown in the use of organic compounds as inhibitors for the aqueous corrosion of metals. High corrosion inhibition potentials are exhibited by many organic molecules, however, these materials pollute the environment during their synthesis and use. Because of the currently imposed environmental requirements for ecofriendly corrosion inhibitors, there is a growing interest in the use of natural products such as leaves or seed extracts. The term "ecofriendly corrosion inhibitor" or "green inhibitor" refers to substances that are biocompatible, such as plant extracts, because they are of biological origin. Green approaches to corrosion mitigation entail the use of substances, techniques and methodologies that reduce or eliminate the use of products, byproducts, solvents, reagents, and so forth that are hazardous to human health or the environment in combating corrosion[9-16].

Present work is focused on methanolic leaves extract of *Hibiscus rosa sinensis*. The main objective of this work was to study the inhibitory effects of the methanolic leaves extract of *Hibiscus rosa sinensis* corrosion inhibition of Mild Steel in 2 M Hydrochloric acid. The corrosion behavior of Mild Steel in 2 M Hydrochloric acid with and without inhibitor was studied using weight loss measurement and spectrophotometric technique. Characterization of *Hibiscus rosa sinensis* leaves extract was carried out using GC-MS technique.

Method and Material

Preparation of specimens

The elemental composition of Mild Steel is as follows , C (0.038%), Si (0.020%), Mn (0.070%), P (0.021%), S (0.016%), Cr (0.035%), Ni(0.012%), Mo (0.028%), Cu(0.018%), Fe (99.742 %). Square specimen of Mild Steel area 3.0 cm X 3.0 cm (thickness = 1.2 cm) with a small hole of about2 mm diameter. A specimen of the Mild Steel was first washed with distilled water several times and dried. It was then cleaned by buffing to produce a mirror finish and were then degreased. The final polishing was done using jeweler rouge, which gave a mirror like finish. The specimen was finally degreased by immersion in A.R. Carbon tetrachloride (sulphur free).

Preparation of green inhibitor

Sample of *Hibiscus rosa sinensis* leaves were obtained from a botanical garden located at Gujarat university, Ahmadabad, Gujarat, India. Leaves were sun dried, grounded using motor and pestle and 10 grams powder was weighed and refluxed in 100 mL methanol for 2 hours and was kept overnight to extract the basic components. The solution was filtered using vacuum pump and the filtrate was used to prepare different concentrations by adding 0.260, 0.739 and 1.086 % v/v in 2M Hydrochloric acid solution, respectively[17-19].

Preparation of Test media

230mL of 2M Hydrochloric acid solution was measured into beakers. The extract solution of *Hibiscus rosa sinensis* leaves in concentrations of 0.260, 0.739 and 1.086 % v/v was put separately in the beakers. Leaves extract was not added to the beaker, which was used as blank.

Weight loss Method

Mild Steel specimen was immersed in 230 mL test solution of 2M Hydrochloric acid in presence and absence of inhibitor at different concentrations for 3hrs at 35^{o}C. One specimen was suspended by a glass hook in each beaker containing 230 mL of test solution which was open to the air at 35^{o}C to the same depth of about 1.5 cm below the surface of the test solution. The weight of specimen before and after the immersion was determined. The inhibition efficiency, corrosion rate and surface coverage were calculated by Equation 1, 2 and 3 respectively.

$$\text{Inhibition efficiency (IE \%)} = \frac{W_u - W_i}{W_u} \text{ X } 100 \quad ...(1)$$

where, W_u = weight loss in uninhibited acid, and W_i = weight loss in inhibited acid.

The corrosion rate was calculated as follows:

$$\text{Corrosion rate} = \frac{\Delta W}{A.t} \quad .... (2)$$

where, ΔW = difference of weight loss, A= surface area of specimen, t = time

$$\text{Surface coverage } (\theta) = \frac{W_u - W_i}{W_u} \quad ....(3)$$

where, W_u = weight loss in uninhibited acid, and W_i = weight loss in inhibited acid

Spectrophotometric study

Preparation of Standard solution

Take 30mL of 1, 10-Phenanthroline solution, Add 10mL of buffer, as well as 1mL of Hydroxylamine hydrochloride and add 2,4,6,8,10 mL aliquots of the ferrous solution, respectively, to the flasks. Allow to stand 10min. Measure the absorbance at 510nm.

Preparation of Sample

Take 0.3mL sample, make it neutral with Sodium hydroxide, Add 30mLof 1, 10-Phenanthroline solution and 10mL of the buffer, as well as 1 mL of Hydroxylamine hydrochloride to the flasks. Allow to stand 10min. Measure the absorbance at 510nm.

Visible spectrophotometric method was carried out on the prepared Mild Steel samples after immersion in 2M Hydrochloric acid with and without addition of inhibitor at 35^{o}C for 3 hrs.

$$\eta\% = \frac{Co - Ci}{Co} \times 100$$

where C_o mg /230 mL and C_i mg/230 mL are ferrous ions concentrations after immersion in 2M HCl without and with inhibitor respectively[20].

GC-MS analysis of *Hibiscus rosasinensis*

The GC-MS analysis of *Hibiscus rosasinensis* leaves extract was performed by using GC-MS QP-2010 plus of Shimadzu. The column used was Rtx-5 of 30m X 0.25mm X 0.25µm size. The initial column temperature was 80°C rising 270°C at a rate of 5°C/min and the temperature was maintained for 8 minutes. The temperature was further increased to 310°C at a rate of 15°C/min with a hold time of 17 minutes. Split injection was done for the analysis with a split ration of 10. The ion source of mass spectrometer was held at 230°C with an interface temperature of 280°C. Detection was performed in full scan mode from m/z 40 to 650.

Results and Discussion

Weight loss study

The percentage of inhibition efficiency obtained at different concentrations of *Hibiscus rosasinensis* leaves extract at 35°C are summarized in the Table 1. It is indicated that inhibition efficiency of the extract on corrosion of mild steel increases with increase in concentration.

Table 1: Effect of inhibitor concentration on weight loss and inhibition efficiency for Mild Steel in 2 M HCl at 35°C for 3 hrs

Inhibitor concentration (%v/v)	Weight loss (mg)	Weight loss (mg/dm^2)	Corrosion rate ($mg/dm^2/hr$)	Surface coverage (θ)	Inhibition efficiency (%)
HCl only	235	1,222	-	-	-
0.260	75.5	392.6	13.14	0.678	67.87
0.739	44.0	228.8	12.24	0.812	81.27
1.086	30.0	156.0	10.22	0.872	87.23

Adsorption behavior

Adsorption isotherm is usually used to describe the adsorption process. The relationship between the coverage of an interface with an adsorbed species (the amount adsorbed) and the pressure of the gas or the concentration of the species in solution is given by an adsorption isotherm [21]. The most frequently used adsorption isotherms are Langmuir, Temkin, and Freundlich. But it was found that the leaves extract obeys Langmuir, Temkin, and Freundlich adsorption isotherm for corrosion inhibition of Mild Steel in 2M Hydrochloric acid as can be seen from correlation coefficient value of r^2 = 0.9981 for Langmuir, r^2= 0.9998 for Freundlich and r^2 = 0.9982 for Temkin adsorption isotherm.

Langmuir isotherm

$$\frac{C}{\theta} = \frac{1}{K} + C$$

Where θ is the degree of surface coverage, C is the %v/v of inhibitor concentration in solution, K_{ads} is the equilibrium constant for adsorption process Figure 1.

Freundlich isotherm

$$\theta = C_1 P^{1/C_2} \text{ or } = C_1 C_{inh}^{1/C_2}$$

Where, C_1 and C_2 are constants, P is the gas pressure and C_{inh} is the concentration ofa inhibitor.

$$\log \theta = k \log C_{inh} + k_1 \log C_{inh}$$

Hence a plot of log θvs log C_{inh} will give a straight line Figure 2. The isotherm is believed to be valid at low coverage.

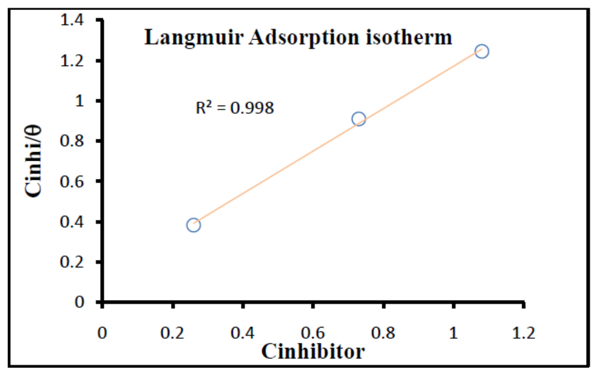


Figure 1: Langmuir isotherm for adsorption of *Hibiscus rosa sinensis* leaves extract onto Mild Steel surface in 2M HCl

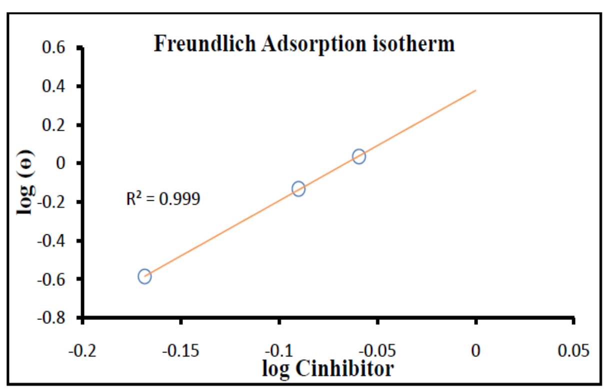


Figure 2: Freundlich isotherm for adsorption of *Hibiscus rosa sinensis* leaves extract onto Mild Steel surface in 2M HCl

Temkin isotherm

An assumption of the Langmuir isotherm is the independence and equivalence of the adsorption sites. Deviations from the isotherm can be often traced to the failure of these assumptions. For example, the enthalpy of adsorption becomes less negative as θ increases, which suggests that the energetically most favorable sites are occupied first.

The Temkin isotherm is represented as:

$$\theta = C_1 \ln (C_2 P)$$

where, C_1 and C_2 are constants, corresponds to assuming that the adsorption enthalpy changes linearly with pressure. The isotherm is also written in the form:

$$\theta = \frac{1}{a} \log C_{inh} + \text{constant}$$

It assumes a linear dependence of the adsorption energy on fractional coverage, and a plot of θ vs log C_{inh} yields a straight line shown in Figure 3.

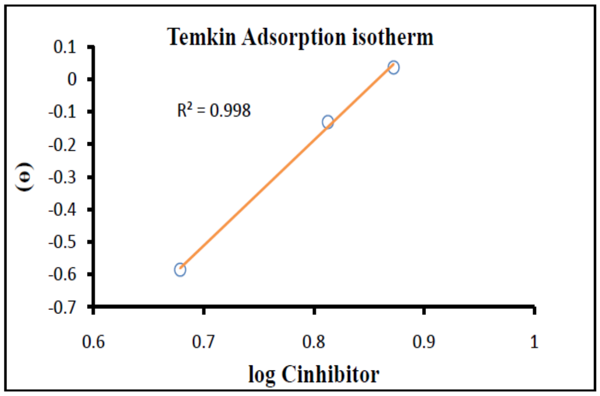


Figure 3: Temkin isotherm for adsorption of *Hibiscus rosa sinensis* leaves extract onto Mild Steel surface in 2M HCl

Figure (1-3) reveals that the inhibitor obeys Langmuir, Freundlich, Temkin adsorption isotherm. Data for adsorption isotherm shown in Table 2.

Table 2: Data for Adsorption isotherm

C inhibitor (%v/v)	Log C inhibitor	Weight loss Wi (mg)	(θ)	Log (θ)	C inhibitor / θ
0.260	-0.5850	75.5	0.6787	-0.1683	0.383
0.736	-0.1313	44	0.8127	-0.0900	0.909
1.086	0.0358	30	0.8723	-0.0593	1.244

Spectrophotometry Technique

The amount of ferrous ions dissolved in solution for both uninhibited and inhibited samples were determined by using JASCO V570 UV-Visible absorption spectrophotometer results given in Table 3.

Absorbance spectra of standard, sample, and relation between the concentrations of the inhibitor and ferrous ions shown in Figure (4-6).

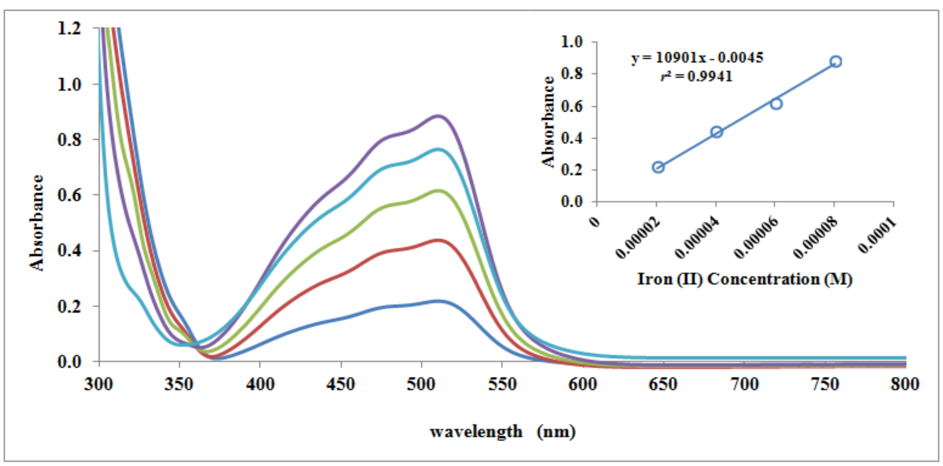


Figure 4 : Representative absorbance spectra for the standard solutions of Iron (II) (0.00002-0.00010 M) and its linearity curve

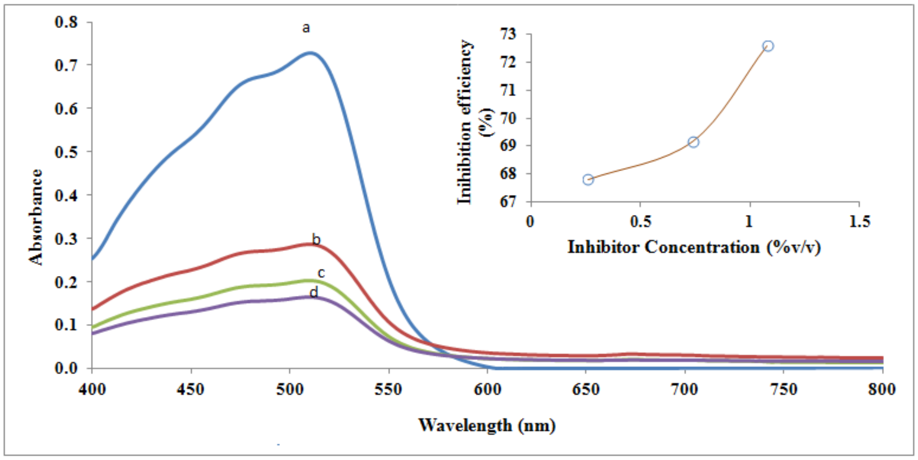


Figure 5:Representative absorbance spectra of sample solutions (a) without the inhibitor, and with (b) 0.26 %v/v (c) 0.739 %v/v and (d) 1.086 %v/v ofinhibitor solution (10 % v/v), and its inhibition efficiency

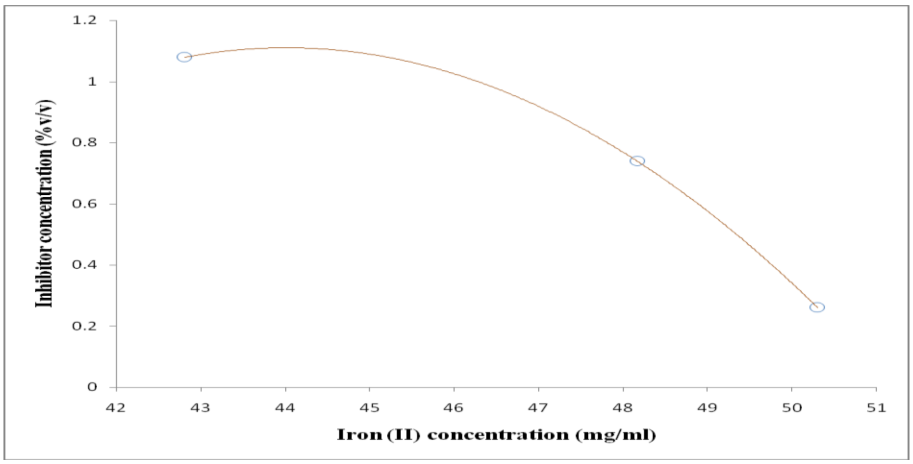


Figure 6: Representations for the relationship between the concentrations of the inhibitor and ferrous ions

Table 3: Effect of inhibitor concentration on ferrous ions concentration and inhibition efficiency for Mild Steel in 2M HCl at 35°C

Time	Inhibitor Concentration (%v/v)	Concentration of ferrous ion(mg/230ml)	Inhibition efficiency (%)
3hrs	Blank	156.28	-
3hrs	0.260	50.30	67.81
3hrs	0.739	48.16	69.18
3hrs	1.086	42.81	72.60

For the spectrophotometric determination of ferous ions in aqueous solution, a tricyclic nitrogen heterocyclic compound, 1, 10 phenanthroline($C_{12}H_8N_2$, phenanthroline) was used as the ligand that reacts with metals such as iron, nickel, ruthenium, and silver to form strongly coloured complexes. With ferrousions (Fe^{2+}), it reacts in a ratio of 1:3 to for man orange red coloured complex$[(C_{12}H_8N_2)_3Fe]^{2+}$ in aqueous medium as per the following equation.

Fe^{2+} + 3 Phen → Fe (Phen) $_3{}^{2+}$

The absorption of UV-Visible radiation by $[(C_{12}H_8N_2)_3Fe]^{2+}$ complexes can be due to, Excitation of iron (II): the type of transition observed commonly will be d→d transitions but it has very low intensity and can't be used for quantitative purpose.

- Excitation of ligand *phenanthroline* is an organic molecule that can undergo π→π* or n→π* transitions and can absorb UV-Visible radiation
- Charge transfer transition: The movement of electrons from metal ions to ligand or vice versa are called Charge Transfer Transitions, which are highly intense. The intense colour of $[(C_{12}H_8N_2)_3Fe]^{2+}$ is due to this type of transitions. The ligand is a weak base that reacts to form phenanthrolinium ion, phen H^+, in acidic medium. Accordingly, the complex format ion may be represented as follows,

$$Fe^{2+} + 3\ PhenH^{+} = Fe\ (Phen)_3^{2+} + 3H^{+}$$

Characterization of *Hibiscus rosa sinensis* leaves extract

GC-MS Technique

The GC-MS analysis detected all organic species quantitatively. Each peak area in the chromatogram (Figure 7) was proportional to the amount of the organic compounds forming that peak.Structural assignment of GC retention data of compounds is based on spectral matching with NIST library (National Institute of Standards and Technology).It can be observed that the *Hibiscus rosa sinensis* leaves extrct consist of 7 main compounds (Table 4).

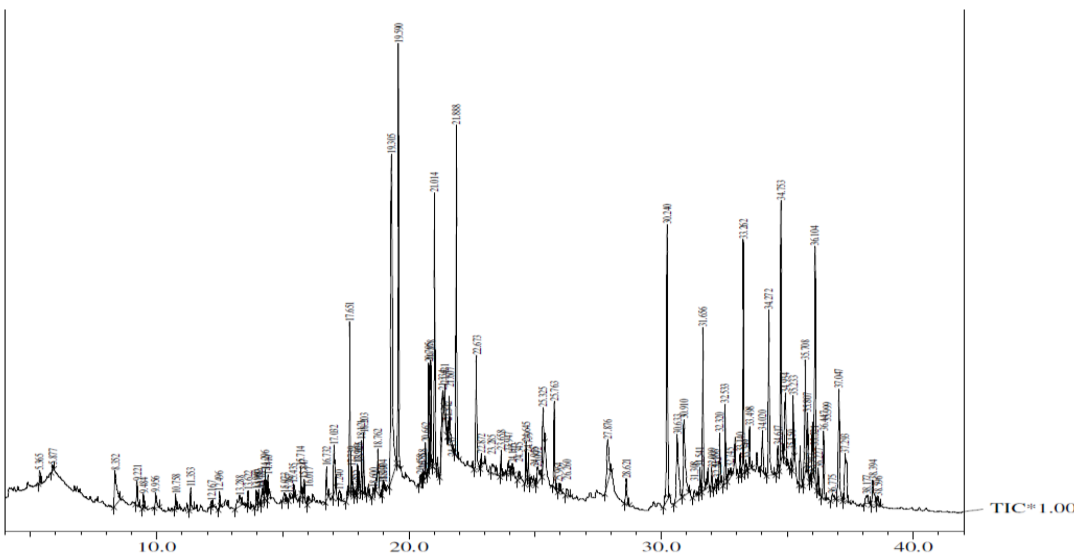


Figure 7: GC-MS Chromatogram obtain for the *Hibiscus rosa sinensis* leaves extract under the defined conditions

Table 4: Data of Main Compounds

Peak#	R. Time	Area	Area%	Name
1	21.014	7469267	3.70	2-Hexadecen-1-OL,3,7,11,15-Tetramethyl-, [R-[R*,R*-(E)]]
2	21.888	8445787	4.19	Octadecanoic Acid, Ethyl ester
3	34.753	8442530	4.19	Stigmast-5-EN-3-OL, (3. Beta)
4	36.104	8750396	4.34	Lup-20(29)-EN-3-YL Acetate
5	30.240	10602031	5.26	2,6,10,14,18,22-Tetracosahexaene, 2, 6, 10, 15, 19, 23-hexamethyl
6	19.590	10673100	5.29	Hexadecanoic Acid , Ethyl ester
7	19.305	17927204	8.89	Phthalic acid, butylundecylester

The peaks belong to the2-Hexadecen-1-OL,3,7,11,15-Tetramethyl-,Octadecanoic Acid, Ethyl ester, Stigmast-5-EN-3-OL, Lup-20(29)-EN-3-YL Acetate, 2, 6, 10, 14, 18, 22-Tetracosahexaene, 2, 6, 10, 15, 19, 23-Hexamethyl, Hexadecanoic Acid , Ethyl ester, Phthalic Acid, Butyl Undecyl ester. Since retention time of majority of compounds is close to each other and it is very difficult to separate them.

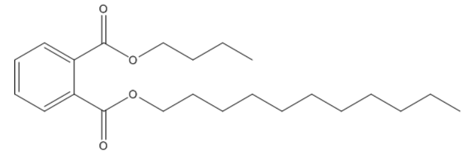
Mechanism of inhibitor

Generally the first stage in the corrosion inhibition mechanism is the adsorption of inhibitor molecules on the mild steel surface. The process of adsorption is influenced by the type of the aggressive electrolyte, the chemical structure of the inhibitor molecules, and the nature and charge of the metal. The charge on the metal surface is due to the electric field generated at the metal/electrolyte interface[22].

Once the inhibitor has adsorbed on the metal surface it can then affect the corrosion reactions in a number of ways: by offering a physical barrier to the difussion of ions or molecules to or from the metal surface, direct blocking of anodic and/or cathodic reaction sites, interaction with corrosion reaction intermediates, change the make-up of the electrical double layer which develops at the metal/solution interface and so affect the rate of electrochemical reactions. Adsorption could proceed via sharing of the donor group electrons like –OH and/or aromatic π-electrons, between the inhibitor molecules and the partially filled d-orbitals of iron.

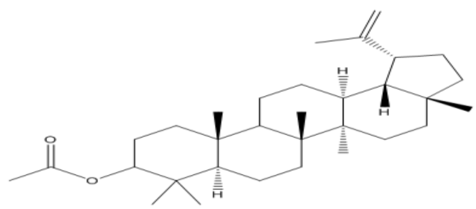
The exact nature of the interactions between a metal surface and an aromatic molecule depends on the relative coordinating strength towards the given metal of the particular groups present. The main constituents of extract of *Hibiscus rosa sinensis* leaves extract are 2-Hexadecen-1-OL, 3, 7, 11, 15–Tetramethyl-, Octadecanoic Acid, Ethylester, Stigmast-5-EN-3-OL, Lup-20(29)-EN-3-YL Acetate, 2, 6, 10, 14, 18, 22-Tetracosahexaene, 2, 6, 10, 15, 19, 23-Hexamethyl, Hexadecanoic Acid , Ethyl ester, Phthalic Acid, Butyl Undecyl ester whose structures are given in Figure 8.

In HCl medium the inhibitor molecules may adsorb through protonated hetero atoms (N, O, and S) and already adsorbed Cl^- ions on the mild steel surface. Initially the protonated forms of inhibitor molecules in acidic medium compete with H^+ ions for electrons on the mild steel surface. After the evolution of H_2 gas from HCl, the cationic form of inhibitors returns to its neutral form and hetero atoms with lone pair electrons promote chemical adsorption. The high electron density on the mild steel surface renders more negative charge to it. Aromatic compounds (which contain the benzene ring) undergo particularly strong adsorption on many electrode surfaces. The bonding can occur between metal surface atoms and the aromatic ring of the adsorbate molecules or ligands substituent groups such as –OH and -OCO- present in the main components of leaves extract of hibiscus.

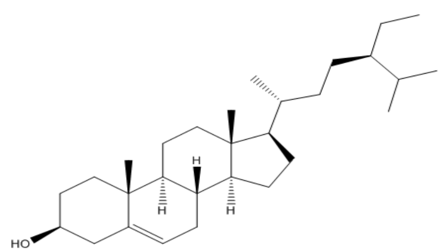


Phthalic acid butyl undecyl ester

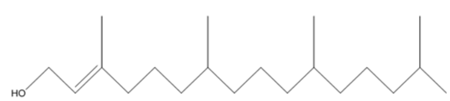
Hexadecanoic acid ethyl ester



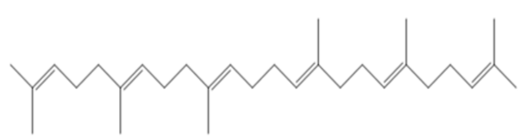
LUP-20(29) -EN-3-yl acetate



Stigmast-5-En-3-ol,3 beta



2-Hexadecen-1-ol,3,7,11,15-tetramethyl



2,6,10,14,18,22-tetracosahexaene 2,6,10,15,19,23-hexamethane

Octadecanoic acid ethyl ester

The high performance of *Hibiscus rosa sinensis* leaves extract could also be due to large size of constituent's molecule which covers wide areas on the metal surface and thus retarding the corrosion. It is not possible to consider a single adsorption mode between inhibitor and metal surface because of the complex nature of adsorption and inhibition of a given inhibitor. Chemical constituents include alkaloids, flavonoids, terpenoids, glycosides, tannins, saponins, fats and oils and carbohydrates.

Conclusion

Hibiscus rosa sinensis leaves extract acted as efficient inhibitor for the corrosion of Mild Steel in 2 M HCl and exhibited 87.23% efficiency at concentration of 1.086%v/v by weight loss method. Visible Spectrophotometry method obtains ferrous ion concentration, as ferrous ion concentration decrease inhibition efficiency increases. Visible Spectrophotometry results show that 72.60% inhibition efficiency at 1.086%v/v concentration of inhibitor. The adsorption follows Langmuir, Freundlich and Temkin isotherm.

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