Chapter 4
Materials and Methods

4.1. Experimental Work

This chapter mainly deals with the methodology adopted for the plant extractions **(aqueous and alcoholic)** and evaluation of corrosion inhibition of plant extracts as green inhibitor on mild steel in 1N HCl medium.

4.2. Objectives

The present research is undertaken to:

1. Study of efficacy of acid extract of the selected plants as corrosion inhibitor for mild steel in 1N HCl by weight loss and electrochemical methods and fit a suitable adsorption isotherm for the thermostat experimental data.
2. To compare the results obtained by various methods.
3. To determine which part of the plants, provide better inhibition.
4. To analyse the surface of substrate by FTIR and SEM techniques.

4.3. Mild steel specimens

Weight loss and electrochemical experiments were conducted on mild steel specimens of dimensions 4 cm X 2 cm X 0.1 cm and having the area of 1 cm^2 for 24 hours having the composition used throughout the present investigation.

Table 3. Composition of mild steel

Elements	C	Mn	Si	P	S	Cr	Mo	Ni	Cu	Fe
Wt %	0.030	0.169	0.015	0.031	0.029	0.029	0.016	0.030	0.017	Bal.

4.4. Preparation of inhibitor

Aqueous extract preparation

The plants are organic in nature and are much used as a **medicinal plant in ayurvedic medicine, mainly; it helps against disease of the digestive tract and fever**. The leaves, flower, (roots), stem, tubers, fruits, (seeds) of the medicinal plants **AL, SS, HI, ML, GSL and PD** were taken and cut into small pieces, and dried in

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room temperature and ground well into powder. 150 g of the powder from each was refluxed in 500 ml distilled water and kept overnight. The refluxed solution was then filtered carefully; the filtrate volume was made up to 1000 ml using double distilled water which was the stock solution. Various concentrations of the plant extracts were prepared by dissolving the known quantity of the resultant powder in acid media. The concentrations of all additives were expressed in v/v. Thus the concentration (5, 10, 15, 20 v/v) was prepared by diluting 5 ml, 10 ml, 15 ml 20 ml of plant extract with 95 ml, 90 ml, 85 ml, 80 ml of HCl acid solution [1-6].

Alcoholic extract preparation

The **leaves, flower, (roots), stem, tubers, fruits, (seeds, seeds peels)** of the medicinal plants ***AL, SS, HI, ML, GSL,*** and ***PD*** were taken and cut into small pieces, and dried in room temperature and ground well into powder. 150 g of the powder from each was refluxed with alcohol in soxlet apparatus for 24 hours kept overnight. The refluxed solution was filtered carefully and the filtrates were heated on water both to evaporate fully the moisture or impurities content and the excess of alcohol was removed by distillation method. The resulting residue was dark green in colour and had a pleasant smell. The crude was boiled with little quantity of activated charcoal to removes impurities; the pure plant extract after drying completely was collected and stored in a desiccator. The residue was used in preparing different concentration of the extract in HCl acid.

4.5. Reason for selection of acid medium

- Corrosion in acidic medium is more commonly encountered in industry than neutral or alkaline medium.
- Due to tremendous increase in industrial activities, HCl are increasingly used in acidic pickling and descaling. So, our experiments are performed in 1N HCl.

4.6. Reagents used

Acids: For this study Analar grade Hydrochloric acid was used and for the preparation of 1N HCl double distilled water was used.

4.7. Equipment

- Electronic balance-SHIMADZU BY-220 S
- Bruker alpha 8400 S FT-IR spectrophotometer
- Electrochemical work station CHI model 660 E Amp Booster & CHI 608 D
- JEOL computer-SEM

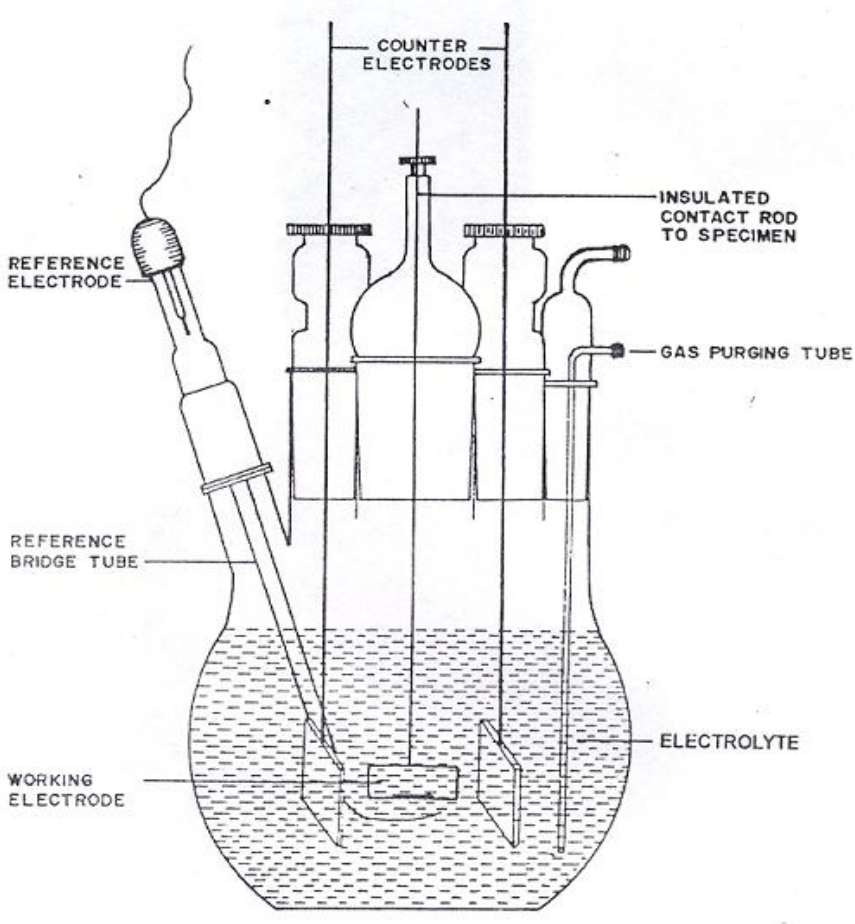


Fig. 16 Electrochemical cell assembly

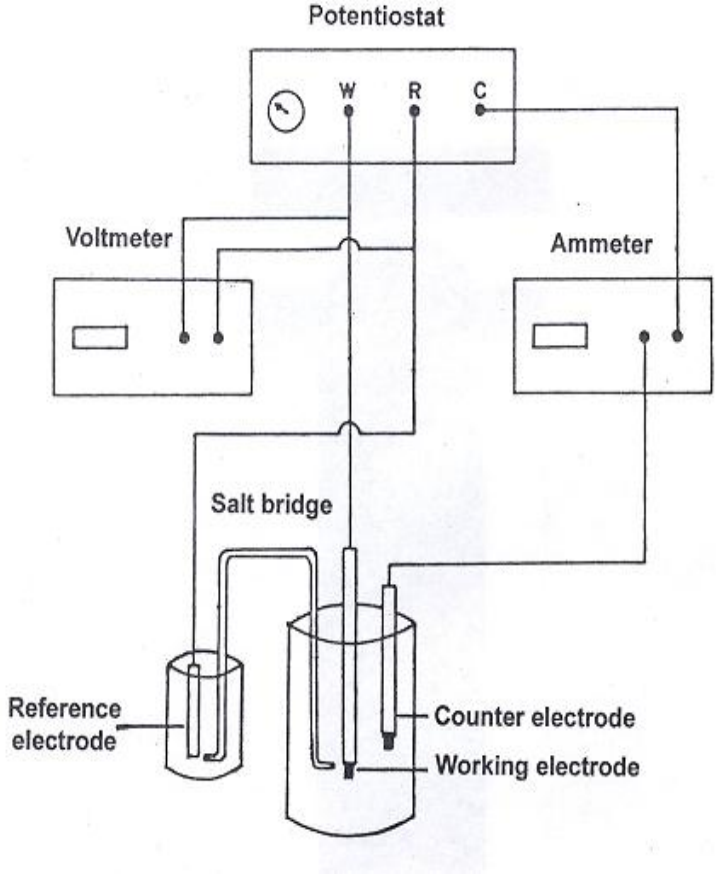


Fig. 17 Schematic representation of the circuit

4.8. Working plan

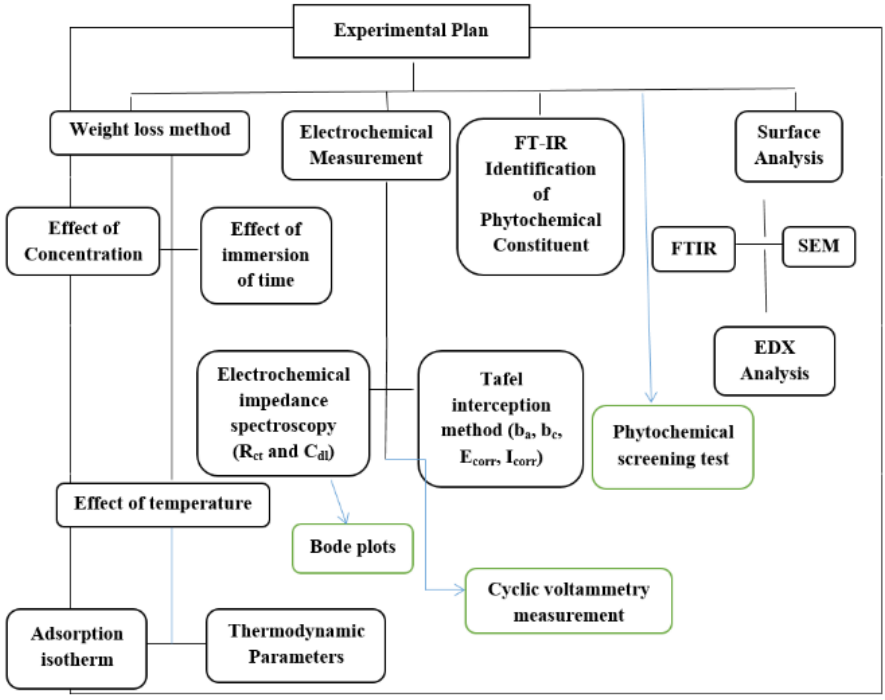


Fig. 18 Work plan for various methods

METHODS

4.9. Weight loss method

Weight loss analysis is one of the basic, easiest and frequently used methods and classical way to determining corrosion inhibition and corrosion rate of mild steel. Coupons were completely immersed vertically in 250 ml of the test solution with and without inhibitor for 24 hours. The experiment was carried out at various immersion period (1, 3, 5, 7, 12 hrs and 3 days) and corrosion inhibition studies were also carried out at various temperature ranges at 303-323 K. From the mass loss measurements, the corrosion rate was calculated by using the following relationship:

$$\text{CR (mmpy)} = \frac{\text{K x Weight loss}}{\text{D x A x t (in hours)}} \qquad (1)$$

Where, K = 8.76×10^4 (constant), D is density in gm/cm^3 (7.86), W is weight loss in grams and A is area in cm^2.

$$IE\ \% = \frac{W_0 - W_i}{W_0} x100 \quad (2)$$

Where, W_0 and W_i are the weight loss with and without of the inhibitor.

4.10. FT-IR spectra

FT-IR spectroscopy allows us to examine the molecular structure and confirmation of biological macro molecules because it measures the absorption energy, which produces an increase in the vibrational or rotational energy of atoms or groups of atoms within the molecules. FT-IR spectrum (KBr pellets) of the surface film was recorded using Bruker alpha 8400 S spectrophotometer in the wave number range of 4000- 400 cm^{-1}.

4.11. Electrochemical studies

Electrochemical (Polarization and impedance) measurements were obtained using CHI 660 E Electrochemical workstation. An electrochemical cell with a three electrode cell set up was used. Mild steel ($1cm^2$) was used as a working electrode; Pt electrode was used as counter electrode, and a saturated calomel electrode was used as reference electrode. The working electrode was polished with 1/0, 2/0, 3/0 and 4/0 grade emery papers and washed with distilled water before usage. Prior to experiment the working electrode was immersed in the test solution for 20 minutes to reach open circuit potential (OCP). The anodic and cathodic polarization curves were obtained from -800 to -200 mV at a scan rate of 1 mVs^{-1}. The percentage inhibition efficiency was calculated by using this equation:

$$IE\ \% = \frac{I_{Corr} - I_{*Corr}}{I_{Corr}} x\ 100 \quad (3)$$

Where, I_{corr} and I_{*corr} are corrosion current without and with inhibitors.

Impedance spectroscopy is one of the most simple and consistent techniques and also used to study the characterization of electrode (surface) behavior in 1N HCl solution. AC signal with amplitude of 10 mV at OCP in the frequency range from 100 KHz to 10 MHz. The impedance parameters were obtained from Nyquist plots. The double layer capacitance (C_{dl}) was determined using formula:

$$C_{dl} = \frac{1}{2\pi} f_{max} R_{ct} \quad (4)$$

Where, R_{ct} is charge transfer resistance, and C_{dl} is double layer capacitance.

The percentage inhibition efficiency (IE%) was calculated by using the following formula:

$$IE\% = \frac{R_{ct} - R_{ct}^0}{R_{ct}}\ x\ 100 \quad (5)$$

Where, R_{ct} and R^0_{ct} are the charge resistance values for inhibited and uninhibited solution.

4.12. Surface Analysis

The mild steel specimens used for surface morphology examination were immersed in 100 ml of 1N HCl acid (blank solution) containing (optimum) various concentrations of green inhibitor for a period of one day. Then, they were removed, rinsed quickly with double distilled water, dried and examined for their surface morphology using scanning electron microscope.

4.13. Cyclic Voltammetry Measurement

Cyclic voltammetry method was carried out with CHI-660 E Electrochemical analyzer. The experiments were carried out in conventional three electrode cell assembly as that for cyclicvoltammetry. The plants extract was performed in acidic medium. The cyclic voltammograms (CVs) corresponding to 10 cycles of plant extract in 1 N HCl acid, during the sequential potential range between – 0.4 v to 1.5 v at a scan rate of 10 mVs^{-1}.

4.14. Phytochemical Screening

Phytochemical screening was carried out on the aqueous and alcoholic extract freshly prepared according to the common Phytochemical method described earlier by **Harborne** [7]. The different chemical constituents were tested which included Alkaloids, Terpinoids, Sterols, Triterpenes, Anthraquinones and Flavonoids etc.

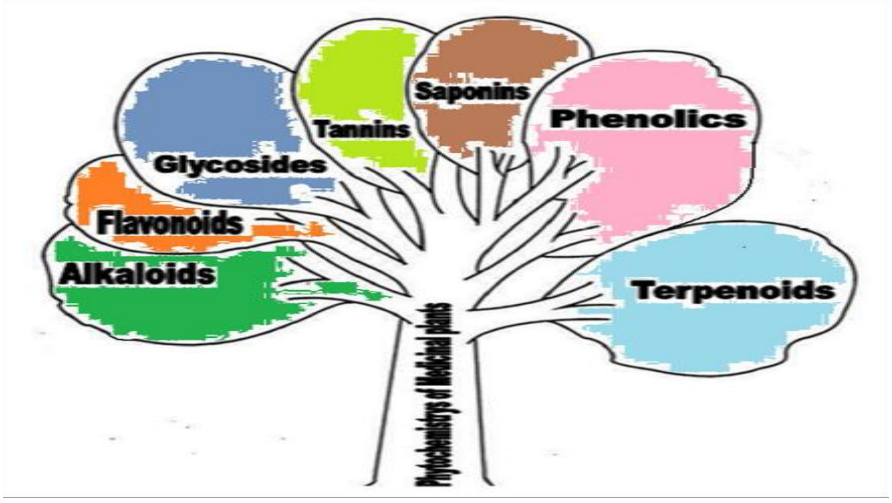


Fig. 19 Various test for phytochemical screening test.

4.15. Description of plants

In view of the advantages of green corrosion inhibitors we had explored the plant species belonging to various families. Our research on these families had resulted in the report of alkaloids and terpinoids as anti-corrosive agents. In continuation of our study on green corrosion inhibitors, few plants have been chosen in view of the reports of presence of heterocyclic constituents particularly alkaloids. Thus, the study is aimed at screening the chosen species of plants belonging to these few families for their anti-corrosion effects and the mechanism of action of these plant extracts by standard methods.

Literature survey of various plant sources belonging to Gloriosa Superba Linn, Madhuca longifolia, Alangium lamarckiii, Holoptelea integrifolia, Pithecellobium dulce and Schreabera swietenioids were chosen for the study.

i) Madhuca longifolia (ML)
ii) Gloriosa Superba Linn (GSL)
iii) Alangium lamarckiii (AL)
iv) Holoptelea integrifolia (HI)
v) Pithecellobium dulce (PD)
vi) Schreabera swietenioids (SS)

4.16. Madhuca Longifolia (ML)

Madhuca Longifolia (ML) is a tree found largly in the central and north Indian plains and forest. ***Tamil name: iluppai.*** It is fast growing up to 20 m height. The seeds, flowers and wood are used for the care of the skin, to manufacture of soap or detergents, and as a vegetable butter. The saying ***"aalai illaa oorukku iluppai poo charkkarai"*** indicates when there is no cane sugar available; the flowers of this plants can be used as it is very sweet. Several part of the tree is used for various purposes including the bark, flowers for their medicinal properties.

4.17. Glorisa Superba Linn (GSL)

The Gloriosa Superba Linn (GSL) is the state flower of Tamil Nadu. This species is the national flower of Zimbabwe. ***Tamil name: Chengkanthal.*** In general this plant is common worldwide and used both as medicinal and poison. It is a fast growing plant up to 6 m long. It has been used in the treatment of snake bites, kidney problems, cancer, sexually transmitted diseases and many types of internal parasites. The plant can be

propagated sexually by seeds or vegetative by dividing the rhizomes. Both the fruit and the rhizome are harvested. Other uses of this plant include arrow poison in Nigeria and snake repellent in India.

4.18. Pithecellobium Dulce (PD)

Pithecellobium Dulce (PD) is a species of flowering plants in the Fabaceae family. ***Tamil name: Kodukkapuli.*** That tree reaches a height of about 10 to 15 m. In Mexico it is eaten raw as an accompaniment to various meat dishes and used as a base for drink with sugar and water. The seeds are also refined to extract oil, which amount to 10 % of their weight. Barks extract also used against dysentery, chronic diarrhoea and tuberculosis. The ground seed is used to clean ulcers.

4.19. Alangium Lamarckiii (AL)

Alangium Lamarckiii (AL) commonly known as sage leaved alangium is a flowering plant in the alngiaceae family. ***Tamil name: Alinsol.*** In Ayurveda the roots and fruits are used for the treatment of rheumatism and haemorrhoid. The bark is also used in traditional medicine for skin problems and as an antidote for snake bite. It's used as an emetic and purgative and toothbrush. It's considered to be good for making musical instruments and for making furniture.

4.20. Holoptela Intergrifolia (HI)

Holoptelea Integrifolia (HI) has been found to be large deciduous tree to 25 m height. ***Tamil name: Aaya.*** Leaves simple alternate stipules lateral, scarious petiole 5-10 mm. flowers, bark, seeds are used as medicine. Children eat seeds.

4.21. Schreabera Swietenioids (SS)

Schreabera Swietenioids (SS) is a species of plant in the oleaceae family in the Himalayas, India. ***Tamil name: Mahalingam maram***. It has many seeds, flats, winged. Fruting March onwards, almost persistent. Bark, roots, leaves and fruits are used as medicine. These are also used as a remedy for cancer.

Fig. 20 Phytochemicals screening in plant extract

4.22. The standard procedure for phytochemical tests shows the following results.

1. TEST FOR ALKALOIDS:
   Wagner's test: The plant extract was dissolved in chloroform. The chloroform layer was evaporated, and the residue was acidified to which was added few drops of Wagner's reagent (iodine in potassium iodide). Orange red precipitate was observed.
2. TEST FOR SAPONINS:
   Foam test: 1 ml of the plant extract was shaken with 2 ml of water. The foam was produced within 10 minutes, which indicates the presence of saponin.
3. TEST FOR FLAVONIODS:
   Neutral $FeCl_3$ test: To a small quantity of the alcoholic solution of the extract,
   a few drops of neutral $FeCl_3$ solution were added. A green colour was formed which shows the presence of flavonoids.
4. TEST FOR COUMARINS:
   To 1 ml of the plant extract, 1 ml of 10 % NaOH was added. The presence of coumarinsis was indicated by the formation of yellow colour.
5. TEST FOR TANNINS:
   Lead acetate test: To 5 ml of the extract a few drops of 1 % solution of lead acetate was added. Formation of yellow precipitate indicates the presence of tannins.
6. TEST FOR PROTEINS:
   Bireut test: To 1 ml of the extract a few drops of $CuSO_4$ and 1 ml of NaOH were added. Formation of violet colour shows the presence of protein.

7. TEST FOR ANTHROCYANINS:
   To the extract 10 % NaOH was added. No characteristic change was noticed. This shows the absence of anthrocyanins.
8. TEST FOR QUINONES:
   To 1 ml of the extract,1 ml of conc. H_2SO_4 was added, no characteristic change took place. This proves the absence of quinones.
9. TEST FOR ANTHRAQUINONES:
   To 1 ml of plant extract, 2 ml of NH_4OH was added, no characteristic change. Absence of anthroquinones.
10. TEST FOR CARBOHYDRATES:
    Molisch test: To the extract molisch reagent conc. H_2SO_4 was added. Formation of violet colour shows the presence of carbohydrates.

4.23. Properties and application

Plants have long been used as food and a source of lamp oil, but now, it serves as a raw material of biological and paramedical products. The plant contain high concentration of glycosides, alkaloid, phenols, amine, flavonoids, saponins, carbohydrates, steroids, proteins and amino acids and tannings which have fused benzene ring and heteroatom in the ring. Medicinal plant considered an antipyretic, tonic, antidiabetic, immunomodulatory, anti-inflammatory, diuretic, vermifuge, antimicrobial, antihyperlipidemic, anticarcinogenic, antioxidant, anthelmintic, cytotoxic activities etc. due to presence of a variety of active phytochemicals have been reported in the literature [8-16]. In fact, the ***first patented corrosion inhibitors used were either natural product such as flour, yeast*** etc. Later, interest in using natural products as corrosion inhibitors increased substantially and scientists around the world reported several plant extracts as promising green anticorrosive agents.

4.24. Phytochemical screening test

In view of its phytochemical investigation, it was very clear that the presence of phytochemicals is responsible for the anticorrosion action. The screening test was carried out on a freshly prepared extract and various chemical constituents are presented in ***Tables 4-9***. The result indicates that the natural product (alkaloids, tannis, steroids, terpinoids) absolutely inhibited the corrosion product on the metal surface. The possible reason for the absence or presence of these compounds in our test could be a very little amount and/or non-extraction of these compounds using aqueous and alcoholic solution of HCl, because they are below the analytical detection limit [17-26].

Inspection of the chemical structure of these phytochemical constituent revealed that these compounds are easily hydrolysable and the compounds can be adsorbed on the metal surface via the lone pair of electron present in their nitrogen and oxygen atoms (i.e they contain multifunctional group such as OH, NH, CO, COOH as well as O heterocyclic atom) which are barriers for charge and mass transfer leading to decrease the interaction of the metal with the corrosive environment. As a

result, the corrosion rate of the metal was decreased. The formation of film layer essentially blocks the discharge of H^+ and dissolution of the metal ion. Due to electrostatic interaction, the protonated constituent's molecules are adsorbed (physisorption) and high inhibition is expected.

Table 4 Phytochemical screening test of extract of Madhuca Longifolia (ML) plant

	Aqueous extract				Alcoholic extract			
Phytochemical test	**Leaves**	**Fruits**	**Barks**	**Seed peel**	**Leaves**	**Barks**	**Fruits**	**Seed peel**
Alkaloids	+	+	+	+	+	+	-	-
Carbohydrates	+	+	-	-	+	+	+	+
Proteins	+	+	+	-	-	+	+	+
Saponins	-	-	+	+	+	-	-	-
Thiols	+	-	-	-	-	+	-	-
Tannins	-	-	+	-	+	-	+	-
Flavanoids	+	+	-	+	-	+	-	+
Phenol	+	+	+	-	+	+	+	-
Glycosides	-	+	+	+	-	-	-	-

(+).. Presence (-)... Absence

Table 5 Phytochemical screening test of extract of Gloriosa Superba Linn (GSL) plant.

	Aqueous extract				Alcoholic extract			
Phytochemical test	**Leaves**	**Flowers**	**Tubers**	**Stem**	**Leaves**	**Stem**	**Flowers**	**Tubers**
Alkaloids	+	+	+	+	+	+	+	+
Carbohydrates	+	+	+	-	-	-	-	+
Proteins	+	+	+	-	+	+	+	+
Saponins	-	-	+	+	-	+	+	-
Thiols	-	-	-	-	-	-	-	-
Tannins	-	-	+	-	+	-	+	+
Flavanoids	-	+	+	+	+	+	+	+
Phenol	+	+	+	-	+	-	-	+
Glycosides	-	+	+	+	-	-	-	-

(+).. Presence (-)... Absence

Table 6 Phytochemical screening test of extract of Pithecellobium Dulce (PD) plant.

Aqueous extract					**Alcoholic extract**			
Phytochemical test	**Leaves**	**Fruits**	**Barks**	**Seeds**	**Leaves**	**Fruits**	**Barks**	**Seeds**
Alkaloids	+	+	+	+	+	+	+	+
Carbohydrates	+	+	-	-	+	-	-	-
Proteins	+	+	+	+	-	-		+
Saponins	-	-	+	+	-	+	-	+
Thiols	+	-	-	-	+	-	-	-
Tannins	-	-	-	-	-	-	-	-
Flavanoids	-	+	-	-	-	+	-	-
Phenol	+	+	+	-	+	-	+	+
Glycosides	-	+	+	+	-	+	-	+

(+).. Presence (-)... Absence

Table 7 Phytochemical screening test of extract of Alangium Lamarckiii plant.

Aqueous extract					**Alcoholic extract**			
Phytochemical test	**leaves**	**Barks**	**Fruits**	**Seeds**	**Leaves**	**Barks**	**Fruits**	**Seeds**
Alkaloids	+	+	+	+	+	+	+	+
Carbohydrates	+	-	-	-	+	-	+	+
Proteins	+	-	+	+	+	+	-	+
Saponins	-	-	-	+	-	+	+	-
Thiols	-	-	-	+	+	-	-	+
Tannins	-	-	+	-	-	+	-	-
Flavanoids	-	-	-	+	-	-	+	+
Phenol	+	+	+	-	+	+	+	-
Glycosides	-	-	+	+	+	-	-	+

(+).. Presence (-)... Absence

Table 8 Phytochemical screening test of extract of Holoptelea Integrifolia (HI) plant.

Aqueous extract					**Alcoholic extract**			
Phytochemical test	**Leaves**	**Seeds**	**Flowers**	**Barks**	**Leaves**	**Seeds**	**Flowers**	**Barks**
Alkaloids	+	+	+	+	+	+	+	+
Carbohydrates	+	+	-	-	+	-	+	+
Proteins	+	+	+	-	+	+	+	-
Saponins	-	-	+	+	-	-	+	-
Thiols	+	-	-	-	-	_	-	+
Tannins	-	-	-	-	-	-	-	-
Flavanoids	-	-	+	+	-	+	+	-
Phenol	+	+	+	-	+	+	-	+
Glycosides	+	-	-	+	-	-	+	-

(+).. Presence (-)... Absence

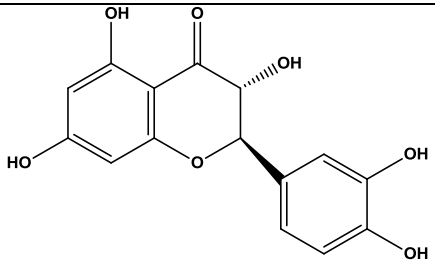
Table 9 Phytochemical screening test of extract of Schreabera Swietenioids (SS) plant.

	Aqueous extract				Alcoholic extract			
Phytochemical test	**leaves**	**fruits**	**seeds**	**barks**	**leaves**	**fruits**	**Seeds**	**barks**
Alkaloids	+	+	-	+	+	-	+	+
Carbohydrates	+	-	-	-	+	-	-	-
Proteins	+	-	+	-	+	+	+	-
Saponins	-	-	+	+	-	-	-	-
Thiols	-	-	-	-	-	-	-	-
Tannins	-	-	-	-	-	-	-	-
Flavanoids	-	-	+	-	-	-	-	+
Phenol	+	+	+	+	+	+	+	+
Glycosides	-	-	-	+	-	-	+	-

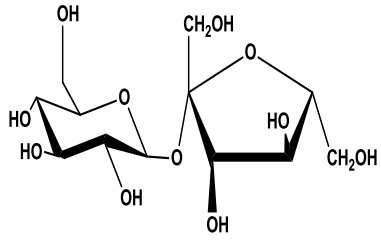
(+).. Presence (-)... Absence

Gas chromatography – mass spectroscopy examination identified all organic species quantitatively, and can be detected that all species consists of possible major compounds [27-31]. Since holding time of majority of composites is close to each other and it is very difficult to separate them, the plant extract as such was used for corrosion inhibition studies. The following component with structure contains electron rich oxygen and nitrogen that can be served as good active ingredients which are responsible for corrosion inhibition in plants.

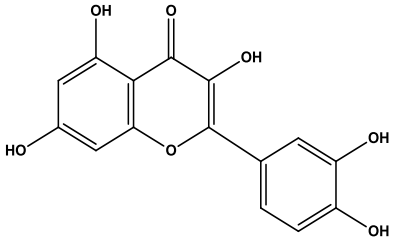
Madhuca Longifolia (ML)



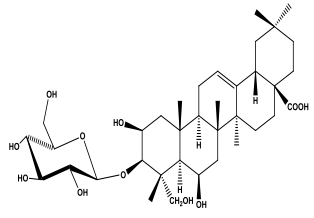
Taxifolia



Sucrose



Quercetin



3-o-β-D-Glacopyrasosylprofobassic acid

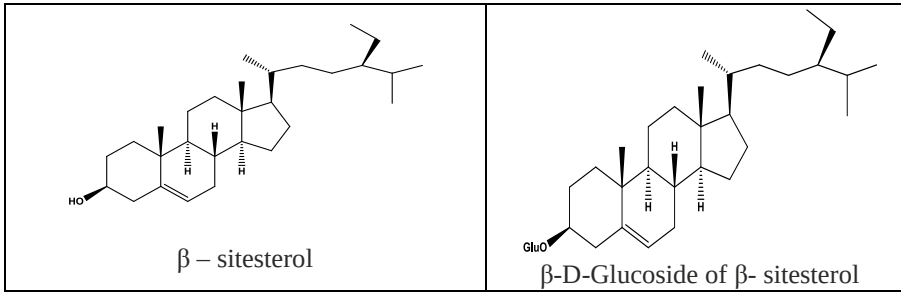


Fig. 21 The chemical molecular structure of major constituents of ML plants extract

Gloriosa Superba L (GSL)

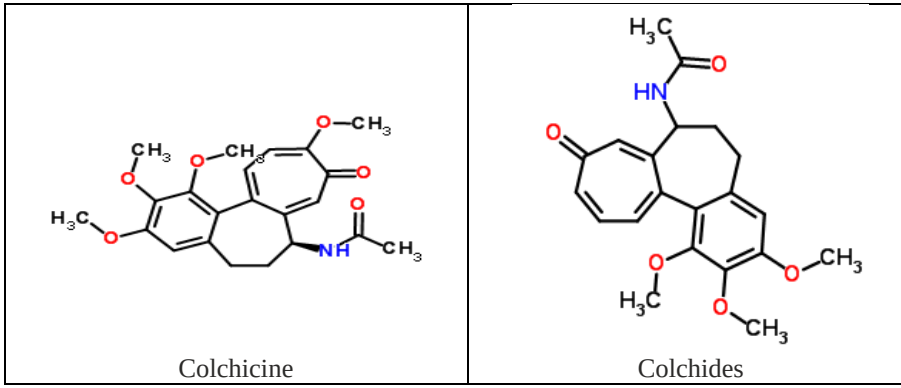


Fig. 22 The chemical molecular structure of major constituents of GSL plants extract

Pithecellobium Dulce (PD)

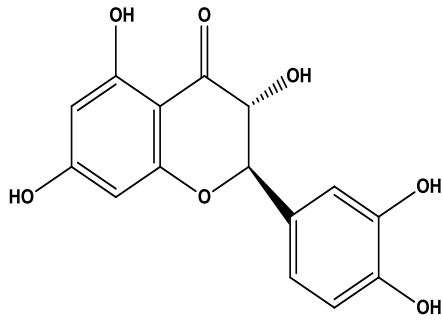


Fig. 23 The chemical molecular structure of major constituents of (Quercetin) PD plants extracts.

Alangium Lamarckiii (AL)

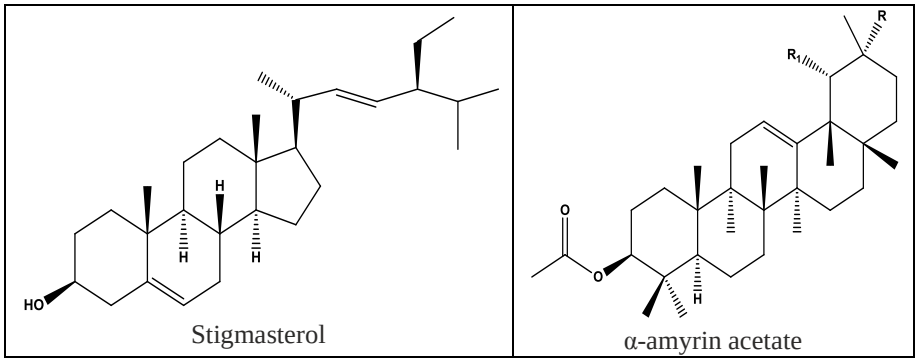


Fig. 24 The chemical molecular structure of major constituents of AL plants extract

Schreabera Swietenioids (SS)

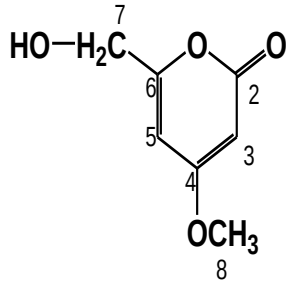
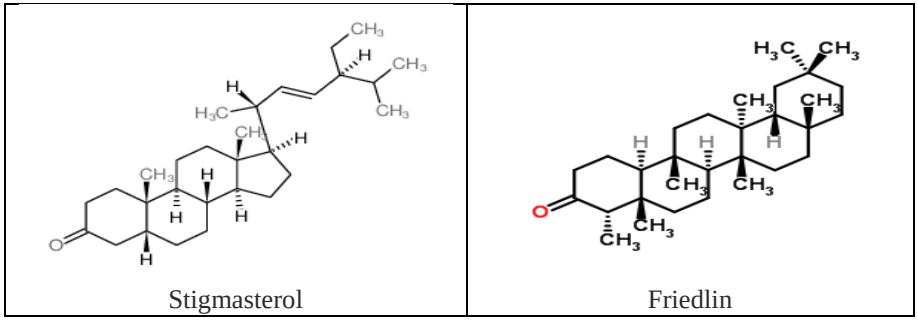


Fig. 25 The chemical molecular structure of major constituents (opuntiol) of SS plants extract

Holoptelea Integrifolia (HI)



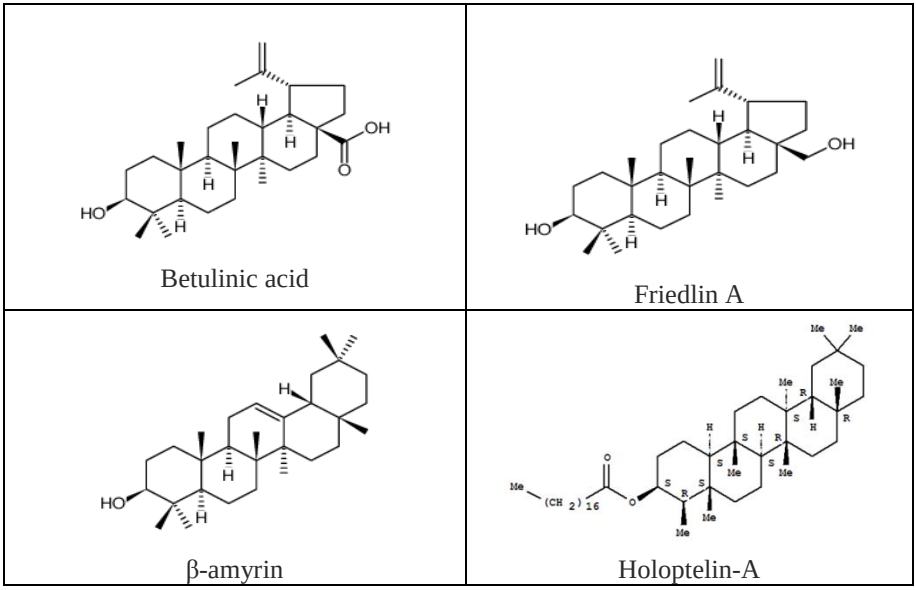


Fig. 26 The chemical molecular structure of major constituents of HI plant extracts

From the figure of constituent, it is clear that these constituents are having pi bonds and heteroatom (oxygen). Therefore, the adsorption process occurs either by the electrostatic interaction in charged molecules and constituents of inhibitors or by the interaction of unshared electrons of inhibitor molecules. The adsorption of the protonated molecules in the plants extract also occurs on the mild steel surface by direct interaction of the lone pairs of electrons on O and N with the vacant d- orbitals of Fe. These lone pair of electrons could also from metal inhibitor complex with Fe^{2+} ions formed from the anodic dissolution of steel. These complexes may adsorb onto the steel surface through Vander Waals force, providing more protection against corrosion [32-36]. In this present study, our result shows that due to lack of isolating these compounds, it is not possible to determine what components presents in plant extract created their relatively high ability to inhibit corrosion.

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