

**IRESINE HERBSTII AS GREEN INHIBITOR FOR  
MILD STEEL CORROSION IN 1 M H<sub>2</sub>SO<sub>4</sub> SOLUTION**



**A MINI RESEARCH REPORT SUBMITTED TO  
OFFICE OF DEAN  
INSTITUTE OF SCIENCE AND TECHNOLOGY  
TRIBHUVAN UNIVERSITY  
KATHMANDU, NEPAL**

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**Falgun, 2080**

## **CERTIFICATE OF APPROVAL**

This mini research report entitled "**IREFINE HERBSTII AS GREEN INHIBITOR FOR MILD STEEL CORROSION IN 1 M H<sub>2</sub>SO<sub>4</sub> SOLUTION**" by Naresh Prashad Pant, Department of Chemistry, Amrit Campus, Institute of Science and Technology (IoST), Tribhuvan University (T.U.), is hereby submitted has been accepted by institute of science and technology research committee.

# RECOMMENDATION LETTER


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## LETTER OF CONSENT

This mini research report entitled "TRESINE HERBSTII AS GREEN INHIBITOR FOR MILD STEEL CORROSION IN 1 M H<sub>2</sub>SO<sub>4</sub> SOLUTION" by Naresh Prashad Pant, Department of Chemistry, Amrit campus, Institute of Science and Technology (IoST), Tribhuvan University (T.U.), is hereby completed under my mentorship. This is novel work and has been performed genuinely and good ethics of research. This research is useful in the estimation of minerals in Nepal and expected to work in policy design. This work is performed under support of Mini Research Grant-2080 provided by Dean's Office, Institute of Science and Technology, Tribhuvan University.



  
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## DECLARATION

I, Naresh Prashad Pant, hereby declare that the Mini Research Report entitled “**IREFINE HERBSTII AS GREEN INHIBITOR FOR MILD STEEL CORROSION IN 1 M H<sub>2</sub>SO<sub>4</sub> SOLUTION**” is being submitted to the office of Dean, Institute of Science and Technology (IoST), Tribhuvan University (T.U.), Nepal. The research work is carried out by me and all the proposed goal in the proposal has been achieved and have been included in this report.

The work is original and has not been submitted earlier in part or full in this or any other form to any university or institute, here or elsewhere, for the award of any degree.



.....  
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**January 2023**

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**Naresh Prashad pant**

**January 2024**

## ABSTRACT

The study examined the methanol extract obtained from *Irsine herbstii* stem as a green inhibitor for mild steel corrosion in a 1M H<sub>2</sub>SO<sub>4</sub> environment. Characterization of the extract was carried out using Fourier Transform Infrared Spectroscopy and potentiostat. Additionally, inhibition efficiency was determined through both weight loss and electrochemical measurement methods, with results indicating a maximum inhibition efficiency of 87.28%. Thermodynamic parameters further confirmed the feasibility and spontaneity of the adsorption process. Overall, the findings underscored the potential effectiveness of the inhibitor derived from *Irsine herbstii* alkaloid extract in mitigating mild steel corrosion in acidic environments.

Keywords: *Irsine herbstii*, solvent extraction, green inhibitor, phytochemicals, weight loss

## शैधसँर

अध्ययनले 1M H<sub>2</sub>SO<sub>4</sub> वातावरणमा हल्का स्टीलको क्षरणको लागि हरियो अवरोधकको रूपमा Irsine herbstii स्टेमबाट प्राप्त मेथानोल निकासीको जाँच गन्यो। फुरियर ट्रान्सफर्म इन्फ्रारेड स्पेक्ट्रोस्कोपी र पोटेंटियोस्टेट प्रयोग गरेर एक्स्ट्र्याक्टको क्यारेक्टराइजेशन गरिएको थियो। थप रूपमा, निषेध दक्षता दुवै वजन घटाने र इलेक्ट्रोकेमिकल मापन विधिहरू मार्फत निर्धारण गरिएको थियो, परिणामहरूले अधिकतम अवरोध दक्षता ८७.२८ प्रतिशत संकेत गर्दछ। थर्मोडायनामिक प्यारामिटरहरूले सोखन प्रक्रियाको सम्भाव्यता र सहजतालाई थप पुष्टि गन्यो। समग्रमा, निष्कर्षहरूले अम्लीय वातावरणमा हल्का स्टीलको क्षयलाई कम गर्न Irsine herbstii alkaloid एक्स्ट्र्याक्टबाट व्युत्पन्न अवरोधकको सम्भावित प्रभावकारितालाई रेखांकित गन्यो।

## LIST OF ABBREVIATIONS

EIS:	Electrochemical Impedance Spectroscopy
FTIR:	Fourier Transform Infrared Spectroscopy
GDP:	Gross Domestic Product
IE:	Inhibition Efficiency
MS:	Mild Steel
ppm:	Part Per Million
SEIH:	Stem Extract of <i>Iresine herbstii</i>
UV:	Ultraviolet Radiation

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# CHAPTER 1

## 1. INTRODUCTION

### 1.1. Introduction to Corrosion

Metals and their alloys are the major constituents for the material productions due to their high mechanical & tensile strength, rigidity, ductility, thermal, magnetic, and electrochemical properties (Kakani & Kakani, 2004). However, in the material form they try to attain stability through interactions with the surrounding environment which makes them vulnerable and prone to corrosion (Groisman, 2010).

Corrosion is a natural irreversible interfacial reaction of a material with its environment, resulting in the loss of material or in the dissolving of one of constituents of the environment into the material (Groisman, 2010; Sastri, 2011; Revie & Uhlig, 2008). Mild steel (MS) containing 0.05- 0.25% carbon is an excellent material used in structural and construction applications due to its ductility, affordability, mechanical strength, malleability and weld ability. Additionally, it is less tensile than other steel and most likely to undergo corrosion (Baker, 2020). The deterioration of metal in acidic solution is analogous to an irreversible oxidation-reduction (redox) reaction (Landolt, 2007). Corrosion is mostly problematic and unavoidable. However, there are several methods to minimize the corrosion rate such as electro-polishing (Hryniewicz et al., 2008), coatings (Voevodin et al., 2003), inorganic and organic inhibitors (Breston, 1952). However, the application of organic and inorganic inhibitors has shown a toxic effect on the environment, and they are very expensive.

An application of nontoxic, environment-friendly, cost-effective plant products as an inhibitor is a green corrosion inhibitor (Sastri, 2011). Plants are rich in various bioactive components i.e., phytochemicals such as primary and secondary metabolites that enable them to acquire physical and chemical diversity. Among them alkaloids are secondary metabolite components that have nitrogen incorporation in the heterocyclic ring of the amino acid. Several natural products particularly plant extracts have shown effective inhibition with greater benefits (Fayomi et al., 2019) over traditionally used organic and inorganic inhibitors based on their cost, toxicity, availability, and accessibility (Sastri, 2011).

Among all the phytochemicals flavonoids, tannins, anthraquinone, amino acids and proteins are responsible for inhibitory action (Abrishami et al., 2018; Odiongenyi et al., 2009). Their intricate phytochemical composition is attributed to the inclusion of hetero elements like nitrogen, sulfur, and oxygen (Alrefaee et al., 2021).

Oxidation occurs on the metal (anode) and the reduction of ions occurs in corrosive environments. The most common reaction of corrosion of metals and metal alloys in an acidic environment can be represented as follows:

### **Anodic Reaction**

Oxidation or loss of electrons takes place in an anodic reaction.

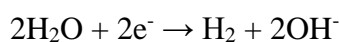
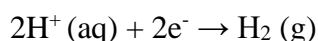


Where, M= metal

n= number of electrons released by an atom of the metal

### **Cathodic Reaction**

In cathodic reaction, reduction or gain of electrons takes place in an acidic medium, the reactions are:



## **1.2. Mild Steel Corrosion**

Mild steel is also known as low carbon steel. It is an alloy of carbon and iron, which contains 0.3 to 2.0% carbon. Mild steel is a low-priced ferromagnetic material i.e. it has good magnetic properties. It has high durability, welding ability, machinability properties, and very affordability that make it such a popular choice of steel for consumers (Yaro et al, 2013). Although its high mechanical properties, it can get rusted in contact with oxygen if it is not coated well because it has a relatively high amount of iron content. It is used worldwide because of its most common applications such as in structural steel, automobiles, furniture, wire, fencing, decorations, and nails (Yaro et al, 2013). Therefore, corrosion in mild steel has a great impact in economic value.

## **1.3. Corrosion Prevention Techniques**

There are many more techniques applied for corrosion prevention in various areas based on the corroding material and environment. Some processes like acid pickling,

descaling, production of various chemicals, fertilizers, and leaching of metallic ore sulphuric acid are widely used. To prevent and minimize corrosion, inhibitors are used. There are many organic and inorganic compounds that are widely used as corrosion inhibitors, but the inorganic compounds are toxic, costly, less effective, and non-environmentally friendly. Thus, its current use in corrosion inhibition is strictly inhibited because of ecological awareness and environmental importance. Therefore, it is replaced by green corrosion inhibitors (Verma et al., 2021).

As we all know, mild steel also can be protected from corrosion by using corrosion inhibitors and green corrosion inhibitors are very effective, inexpensive, and distributed worldwide. Extract of different parts of the plant can inhibit the corrosion process by virtue of the adsorption of phytochemical constituents on the corroding mild steel surface (Oguzie, 2010).

### **1.3.1. Synthetic Corrosion Inhibitor**

In recent years, polymers have been studied widely to be used as potential corrosion inhibitors because of their stability and cost-effectiveness. The organic compounds used in polymers having heteroatoms such as N, S, P, O, etc. are the most effective corrosion inhibitor as they can form metal complexes that cover the metal surface. This cover layer prevents the contact of corrosive media with the metal. Polymers such as polyethylene glycol, polyvinyl pyridine, polyacrylic acid, polyaniline, etc. have been reported for use as corrosion inhibitors. Due to the presence of heteroatoms, these are strong corrosion inhibitors for various metals in acidic conditions. However, due to their toxicity, complex synthetic procedure and cost, the use of synthetic inhibitors is limited (Arshad et al., 2019).

### **1.3.2. Green Corrosion Inhibitor**

Due to the drawbacks of synthetic inhibitors and easy availability of plant-based inhibitors they are widely used for corrosion inhibition. Green inhibitors are ecofriendly, biodegradable, and cheaper in nature due to which they are preferred over other inhibitors. Green inhibition is a process of protecting the metals from getting corroded by using different parts of plant extract such as root, shoot, bark, leaf, flowers, fruits, etc.

Plants produce several chemical constituents which are known as phytochemical. The biological roles of phytochemicals are to help in growth and protect from pathogens and predators. Naturally, different parts of the plant contain different kinds of chemical compounds such as alkaloids, flavonoids, tannins, unsaturated sterols, triterpenoids, essential oils, etc. (Gandhiraja et al., 2009). And phytochemical screening is the study of the biological potential of phytochemical constituents of plants including antioxidant, antiviral, antifungal, antibacterial, anticancer effects, and so on (Cocan et al., 2018).

Plants such as *Brassica oleracea* (Li et al., 2021), *Lilium brownie* (Zuo et al., 2021), *Ginkgo* leaf (Qiang et al., 2018), etc. were studied as green corrosion inhibitors and the studies show that all have more than 90% potential for corrosion inhibition. For example, natural gums are used as environmentally safe corrosion inhibitor for mild steel, and it has received tremendous attention in recent times which has non-toxic and biodegradability properties (Peter et al., 2015). The corrosion inhibition effect of plant extracts was found to be dependent on concentration and adsorption properties on the mild steel surface of extracts (Odewunmi et al., 2015).

The corrosion inhibition potential of *Phyllanthus emblica* leaves extract was investigated by evaluating the mild steel immersed in 1N HCl-containing extract and the results were shown that is necessary to improve surface condition for better corrosion protection (D'souza & Chatree, 2015).

*Azadirachta indica* commonly known as "Neem", is the most fruit source. It has high medicinal values as well as chemical and biological properties and increases current interest as a green corrosion inhibitor. Its extract has been used in inhibiting corrosion, especially of mild steel, aluminum, and tin (Sharma et al., 2015), and has a better effect of inhibition efficiency of 85% for stainless steel (Obiukwu et al., 2013).

#### 1.4. *Iresine herbstii* alkaloids Extract as Green Inhibitor



##### Scientific Classification

Kingdom:	Plantae
Order:	Caryophyllales
Family:	Amaranthaceae
Genus:	<i>Iresine</i>
Species:	<i>herbstii</i>

*Figure 1: Vegetative part of the Iresine herbstii*

*Iresine herbstii* is commonly known as blood leaf or beefsteak plant, a species of flowering plant of the family Amaranthaceae. It is a native plant of Brazil, generally used as decorative and ornamental purposes. The blood leaf plant is a short-lived perennial plant, that grows up to 5f tall and spreads 3f wide. It has antimicrobial and antibacterial properties, so the plant extracts, decoction, or pastes are hugely used for treatments of wounds, cuts, and burns (Dipankar et al. 2011).

#### 1.5. Corrosion Monitoring Methods

##### 1.5.1. Weight Loss Method

The weight loss method is the best and easiest corrosion monitoring method that can be implemented in any type of environment. It can be carried out at a very low cost in comparison to other methods. In this method, the rate of corrosion can be determined by monitoring changes in the weight of metals before and after exposure to the corrosive environment.

Mathematically, the rate of corrosion is estimated by the equation.

$$\text{Corrosion rate (CR)} = \frac{K \times \Delta W}{A \times t \times \rho} \quad \dots (1)$$

$$\text{And the Inhibition Efficiency (IE) \%} = \frac{W_0 - W_i}{W_0} \times 100 \quad \dots (2)$$

Were,

$\Delta W$  = Weight loss of the sample in grams (g)

A = Area of sample in  $\text{cm}^2$

t = time in hours

$\rho$  = density of the sample in  $\text{g/cm}^3$

K = Constant for unit conversion = 87600

$W_0$  = Weight loss of Mild steel in the absence of an inhibitor

$W_i$  = weight loss of the mild steel in presence of inhibitor

## 1.6. Objectives

The general and specific objectives of this research work are as follows.

### 1.6.1. General Objective

The general objective of this study is to prepare stem extract of *Iresine herbstii* as green corrosion inhibitor of MS corrosion in 1M  $\text{H}_2\text{SO}_4$  solution.

### 1.6.2. Specific Objectives

Specific objectives of this study are:

- Preparation of methanol extract from *Iresine herbstii* stem.
- Characterization of methanol extract by FTIR spectroscopy.
- Examine Inhibition efficiency of the extract by weight loss measurement method.
- Determination of adsorption isotherm and calculation of thermodynamic parameters.
- Determination of inhibitor efficiency of the extract by electrochemical method (Polarization and EIS method).

## CHAPTER 2

### 2. LITERATURE REVIEW

#### 2.1. Literature Survey

Several plants have been identified as sources of effective green corrosion inhibitors in acidic environments. Some of these plants have been incorporated into this project, and the outcomes are outlined below.

The study of alkaloid of *Rhynchostylis retusa* as green corrosion inhibitor for mild steel corrosion in 1M H<sub>2</sub>SO<sub>4</sub> solution showed that effective corrosion inhibition potential by forming protective film on the surface of mild steel. The experiment was monitored by weight loss and electrochemical polarization method. The results explored high inhibition capacity of 93.24 % for mild steel in sulphuric acid medium (Chapagain et al., 2022).

Li, et al., 2021, studied green *Brassica oleracea* leaf extract as a novel corrosion inhibitor for Q235 steel in two typical acid media viz. 0.5M H<sub>2</sub>SO<sub>4</sub> and 1M HCl. Inhibition capacity was testified by various techniques such as FTIR, UV-Vis and X-ray diffraction and the results showed great inhibition efficiency 92.3% & 93.8% in H<sub>2</sub>SO<sub>4</sub> and HCl respectively. Corrosion inhibition and adsorption effect on the surface of mild steel leaf extract of *Luffa cylindrica* was investigated in a hydrochloric acid environment by using GC-MS, FTIR spectrophotometer, and SEM techniques. The adsorption of leaf extract of *Luffa cylindrica* on the surface of mild steel was followed by Langmuir isotherm and physical adsorption mechanism. And the result was obtained in this study leaf extract of *Luffa cylindrica* is a good corrosion inhibitor for mild steel (Ogunleye et al., 2020).

One of the green corrosion inhibitors for mild steel is the fruit extract of *Citrullus lanatus*. It contains several phytochemicals such as *citrulline*, hesperidin, resveratrol, and riboflavin. Those phytochemicals of *Citrullus lanatus* fruit were used for mitigating the corrosion of mild steel in 1M HCl. The characteristics of corrosion inhibition were examined by electrochemical (EIS and polarization) and morphological techniques. 91% inhibition capacity was derived as a result of the electrochemical technique and the results of morphological (SEM and AFM) techniques *Citrullus lanatus* fruit extract was greatly influenced by protecting the surface of mild steel from corrosion (Devghani et al., 2019).

Methanolic extract of barks of *Lantana camara* in 1M HCl was studied for corrosion inhibition of mild steel. This research was carried out by using potentiodynamic polarization for testing the efficiency of corrosion inhibition of bark extract *Lantana camara*. It is reported that the inhibition shown by 1000 ppm inhibitor concentration is 97.33% (Shrestha et al., 2019). The corrosion inhibitor effect of seeds extract *Saraca ashoka* was examined with corrosion of mild steel in 0.5M H<sub>2</sub>SO<sub>4</sub>. The plant belongs to the family of leguminous. Weight loss measurements, potentiodynamic polarization measurement, and electrochemical impedance spectroscopy (EIS) techniques were used in the investigation of the corrosion inhibition effect and the adsorption effect of seeds extract *Saraca ashoka* was investigated by using AFM study, SEM study, and absorption spectroscopic techniques. The seed extract of *Saraca ashoka* was confirmed as a good corrosion inhibitor due to the existence of hetero atoms in the main components (Saxena et al., 2018).

As an effective green corrosion inhibitor for mild steel, the flower extract of *Canna indica* was studied. The study showed that the increase in concentration was directly proportional to the efficiency of its corrosion inhibition properties and decreased its inhibition efficiency with an increase in temperature. And the study confirmed that *Canna indica* flower extract was eco-friendly, cost-effective, and smoother on the surface of mild steel (great corrosion inhibitor for mild steel) (Mathina & Rajalakshmi, 2016). The study of corrosion inhibition efficiency of flower extracts of *Heliconia rostrata* in 1M hydrochloric acid on mild steel by weight loss measurement, electrochemical impedance, and potentiodynamic polarization techniques was conducted. As overall results inhibition efficiency was increased with an increase in concentration and scanning electron microscopy (SEM) was used to confirm the inhibitive nature of plant extracts (Mathina & Rajalakshmi, 2016).

Peel extract of *Musa paradisiaca* in 1M HCl, has corrosion inhibition efficiency as well as an adsorption effect on the mild steel surface. The UV-visible spectroscopy, FTIR spectroscopy, and HPLC techniques were studied for the characterization of *Musa paradisiaca*. The study of electrochemical techniques showed decreased inhibition ability in the maturity stage (Ji et al., 2015). *Ketosulfone* drug was studied as a green corrosion inhibitor in 1M HCl, which acts as a mixed-type inhibitor for mild steel. The morphological (SEM) and electrochemical techniques were shown to have good

corrosion inhibition ability and adsorption ability of *Ketosulfone* extract on the surface of mild steel (Matad et al., 2014).

The inhibitory impact of *Argemone mexicana* plant extract on mild steel corrosion in 1M HCl solution has been studied by weight loss and electrochemical polarization method. Obtained results showed that extract of *Argemone mexicana* plant found as good green inhibitor for mild steel in an acidic environment. And the results also showed the effectiveness of inhibitor was increased with increasing concentration. The maximum inhibition efficiency was found to be 92.5% and the inhibition efficiency was studied by UV-Vis, electrochemical and surface imaging techniques (Ji et al., 2011). By using extracts of stem bark of *Jatropha curcas* in ethanolic, methanolic, and water the in vitro antimicrobial activity was investigated in different concentrations. The clarity of that study was the plant has antifungal and antibacterial properties, which inhibit the fungal and bacterial growth in the plant. And the bark stem of *Jatropha curcas* contains several phytochemicals such as saponin, steroids, tannin, flavonoids, alkaloids, and glycosides (Igbiosa et al., 2009).

Green corrosion inhibition by naturally occurring biomolecule caffeic acid on the mild steel in 0.1M H<sub>2</sub>SO<sub>4</sub> was investigated. Using different techniques such as electrochemical impedance, potentiodynamic polarization, weight methods, and Raman spectroscopy confirmed that caffeic acid has a good corrosion efficiency and adsorption effect on the surface of mild steel (de Souza & Spinelli, 2009).

Satapathy, et al., 2009, investigated the corrosion inhibition effect of plant extract of *Justicia gendarussa* in hydrochloric solution for mild steel corrosion by electrochemical and weight loss measurements. The study showed the 93% inhibition efficiency of plant extract of *Justicia gendarussa* at 25 °C due to formation of protective film and adsorption on the surface of mild steel.

## **2.2. Research Gap**

The utilization of acids in cleaning procedures may result in heightened corrosion of different tools. Inhibitors are commonly employed to minimize the corrosion in these processes. Among these inhibitors, green inhibitors have gained attention for their potential in corrosion control. Furthermore, achieving optimal corrosion inhibition at elevated temperatures, even with a 100% concentration of inhibitors, requires

investigating the corrosion inhibition mechanism and enhancing the effectiveness of green inhibitors, which might initially exhibit low efficiency.

To overcome these limitations, it is essential to identify potent phytochemical, as they vary among plants and even within different parts of the same plant. Any part of *Iresine herbstii* was not researched yet as green corrosion inhibitor. This project aims to investigate the phytochemicals present in the stem of *Iresine herbstii*. Subsequently, the corrosion inhibiting efficiency of this plant on mild steel will be studied in a sulphuric acid solution with a concentration of one molar.

## CHAPTER 3

### 3. MATERIALS AND METHODS

#### 3.1. Collection of plant

The stem of *Iresine herbstii* (Bloodleaf plant) was collected from Neelkantha-11, Dhading district (Latitude: 27.98317 °E, Longitude: 84.88624 °N and Altitude: 1400m to 1600m), Bagmati province, Nepal. The detail of the geographical map of this area is shown in the figure 2.

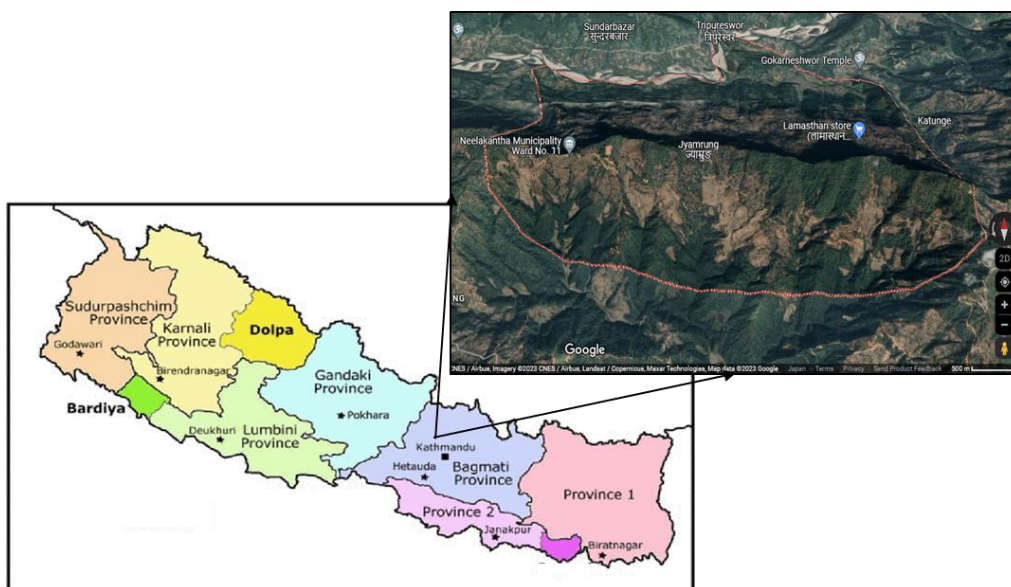


Figure 2: Geographical map showing plant collection area.

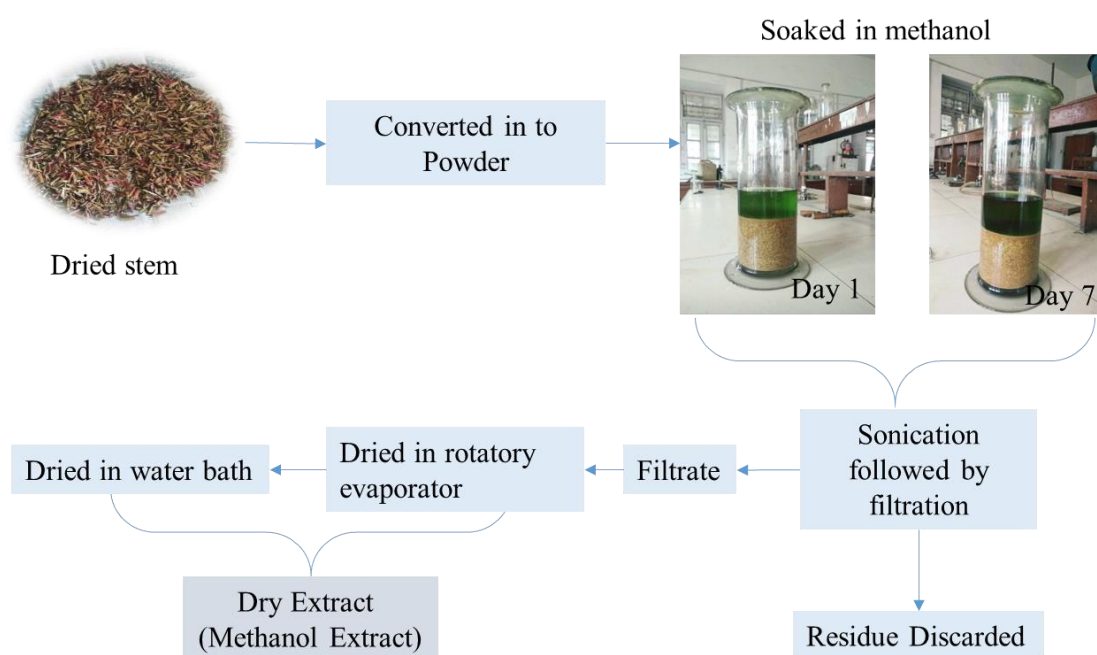
#### 3.2. Preparation of powder

The collected plant stem was cleaned with water and dried in the shade and then the dried stem was grinded into a fine powder with the help of a herbal disintegrator. Thus, ground sample was sieved and stored in dry airtight vessel prior to use.

#### 3.3. Preparation of plant extract

Methanol extract which later will be called inhibitor was extracted from *Iresine herbstii* stem powder. For which 100 g of grinded powder was weighed in a weighing machine and then transferred to tall jar. The powder was then soaked in 800 mL methanol for 7 days. The mixture was then taken in two beakers and sonicated with the help of a sonicator for 1hour and then it was left for one day and then filtered. Filtrate was collected in a beaker and phytochemical tests were done by using little amount of

filtrate solution. The process was repeatedly done to obtain more extract. The filtrate obtained was concentrated using (IKA RV 10) digital rotatory evaporator 5-6 times and collected in a beaker. The obtained concentrated extract was vaporized in a water bath to obtain dried extract which was further used to prepare inhibitor solution. Schematic diagram of extract preparation is shown in figure 3.



*Figure 3: Schematic diagram showing the extraction.*

### 3.4. Preparation of alkaloids

The concentrated filtrate (methanol extract) was treated with 5% acid (HCl or tartaric acid) until a pH of 2-3 (2.5) was maintained, thereby converting the alkaloids of the filtrate into their salts. Dichloromethane (DCM) was then added, as organic compounds are soluble in DCM while alkaloid salts are acid soluble. The DCM layer was separated from the aqueous layer using a separating funnel and discarded to eliminate other phytochemicals and leaving the alkaloid salts in the aqueous layer. Subsequently, ammonium hydroxide (NH<sub>4</sub>OH) was introduced to the aqueous layer until the pH reached 10-12 (11), facilitating the conversion of alkaloid salts into alkaloids. Another round of DCM extraction followed, separating the DCM layer containing the soluble alkaloids from the aqueous layer. The DCM layer was then dried to recover the alkaloids.

### **3.5. Preparation of mild steel coupon**

The sample of mild steel sheet was collected from local market of Kathmandu, Nepal and was cut into coupons of nearly equal dimensions (3×3×1) cm. Each coupon was polished by using silicon carbide papers of different grits (150-2000). Then the polished coupons were sonicated in hexane for 15 minutes and then dried. Prior to each experiment, the dimensions of each coupon were measured with the help of a digital Vernier caliper and weight was measured by weighing machine and noted.

### **3.6. Preparation of 1M H<sub>2</sub>SO<sub>4</sub> Solution**

One molar sulphuric acid was prepared by diluting concentrated acid. For which, 55.10 mL of Conc. H<sub>2</sub>SO<sub>4</sub> was added in installments into 500 mL distilled water in 1 L volumetric flask with frequent stirring. Then the volume was made up to mark by adding distilled water. The concentration of 1M sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) was ensured by titrating with 1M Na<sub>2</sub>CO<sub>3</sub> solution.

### **3.7. Preparation of inhibitor solution**

The stock solution of inhibitor was prepared by dissolving 1 g plant extract in 1000 mL of 1M H<sub>2</sub>SO<sub>4</sub> in 1000 mL of the volumetric flask to make a 1000 ppm solution. All the extract was not dissolved properly it was filtered and the undissolved residue was discarded. After that different concentration (200, 400, 600, 800 and 1000 ppm) solutions of inhibitor were prepared from 1000 ppm stock solution of inhibitor by serial dilution.

### **3.8. Test for Phytochemicals**

To know the presence of alkaloids, flavonoids and polyphenols in the extract, the phytochemical test was performed. It is assumed that nitrogen containing phytochemicals could show good inhibition against mild steel corrosion, so, mainly alkaloid test was performed.

#### **3.7.1. Dragendorff's Test**

To prepare Dragendorff's Solution A: 3 grams of bismuth nitrate were dissolved in 8 milliliters of 1M H<sub>2</sub>SO<sub>4</sub>, while for Dragendorff's Solution B, 12 grams of KI were

dissolved in 18 milliliters of distilled water. Subsequently, solutions A and B were combined in a 50 milliliter volumetric flask with distilled water.

### 3.7.2. Mayer's Test

A freshly prepared Mayer's reagent was made by dissolving 1.36 grams of mercuric chloride and 5 grams of potassium iodide in 100 milliliters of water. A small portion (2 milliliters) of the extract was then treated with Mayer's reagent, also known as potassium mercuric iodide ( $K_2HgI_4$ ).

### 3.9. Weight loss measurement method

The weight loss measurement method is a quantitative method used to monitor and measure the overall corrosion of metal surfaces. In this method, the quantity of corrosion of metal can be determined by recording the difference in weight of metal before and after immersion in the corrosive environment over specific time intervals.

The effect of immersion time, the concentration of inhibitor solution, and the effects of different temperatures on the mild steel corrosion were studied. At first, 60 mL each acid and inhibitor solutions of different concentration were taken in separate 100 mL beakers. Then the polished and weight recorded coupons were dipped in the solutions making one in each and left for 30 minutes. After 30 minutes, each coupon was taken out and washed thoroughly and dried and the weight of the coupons was measured. The difference in weight of each coupon before and after immersion was calculated. Similar process was repeated for 1, 3, 6, 9, 18, and 24 hours. Similarly, the effect of temperature was also studied by immersing samples in each solution for 1 h at a different temperature. The constant temperature was maintained using a water bath. Corrosion rate and inhibition efficiency were determined by using equations (1) and (2) respectively.

Mathematically, the rate of corrosion is estimated by the equation.

$$\text{Corrosion rate (CR)} = \frac{K \times \Delta W}{A \times t \times \rho} \quad \dots (1)$$

$$\text{And the Inhibition Efficiency (IE) \%} = \frac{W_0 - W_i}{W_0} \times 100 \quad \dots (2)$$

Were,

$\Delta W$  = Weight loss of the sample in grams (g)

$A$  = Area of sample in  $\text{cm}^2$

$t$  = time in hours

$\rho$  = density of the sample in  $\text{g/cm}^3$

$K$  = Constant for unit conversion = 87600

$W_0$  = Weight loss of Mild steel in the absence of an inhibitor

$W_i$  = weight loss of the mild steel in presence of inhibitor

### **3.10. Potentiodynamic polarization**

The corrosion potential, corrosion rate and Tafel slopes were all determined using polarization measurement technique. The polarizations were conducted with three electrode cell set up. The experiment was done within same electrolytic cell which was used to measure the OPC. The sample was polarized cathodically and anodically in range from  $-0.9\text{V}$  to  $-0.1\text{V}$  from OPC. by using different inhibitor solution as well as acid solution, the polarization measurement was performed on mild steel surface. Tafel slope, corrosion current and corrosion potential were done by using polarization curves.

### **3.11. FTIR Analysis**

FTIR spectroscopy is a powerful tool for identifying the types of bonds in organic molecules, particularly functional groups. The FTIR spectra of alkaloids and steel coupons immersed in a 1000ppm inhibitor were obtained at Amrit Campus, Kathmandu, using a PerkinElmer Spectrum IR (Version 10.6.2) FTIR spectrometer. By analyzing the FTIR spectra, the presence of aromatic and aliphatic structures in the alkaloid samples can be determined based on their functional groups.

## CHAPTER 4

### 4. RESULTS AND DISCUSSION

#### 4.1. Test of Alkaloids

The stem of *Iresine herbstii* was used to extract the phytochemicals by using solvent extraction method. Alkaloids content in the methanol extract was confirmed by chemical test methods. The formation of precipitates with characteristic color for each test confirmed the presence of alkaloids. The results obtained are in table 1.

*Table 1: Chemical test for alkaloids*

S.N.	Experiment	Inference	Result
1.	Dragendorff's Test	+Ve	The appearance of orange-red color
2.	Mayer's Test	+Ve	The appearance of orange precipitate

#### 4.2. FT-IR Analysis

FTIR spectroscopy, using the PerkinElmer Spectrum IR version 10.6.2, enables the identification of bond types, aliphatic and aromatic structures, and crucially, functional groups in organic compounds. The spectrum of the extract from *Iresine herbstii* was analyzed using FTIR, revealing the presence of various organic compounds and functional groups.

In the provided figure, the FTIR spectra of the extract exhibit characteristic functional groups. For instance, the O-H stretching of alcohols, phenols, and carbohydrates, as well as the N-H stretching of amines, manifest in the bandwidth ranging from  $3400\text{ cm}^{-1}$  to  $3290\text{ cm}^{-1}$ . A peak at  $1624\text{ cm}^{-1}$  indicates C=C stretching, which can denote the presence of oximes, amides, or C=N groups. Additionally, the absorption band at  $1390\text{ cm}^{-1}$  corresponds to the O-H bending of carboxylic acids. The presence of aromatic amines is evidenced by a strong peak at  $1033\text{ cm}^{-1}$ , representing the C-N stretching. Furthermore, the bending of alkenes, reflected in the C=C bending, is depicted by another band at  $607\text{ cm}^{-1}$  (Karki, et al., 2020).

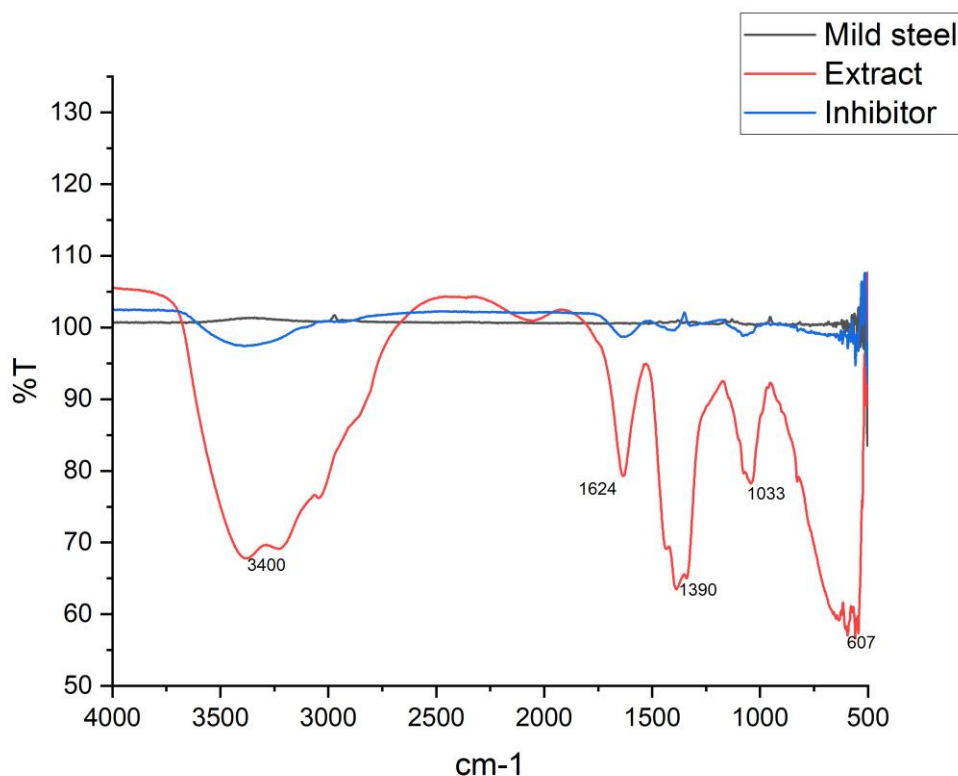


Figure 3: FT-IR spectrum of MS, extract(alkaloid), and MS immersed in 1000ppm inhibitor in 1 hr.

### 4.3. Weight Loss Measurements

#### 4.3.1. Variation of weight loss with immersion time

The provided figure presents the outcomes of a weight loss experiment conducted on MS coupons immersed in 1M H<sub>2</sub>SO<sub>4</sub>, both with and without inhibitor. According to the weight loss measurements, the presence of alkaloids reduces the metal's weight loss in acidic inhibitor medium. The experiment was conducted at room temperature (15°C) over various time intervals: 1, 3, 6, 9, 18, and 24 hours.

The results indicate that as the duration of immersion increases, the metal's weight loss also increases in both scenarios. However, the ratio of weight loss decreases in the

presence of the inhibitor compared to the acidic environment alone. This phenomenon can be attributed to the deposition of alkaloids on the metal surface, leading to the formation of a protective deposition layer.

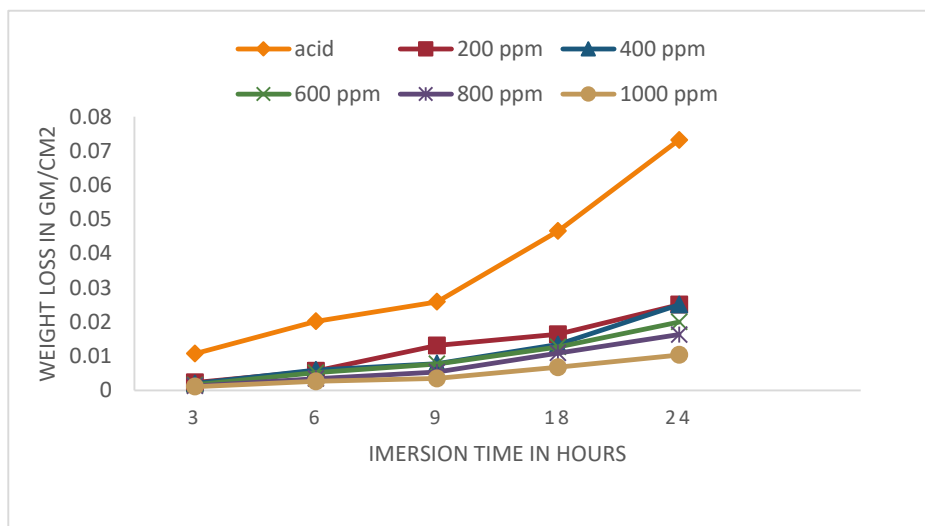


Figure 4: The weight loss variation over immersion time was studied for mild steel corrosion in two different solutions: 1M H<sub>2</sub>SO<sub>4</sub> and solutions with varying inhibitor concentrations of different inhibitors.

The inhibitor efficiency of the inhibitor molecules of different concentration in 1M H<sub>2</sub>SO<sub>4</sub> shown in given below. The efficiency of 1000 ppm inhibitor is highest with 87.28% at 3 h immersion time.

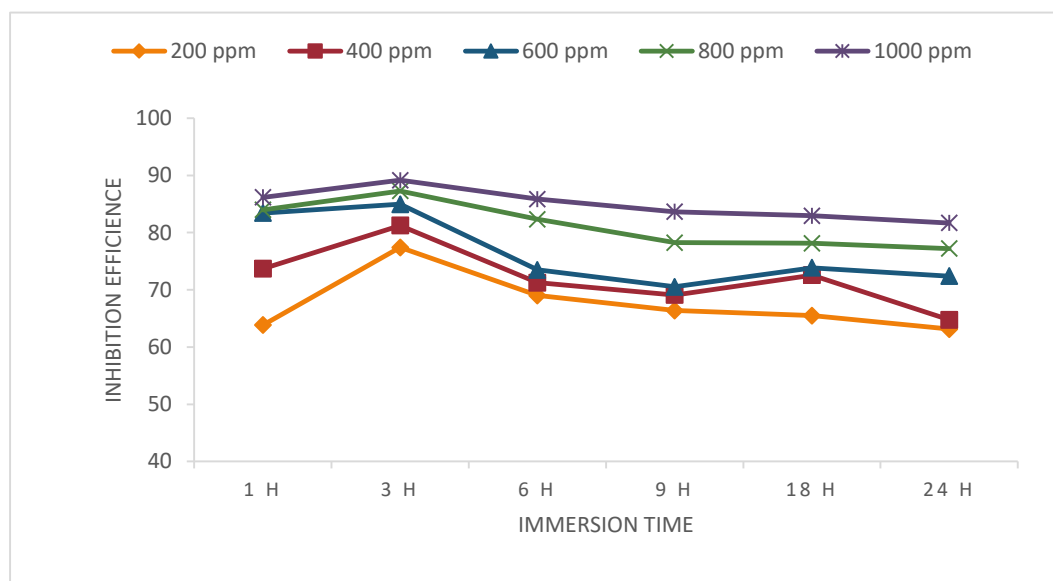


Figure 5: The Variation of inhibitor efficiency of different concentration inhibitor solutions in 1M H<sub>2</sub>SO<sub>4</sub> vs time for the mild steel at different times.

### 4.3.2 Weight loss of Mild Steel in various concentration of alkaloid at different time

When mild steel coupons are submerged in an acidic solution, a reaction occurs between the acid and the metal, leading to the development of corrosion on the surface. This corrosion process results in a decrease in the weight of the mild steel coupons. Factors such as temperature, duration of exposure, and concentration of the solution also influence the rate of corrosion.

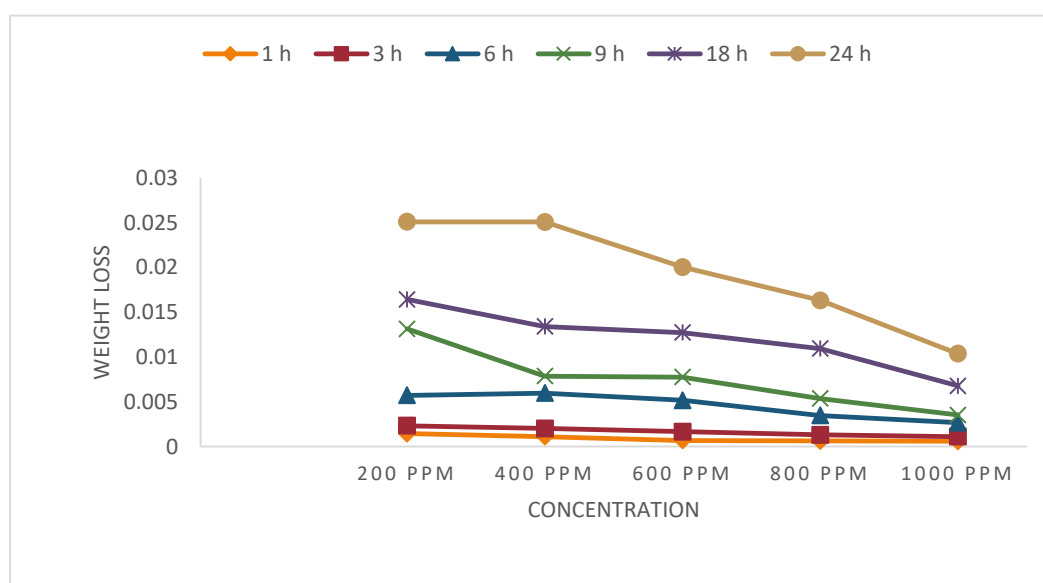


Figure 6: Variation of weight loss vs different concentration of extract on MS in 1M H<sub>2</sub>SO<sub>4</sub> in various time.

In the depicted figure, it's observed that within the initial hour, all concentrations resulted in minimal weight loss. However, an unexpected increase in weight loss occurred in the 1000ppm concentration, likely due to handling errors. Contrary to the pattern observed at earlier time intervals (1hr, 3hr, 6hr, 9hr, and 18hr), the 24-hour mark indicates a higher weight loss. This discrepancy could be attributed to the desorption of inhibitor molecules over an extended period (Karki, et al., 2021).

### 4.3.3 INHIBITOR EFFICIENCY OF DIFFERENT CONCENTRATION SOLUTION OF EXTRACT.

At all concentration, inhibitory efficiency of inhibitor solution is greater than 60% through out any time immersion period. This illustrates that there is sufficiently inhibitor in the surface of ms, increasing of surface covered of ms surface is indicated by increasing order of concentration of solution. The 1000ppm concentration exhibited higher efficiency across all time periods, attributed to a sufficient absorption of inhibitor molecules onto the surface of the mild steel, consequently leading to reduced weight loss.

Typically, there's a trend of increasing inhibitor efficiency from low to high ppm concentrations across all immersion times, with maximum efficiency observed at the 1000 ppm concentration.

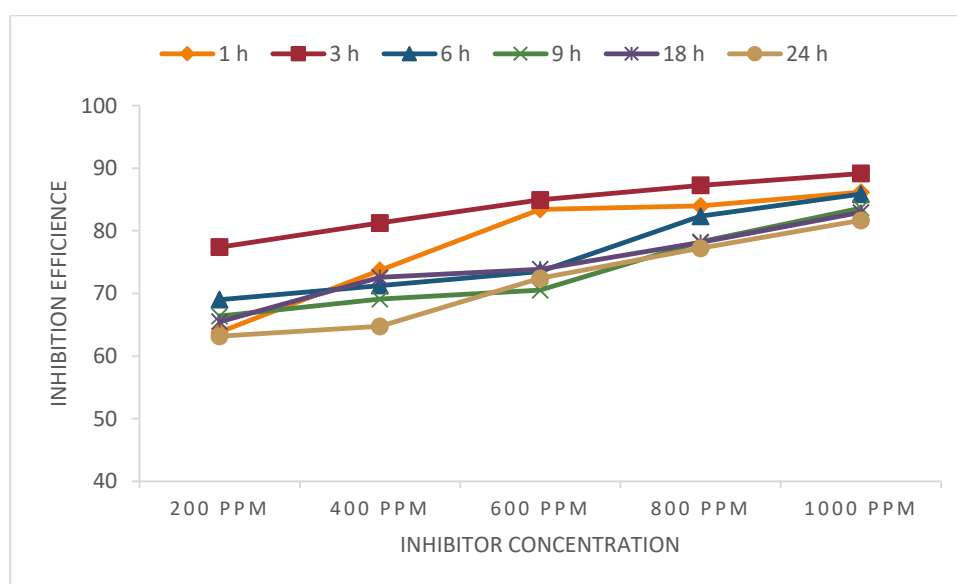


Figure 7: The efficiency of inhibitors at various concentrations on mild steel in a 1M H<sub>2</sub>SO<sub>4</sub> solution was investigated at different time intervals.

### 4.3.4 The Variation of weight loss vs temperature

concentrations (200 to 800 ppm), there is a consistent pattern where weight loss remains relatively stable as temperature increases. Notably, the weight loss values are very close to each other for these concentrations, suggesting that the inhibitor's protective effect is similarly effective across this range. In contrast, the line representing "Acid" shows a

significant increase in weight loss as the temperature rises to 35°C, indicating a marked acceleration of corrosion or deterioration in this condition. Based on this data, one can conclude that the presence of the inhibitor significantly reduces weight loss due to corrosion across a temperature range of 15°C to 35°C, as compared to the acidic condition without an inhibitor (Andor, et al., 2021). The effect of temperature on corrosion rate is mitigated by the inhibitor, as evidenced by the stable weight loss values at varying temperatures.

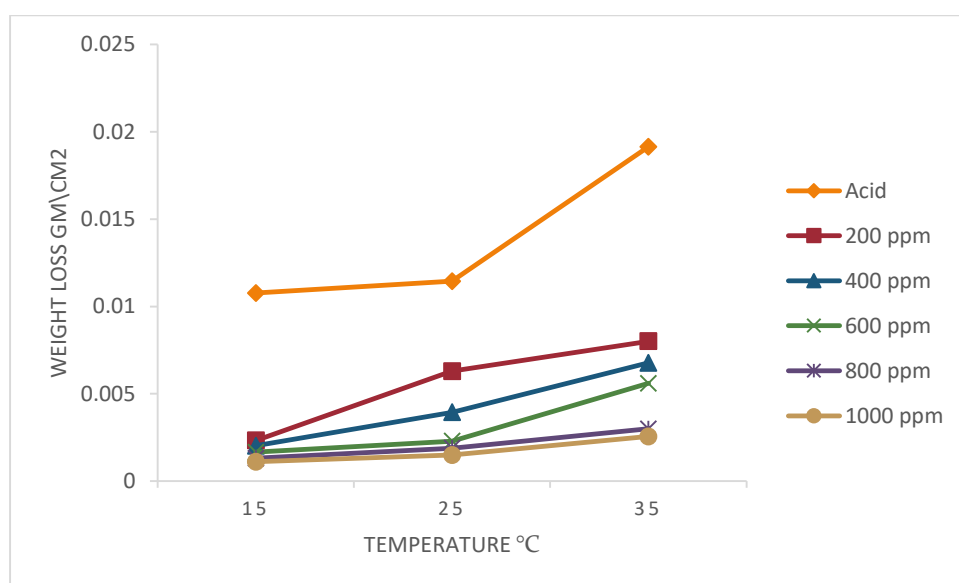


Figure 8: Variation of weight loss of MS in 1M H<sub>2</sub>SO<sub>4</sub> at different temperature

#### 4.3.5 Effect of Temperature on Inhibitor Efficiency

From the observed data, it is evident that the inhibitor's efficiency varies with temperature and concentration. At lower temperatures (15°C), the efficiency is higher across all concentrations, indicating reduced efficiency. With the temperature rising from 15°C to 35°C, a notable increase in efficiency is observed, particularly at the 1000 ppm concentration. This enhancement is attributed to the adsorption of alkaloids on the surface of mild steel, indicating that the inhibitor exhibits optimal performance within this temperature range.

Additionally, the figure indicates that the efficacy of the 200 ppm inhibitor solution is comparatively lower across all temperatures. However, up to 35°C, the efficiency of the 1000 ppm inhibitor reaches its peak

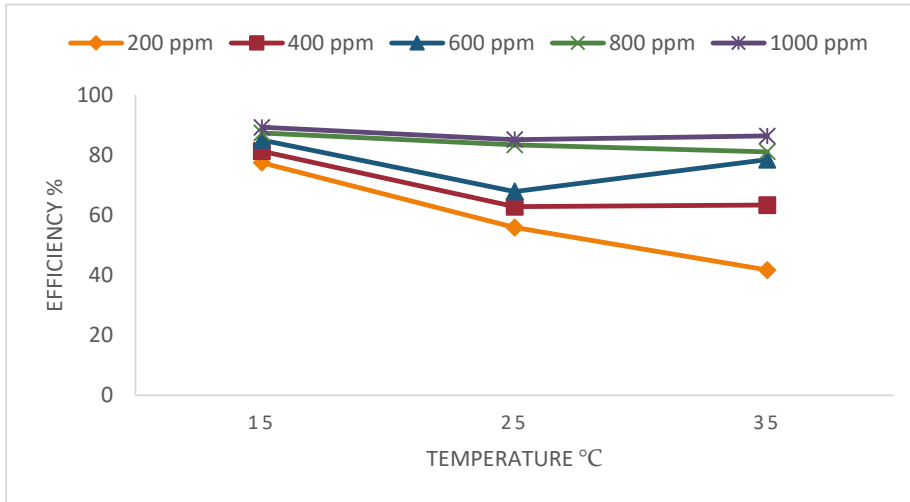


figure 9: Variation of inhibitor efficiency with temperature in 1M H<sub>2</sub>SO<sub>4</sub>

#### 4.4 Adsorption Isotherm

If the objects are submerged in solutions, solution molecules are adhering to the surface of the object, a process known as adsorption. In the case of MS submerged in acid, the acid molecules attach to the surface, causing corrosion. However, when an inhibitor solution is present alongside the acid, inhibitor molecules adhere to the metal surface instead, reducing corrosion.

The adsorption isotherm offers crucial insights into how inhibitors interact with the surface of MS. Initially, water molecules adhere to the surface of mild steel in an aqueous solution, but over time, inhibitor molecules displace these water molecules through a process resembling quasi-substitution. In the aqueous solution, the adsorption of inhibitor compounds involves a substitution mechanism. Utilizing the Langmuir adsorption isotherm equation allows us to determine whether the adsorption occurs in a monolayer or multilayer manner.

In the presence of an acid-inhibitor solution mixture, inhibitor molecules initially bind to the surface of mild steel, thereby attenuating the degradation of mild steel. A comprehensive understanding of the adsorption isotherm is crucial for fully

comprehending this process. Analysis of adsorption isotherms assists in elucidating the nature of adsorption between the mild steel surface and inhibitor molecules.

The Langmuir adsorption is as,  $C_{inh}/\theta = 1/K_{ads} + C_{inh}$ . . . . .3

The equation bears resemblance to the linear equation  $y=mx+c$ , indicating a linear relationship between the covered surface ( $\theta$ ) value and the concentration of inhibitor ( $C_{inh}$ ). To ascertain the adsorption isotherm, understanding the linear relation between these variables is essential. A slope of unity in the curve signifies monolayer adsorption.

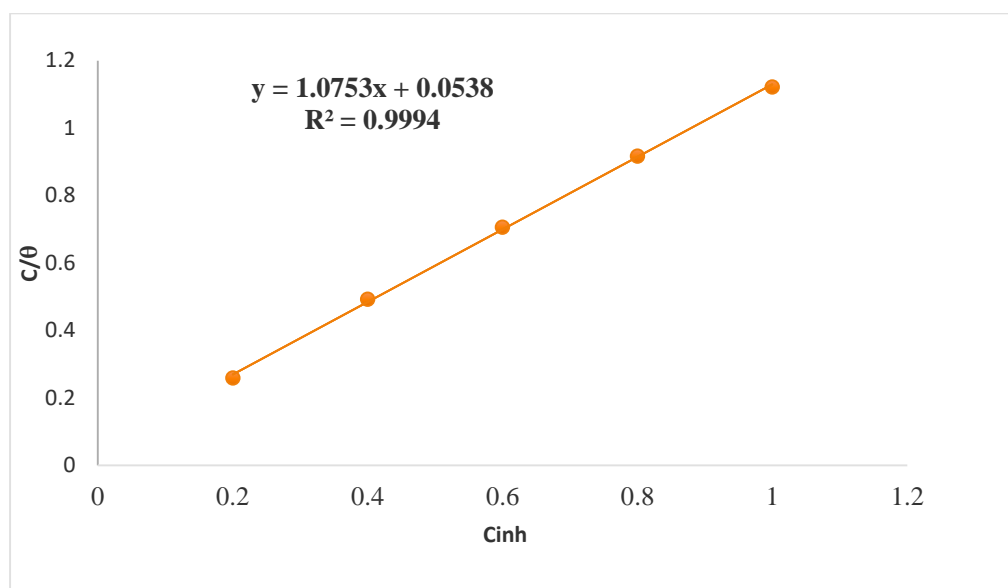


Figure 10: Langmuir adsorption isotherm plot for Acid with different concentration of inhibitor.

The figure illustrates the regression coefficient ( $R^2$ ), and it's evident that the slope is not equal to unity. This discrepancy suggests that the alkaloid molecules are not adhering strictly to the Langmuir adsorption isotherm. Instead, it indicates the possibility of both monolayer and multilayer adsorptions. The  $R^2$  value and slope being slightly distant from unity further support this notion. Consequently, the corrosion process cannot be fully explained by the Langmuir adsorption isotherm. (Ituen, et al., 2017).

## 4.5 Activation Energy.

The activation energy in an electrochemical cell, both in the presence and absence of an inhibitor, is calculated using the Arrhenius equation. It's important to note that the activation energy is directly proportional to the corrosion rate as

$$\text{Log}(\text{CR}) = \log A - E_a/2.303RT \dots\dots\dots 4$$

In the provided equation, where A represents the Arrhenius constant and T denotes the absolute temperature, the activation energy is determined by the slope of the Arrhenius plot. This plot is constructed by graphing  $\log(\text{CR})$  against  $1/2.303RT$

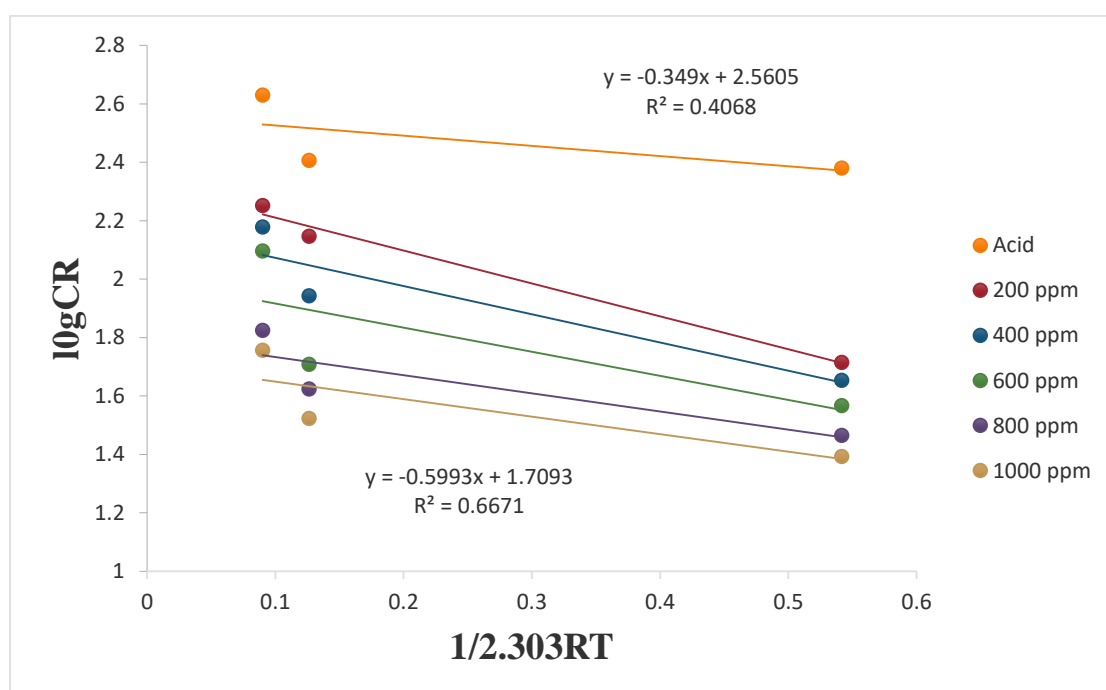


Figure 11: Arrhenius plot for MS in 1M H<sub>2</sub>SO<sub>4</sub> with and without inhibitor

In the presence of the inhibitor, the activation energy for the reaction between the acid and mild steel (MS) increases to 59.9 kJ/mol, compared to 34.9 kJ/mol without the inhibitor. This elevation in activation energy can be attributed to the hindrance of acid molecules from accessing the surface of the MS, effectively impeding the corrosion process. The observed increase in activation energy is indicative of the inhibitor's ability to lower the corrosion rate.

The value of the activation energy lies between the ranges typically associated with physical (less than or equal to 20 kJ/mol) and chemical (greater than 50 kJ/mol) processes. This suggests that the inhibitor operates through a mechanism that involves both physical and chemical interactions, providing an effective barrier against corrosion. The adsorption in MS surface is mixed types of adsorption due to dominance of chemisorption (Karki et al., 2021).

#### 4.5.1. Enthalpy and Entropy measurement

The enthalpy and entropy are calculated by alternative form of Arrhenius equation,

$$\log(CR/T) = \log(R/hN_A) + \Delta s^\circ/2.303RT - \Delta H^\circ/2.303RT \dots\dots\dots 5$$

where, h is Plank's constant,  $6.626 \times 10^{-34}$  Js. And  $N_A$  is Avogadro's number.

By plotting  $\log(CR/T)$  vs  $1/2.303RT$ ,  $\Delta H^\circ$  and  $\Delta s^\circ$  are calculate from slope and intercept respectively (Karki, et al., 2021).

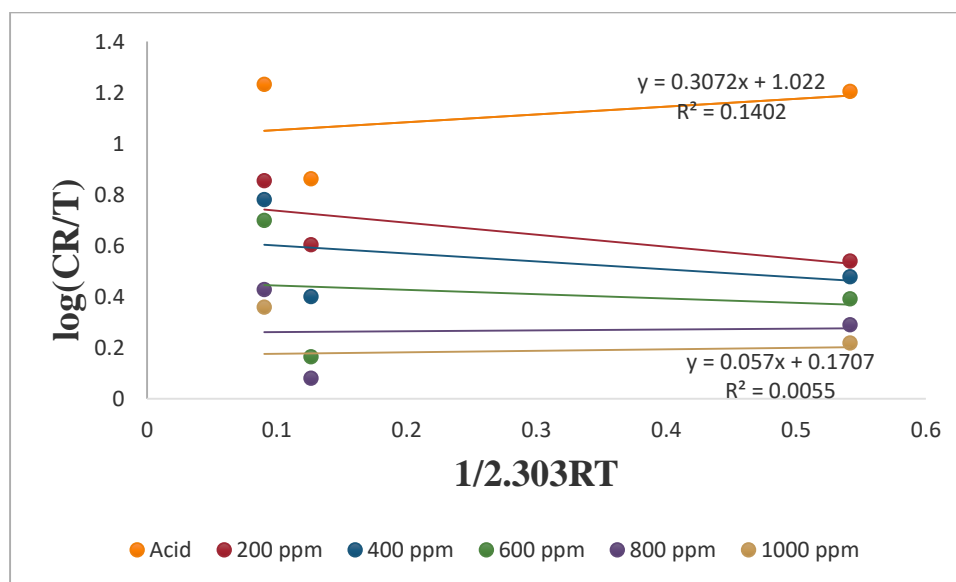


Figure 12: Transition state plot for Mild Steel in 1M H<sub>2</sub>SO<sub>4</sub> both with and without inhibitor

Based on the provided data, the enthalpy of activation was determined to be 30.72 kJ/mol in the absence of an inhibitor, and it increases with increasing inhibitor

concentration. Notably, the enthalpy of activation for the 1000 ppm concentration was found to be higher than other concentrations, measuring 57 kJ/mol.

The transition state plot for mild steel (MS) in 1 M H<sub>2</sub>SO<sub>4</sub>, both with and without an inhibitor, reveals positive  $\Delta H^\circ$  values for inhibitor adsorption on the MS surface, indicating an endothermic adsorption process. The progressive increase in  $\Delta H^\circ$  values suggests a decline in the corrosion rate, primarily regulated by activation kinetic factors.

Furthermore, the involvement of a gaseous reaction, such as the cathodic hydrogen evolution process, leads to a reduction in the total reaction volume when the activation energy ( $E_a$ ) surpasses  $\Delta H^\circ$ . These findings suggest that alkaloid adsorption occurs through a combination of physical and chemical interactions (Ituen, et al., 2017).

## **4.6 Electrochemical Methods**

### **4.6.1. Polarization measurement of 1 hr. Immersed MS sample**

Polarization tests were conducted on mild steel samples immersed in acid solutions containing inhibitor concentrations ranging from 200 to 1000 ppm. The polarization behavior was examined by applying  $\pm 350$  mV in both anodic and cathodic directions using a potentiostat. Analysis using Evans diagrams and Tafel extrapolation revealed a decrease in current density with the introduction of inhibitors. This decline indicates that the mild steel surface becomes coated with inhibitor molecules, thereby reducing the availability of active sites for the corrosion process.

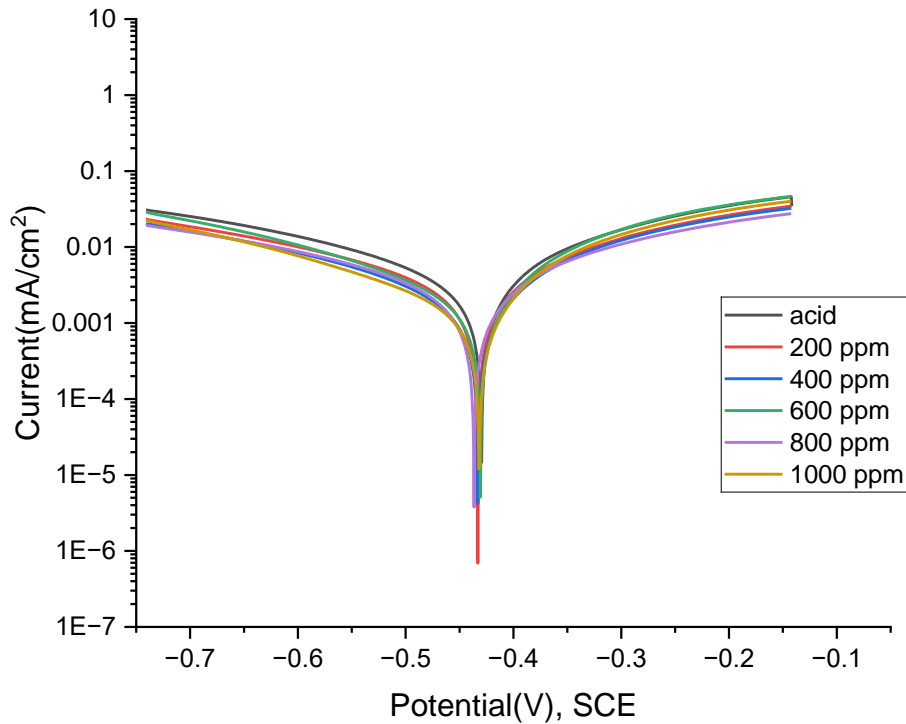


Figure 13: Potentiodynamic polarization curves for MS in 1 M H<sub>2</sub>SO<sub>4</sub>

The polarization measurement was carried at 15°C at laboratory and the decreasing of in corrosion current is due to the blockage of reacting sites by inhibitor molecules.

In the acidic solution, the current density was measured at 0.74092 mA/cm<sup>2</sup>, with a corrosion potential of -0.0225 V. Upon adding the inhibitor, the current density decreased, indicating a reduction in the corrosion rate due to the blocking of the mild steel (MS) surface. At a concentration of 1000 ppm inhibitor solution, the lowest current density observed was 0.1203 mA/cm<sup>2</sup>, accompanied by a corrosion potential of -0.0205 V.

#### 4.6.2. Electrochemical Impedance Measurements

EIS measurement for as-immersed condition reflects the inhibition behavior of the inhibitor from very beginning. EIS data recorded at 15°C in 1M H<sub>2</sub>SO<sub>4</sub> and inhibitor medium pointed the increase in impedance on increasing inhibitor concentrations (200, 400, 600, 800 and 1000 ppm). The impedance value for acid only solution is 310 Ω whereas for 1000 ppm solution is 1230 Ω obtained from Nyquist plot in given figure

(a). The increase in impedance implies that the resistance for corrosion reaction is increased.

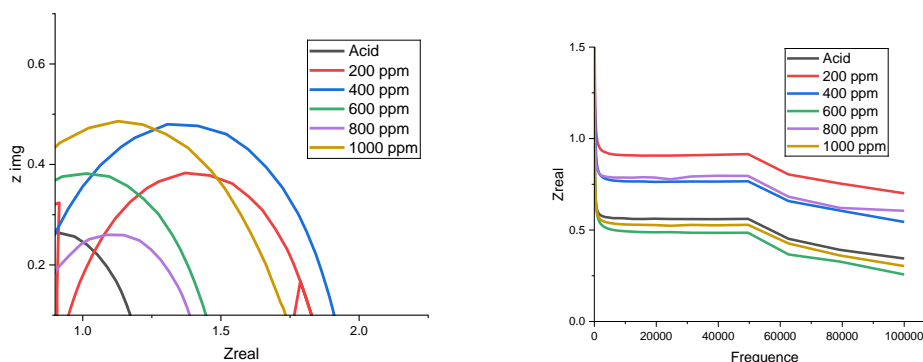


Figure 14 (a) Electrochemical impedance measurement curve for MS immersed for 1 hours in all the inhibitor solution and acid media, (b) Modulus frequency curve obtained from EIS measurement of sample in 1 hour immersed condition

Modulus curve was fitted from EIS measurement and found the resistance at higher frequency range is around  $10\Omega$  indicating highly conductive nature of the solution whereas resistance in lower frequency region is continuously increasing on increasing the inhibitor concentration. At lower frequency the resistance of acid only solution is  $1020\Omega$  whereas resistance of 1000 ppm inhibitor solution is  $642\Omega$  as in figure (b). This implies that on increasing the inhibitor concentration, the inhibitor molecules inhibit and minimize the corrosion reaction.

## Mechanism of Corrosion Inhibition

It seems like you're discussing the corrosion inhibition process on mild steel, focusing on the adsorption of inhibitor molecules onto the metal surface. The inhibitor, *Irsine herbstii*, is described as adsorbing on the mild steel surface through a process involving both physisorption and chemisorption. Physisorption occurs due to van der Waals

forces, while chemisorption involves ionic, covalent, and hydrogen bonding interactions.

The inhibition mechanism of *Irsine* is attributed to the presence of nitrogen and oxygen heteroatoms in its structure, which facilitate its adsorption on the mild steel surface. This adsorption can be explained using either the HOMO-LUMO concept or the HSAB (Hard and Soft Acids and Bases) concept. (Kairi and Kassim, 2013).

According to the HOMO-LUMO concept, the lone pair of electrons located on heteroatoms such as nitrogen or oxygen (the highest occupied molecular orbital, or HOMO) can form a coordinate covalent bond with the unoccupied d-orbital of an iron atom. This interaction results in the accumulation of negative charge on the surface, which is neutralized by sending back electrons to the LUMO (lowest unoccupied molecular orbital), especially to the anti-bonding  $\pi^*$  orbital of the organic molecule. This retro donation adds stability to the complex and enhances the binding affinity between the inhibitor and the active metal site. (O.A. Hatem, 2022).

Moreover, the vacant d-orbital of iron makes it a soft acid, while the electron-rich heteroatom of the inhibitor acts as a soft base. According to the HSAB principle, there is an interaction between soft acids and soft bases, leading to the adsorption of inhibitor molecules on the mild steel surface. Additionally, there could be the formation of an iron-inhibitor complex on the surface, which decreases the solubility of the protective layer formed and blocks the active sites, resulting in a decrease in the rate of metal dissolution (Karki, et al., 2021).

## CHAPTER 5

### 5. CONCLUSIONS AND RECOMMENDATION

#### 5.1 Conclusion

Methanol extract *Irsine herbiste* stem was successfully prepared and its corrosion inhibition efficacy was studied. Following conclusions were drawn from the experiments carried out:

- The corrosion rate and weight loss decreases upon increasing the concentration of inhibitor that subsequently signifies the increase in inhibition efficiency of the inhibitor. The inhibitor can work up to 24 h with significant inhibition efficiency.
- Electrochemical measurements suggest the alkaloids can work significantly efficiency and the mode of inhibition is mixed type. This is in accordance with the thermodynamic parameters.
- Adsorption isotherm, Activation energy value and entropy of the system reflects the good inhibition mode of the inhibitor by forming a stable mixed type of layer on the substrate surface.

#### 5.2 Recommendation

Research work on this topic was done depending on laboratory facilities which are successfully conducted in a given time frame under the supervision of the supervisor. Still, there are a few more topics on this plant that can be carried out in the future which are mentioned below:

- i. Other parts of this plant also can be studied for corrosion inhibition.
- ii. Extraction of fraction of alkaloids only and employed as inhibitor.
- iii. Potentiodynamic polarization and EIS for the determination of inhibition efficiency of the inhibitor can be done.
- iv. Surface of mild steel can be characterized by SEM, EDX, and XPS.

## CHAPTER 6

### 6.1 SUMMARY

The project utilized alkaloid extract from *Iresine herbstii*, a readily available plant in the local area, to synthesize a green corrosion inhibitor specifically targeting mild steel corrosion. This inhibitor offers several advantages, including cost-effectiveness, environmental friendliness, and high effectiveness in combating mild steel corrosion.

The corrosion inhibition efficiency of the *Iresine herbstii* extract was assessed using the weight loss measurement method and electrochemical methods. Results indicated that the inhibitor derived from the alkaloid extract of *Iresine herbstii* (SEIH) is not only suitable for mild steel but can also be effective for protecting other metals against corrosion.

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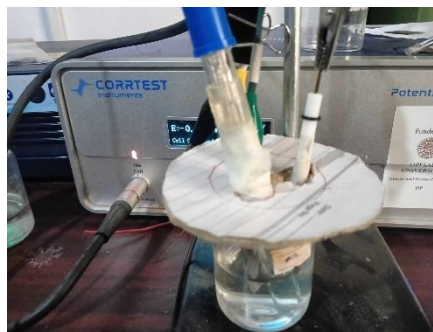
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## APPENDIX

Appendix 1: Pictures captured during sample extraction and weight loss.



Appendix 2: Pictures captured during Polarization



## EXPENDITURE

S.N.	Expenditure Title	Total Cost(NPs)
1	Mentor	5,000
2	Rotatory Shaker	20,000
3	Sample Collection	10,000
4	Chemicals and reagents	10,000
5	Instruments	16,000
7	Transportation	2,000
8	Printing and Binding	10,000
9	Miscellaneous	2,000
	Total	75,000

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**AS GREEN INHIBITOR FOR MILD STEEL CORROSION IN 1 M H2SO4 SOLUTION A** MINI RESEARCH 1  
REPORT **SUBMITTED**

TO OFFICE OF DEAN INSTITUTE OF SCIENCE AND TECHNOLOGY TRIBHUVAN UNIVERSITY KATHMANDU, NEPAL BY  
NARESH PRASHAD PANT

**AMRIT CAMPUS TRIBHUVAN UNIVERSITY, KATHMANDU, NEPAL** MENTOR: **ASST. PROF. Dr.** 1  
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