

**CORROSION INHIBITION BY ALKALOIDS OF
SOLANUM XANTHOCARPUM STEM FOR MILD STEEL
IN 1 M H₂SO₄ SOLUTION**

**A DISSERTATION WORK SUBMITTED FOR THE PARTIAL
FULFILLMENT OF THE REQUIREMENTS FOR THE MASTER
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This dissertation entitled, “**Corrosion Inhibition by Alkaloids of *Solanum Xanthocarpum* Stem for Mild Steel in 1 M H₂SO₄ Solution**” by, **Ms. Onisha Thapa** under the supervision of Assistant Professor Dr. Deval Prasad Bhattarai, Department of Chemistry, Amrit Campus, Tribhuvan University, Kathmandu, Nepal, and under co-supervision of Asst. Prof. Mr. Hari Bhakta Oli, Department of Chemistry, Amrit Campus, Tribhuvan University, Kathmandu, Nepal, hereby submitted has been approved for partial fulfillment of the requirement for completion of her Master of Science (M.Sc.) Degree in Chemistry. This dissertation has not been submitted to any other university or institution previously for the award of a degree.

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DECLARATION

I, Onisha Thapa, hereby declare that the work entitled “**Corrosion Inhibition by Alkaloids of *Solanum Xanthocarpum* Stem for Mild Steel in 1 M H₂SO₄ Solution**” submitted to the Institute of Science and Technology Tribhuvan University as partial fulfillment for the requirements of Master of Science Degree in Chemistry has been done by myself and has not been submitted earlier in part or full in this or any other form to any other university/institute, here or elsewhere for the award of any degree. All sources of information have been specifically acknowledged by reference to the authors or institutions.

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ABSTRACT

Alkaloids from *Solanum xanthocarpum* stem were successfully extracted. Extracted alkaloids were characterized by qualitative chemical tests and spectroscopic measurements. The effectiveness of the inhibitor was determined by weight loss and electrochemical measurement methods. From the weight loss measurement, maximum inhibition efficiency of 93.14% was achieved. The temperature effect study reveals that the inhibitor can work up to a temperature of 58 °C. Electrochemical measurement reveals that alkaloids could inhibit effectively up to 98.14% and serve as a mixed type of inhibitor. A study on kinetic parameters reflects that the inhibitor forms a potential barrier to protecting the MS. The values obtained from the thermodynamic parameters study reflect that the process is a spontaneous endothermic process.

Keywords: *Solanum xanthocarpum*, Extract, Green Inhibitors, Mild steel, Weight loss, Polarization.

LIST OF ABBREVIATIONS

DW	Distilled Water
MS	Mild Steel
HER	Hydrogen Evolution Reaction
CP	Cathodic Protection
AP	Anodic Protection
OCP	Open Circuit Potential
OM	Optical Microscope
PVC	Polyvinyl chloride
Θ	Fraction of Surface Coverage
C_{inh}	Corrosion Inhibitors
CR	Corrosion Rate
E_a	Activation Energy
ϕ_{corr}	Corrosion potential
FTIR	Fourier Transform Infrared Spectroscopy
I_{corr}	Corrosion current
mV	Millivolt
mV/s	Milivolt per second
ppm	Parts per million
SCE	Saturated Calomel Electrode
UV	UltraViolet

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CHAPTER 1

1. INTRODUCTION

1.1 Introduction of Corrosion

Corrosion is the electrochemical process that results in the damage or failure of metallic surfaces. The spontaneous natural phenomenon that deteriorates the material is also called corrosion. It is also known as a destructive attack on the surface of metallic materials by chemical and electrochemical reactions with its environment leading to their degradation (Revie, 2011; Uhlig & Revie, 1985). Corrosion is one of the most damaging and costly naturally occurring events seen today. Corrosion is defined as an undesirable deterioration of metallic materials (metals and alloys) by electrochemical and chemical reactions with their corrosive environments that adversely affect the properties of metals and alloys to be preserved. This definition is widely accepted by all the scientists of the world (Heusler et al., 1989). Corrosion is an all-too-common result of electrochemical reactions between materials and substances in their environment. Because of it, buildings and bridges can collapse, oil pipelines break, chemical plants leak, and bathrooms flood (Shaw & Kelly, 2006; Uhlig & Revie, 1985). The IUPAC recommended corrosion definition is very broad that comprises metallic as well as polymeric materials like plastics, rubbers, etc., and ceramics like concrete, bricks, etc. (Bhattarai, 2010; Krivián, 1991).

It is an electrochemical process, usually occurs not by direct chemical reaction of a metal with its environment but rather through the operation of coupled electrochemical half-cell reaction. A half-cell reaction is one of the two electrodes in a galvanic cell or a simple battery (McCafferty, 2010).

Anodic reaction:

The loss of electrons occurs as an anodic reaction,



'M' stands for metal and 'n' stands for the number of electrons that an atom of the metal will easily release.

Cathodic reaction

The cathodic reaction takes place according to the environment, the reactions are;



Hydrogen evolution from neutral water:



Oxygen reduction in neutral or basic solution



Metal reduction

1.2 Importance of Corrosion Study

Corrosion is a dominant factor that destroys the economy, depletes resources, and causes costly and untimely failures of plants, equipment, and components. Small carelessness regarding the corrosion phenomenon at the beginning of the implementation of any system plants and the process can cause major economic loss during their operation that corrosion is one of the major industries' scientific and technological problems (Sastri, 2012). Nowadays corrosion study is not only the interest of corrosion scientists but also of engineers and industrialists (Ghali et al., 2007; Lynes, 1951). In general, before the engineering materials are subjected to their applications, two things are to be significantly taken into mind. Firstly, under what conditions it is used, and secondly, how it is processed (McCafferty, 2010).

The economic factor is a very important motivation for much of the current research works in the field of corrosion. Losses from corrosion are many billions of dollars annually. According to a study, approximately \$276 billion, or 3.1 % of the gross domestic product (GDP) was lost by corrosion in the United States of America. The cost of corrosion is approximately 3-4 % of the gross national product of most industrialized countries (Koch et al., 2016). In the context of Nepal, there is about 4.2 % of the annual GDP is lost due to corrosion (Karki, 2020).

Corrosion has both direct and indirect effects on our daily life. The industrial importance of the corrosion study is mainly of three folds:

1. Economics: The prime motive of this factor is to reduce the cost associated with material loss
2. Safety: To improve the safety of the different operating equipment.
3. Conservation of precious materials: To conserve the precious metallic resources of the world.

The economic losses are divided into direct and indirect losses. The direct corrosion losses include the costs of replacing corroded structures and machinery or their components, such as condenser tubes, mufflers, pipelines, and metal roofing, including labor. The indirect corrosion losses are more difficult to assess and hence it is generally practiced that the indirect corrosion loss should be the same as that of the direct losses. Therefore, the total corrosion loss is generally estimated by doubling the estimated direct corrosion loss. Some examples of indirect corrosion losses are shutdown, loss of product, loss of efficiency, contamination of product, and overdesign (Heusler et al., 1989; Slepski et al., 2014).

1.3 Mild Steel

Mild steel contains a small percentage of carbon and is strong and easily worked but not readily tempered or hardened. It is also known as low-carbon steel that comprises a wide range of carbon depending on its source. Mild steel is composed of 0.21-0.27 % of C, 0.13-0.45 % Mn, 0.79-1.30 % P, 0.035 % S, and rest of iron. It is available with varying levels of formality (Chapagain et al., 2022). It has a relatively low tensile strength, but it is inexpensive and easy to form, and surface hardness can be increased through carburizing. Even though mild steel has wide applications, it is susceptible to corrosion due to its thermodynamics instability, especially in acidic mediums and hence, the study of mild steel corrosion phenomena has become important, particularly in acidic mediums (Parajuli et al., 2022). It is the material of choice because of its characteristic features and wide application in most chemical industries (Roberge, 2019).

Mild steel has a wide range of applications and is used almost everywhere ranging from constructions, automobiles to marine applications, pipelines, mining, chemical processing, nuclear power plants, fossil fuel power plants, petroleum production refining, and metal processing equipment, and so on (Slepski et al., 2014). Mild steel certainly is the cost-effective and most commonly used construction material, which is used extensively for centuries in many areas, intended for water pipes, boats, tank vessels, etc. It has been used because of its low nobility and structural defects, in interaction with other metals, corrodes practically in all environments (Alkais & Edrah, 2016).

1.4 Corrosion Control

The damage caused by corrosion can't be prevented but the cost of corrosion of material can be reduced significantly using corrosion control methods. The selection of the right materials for the targeted environment is the first and foremost method for corrosion control. The selection of appropriate materials in given corrosive environments is a key factor for corrosion control strategy. Selected metals or alloys for the suitable environmental conditions prevailing (composition, temperature, velocity) taking in to account mechanical and physical properties, availability, methods of fabrication, and the cost of structure (Bhattarai, 2010; Chapagain et al., 2022). Every metal and alloy has inherent and unique corrosion behavior that can range from the high resistance of noble metals. The corrosion resistance of a metal strongly depends on the environment to which it is exposed, like chemical composition, temperature, velocity, and so on (Davis, 2000).

Secondly, the use of coatings or painting is one of the most important methods of corrosion control metallic or polymeric materials that are applied to materials to decrease their resistance to corrosive degradation. The purpose of applying the coating may be decorative, functional, or both (Davis, 2000; Uhlig & Revie, 1985). The coating may be metallic, organic and inorganic however coatings with high resistivity such as epoxies, viny's, chlorinated rubber, etc. are the good ones. The increased electrical resistance of the coating offers a good method of corrosion prevention (Ghali et al., 2007; Revie, 2011).

Corrosion under buried structures is effectively controlled by the cathodic protection method (Palou et al., 2014). Cathodic protection suppresses the corrosion current that causes damage in a corrosion cell and forces the current to flow to the metal structure to be protected. Thus, corrosion or metal dissolution is prevented (Behpour et al., 2009). The impressed current method and the sacrificial anode techniques are most commonly employed for reactive steel or metallic substance to be protected (Ghali et al., 2007).

1.5 Corrosion Protection Using Inhibitors

Mild steel can be protected by using organic or inorganic species to the solution to inhibit the corrosion reaction in acid media, the most common amongst these are the inhibitors based on inorganic nitrites, nitrogen-based materials, and their derivatives

such as Sulphur containing aldehydes, acetylenic compounds, Sulphur containing compounds, thioaldehydes, and various alkaloids such as papaverine, strychnine, quine, and nicotine have been employed as inhibitors. In neutral media, benzoate, nitrite, chromates, and phosphate act as good inhibitors. Several organic compounds containing polar functions in their molecules have been reported as effective corrosion inhibitors for mild steel in acidic media (Solomon et al., 2010).

An inhibitor is a chemical substance that when added in a small concentration to an environment, effectively decreases the corrosion rate. Corrosion inhibitors are most used in the industrial sector due to their low cost and easy practice (Sastri, 1998, 2012). An inhibitor to be appropriate, few characteristics to be considered. Some of them are listed (Palou et al., 2014).

- Ability to protect the metal surface.
- High activity to be used in small quantities (ppm)
- Low-cost compound(s).
- Inert characteristics to avoid altering a process.
- Easy handling and storage.
- Preferably with low toxicity.
- Non-contaminant.
- It should act as an emulsifier.
- It should act as a foaming agent

1.6 Corrosion Inhibitors Application Area

Different acids of high concentration are extensively employed in various industrial processes such as acid pickling, descaling, cleaning of the boiler, acidization of the oil well, and petrochemical process which result in corrosion of metal (Karki et al., 2021). To protect the metals from an aggressive environment, many organic compounds have been studied and employed as corrosion inhibitors (Santana et al., 2020). Corrosion inhibitors are either synthetic or natural chemicals that, when added to an aggressive environment effectively decrease the corrosion rate. Corrosion inhibitors are needed to reduce the corrosion rates of metallic, alloy materials in different mediums (Adams et al., 2019). Interest in synthetic compounds is diminishing due to toxic effects on humans, animals, and aquatic life while natural

products have been prompted as an alternative to replace inorganic and organic inhibitors. Researchers on corrosion inhibition are concentrating on natural products as inhibitors mainly due to their low cost and eco-friendliness (Shrestha et al., 2019).

The anodic or passivating inhibitors are usually used in near-neutral solutions where sparingly soluble corrosion products such as oxides, hydroxide, or salts are formed. They facilitate the formation of passivating films that inhibit the anodic metal dissolution reaction. Chromates, nitrite, phosphates, and molybdates are some examples of anodic inhibitors (Sastri, 1998). However cathodic inhibitors suppress the cathodic reaction in a metal or alloy surface by oxygen or hydrogen reduction mechanism. Polyphosphates and zinc salts are examples of cathodic inhibitors (Sastri, 1998). Nevertheless, some of the long-chain heater elements containing organic compounds suppress both anodic and cathodic reactions to varying degrees. Such inhibitors are called organic inhibitors. Amine, amine salts, esters, imidazolines, mercaptans, amines, and ammonia derivatives, green inhibitors are examples of organic inhibitors (Sastri, 1998).

1.7 Green corrosion inhibitors

Synthetic organic chemicals operating as corrosion inhibitors in the petroleum and concrete industry were a major problem related to their toxicity. Corrosion scientists are not very satisfied with those inhibitors as they are generally not readily available, expensive, water-insoluble, and pollute the environment in their synthesis and application processes. The search for eco-friendly inhibitors is the major pivot for the use of natural products as environmentally friendly inhibitors (Ikeuba et al., 2013). So, interest in synthetic compounds as an inhibitor is diminishing and natural products have been prompted as an alternative to replace inorganic and organic inhibitors. Extracts of natural products contain various chemical compositions which are biodegradable and do not contain heavy metals or other toxic compounds (Uwah et al., 2013). The inhibition performances of plant extract are due to the presence of organic compounds such as alkaloids, flavonoids, polyphenol, tannins, nitrogen bases, phenolics, carbohydrates, protein as well as hydrolysis products, etc. (Africa, 2008; Thapa et al., 2019). These metabolites usually bear polar functional group-containing nitrogen, sulfur, or oxygen as heteroatoms, as well as triple or double conjugate bonds which act as major absorption centers on the surface of the metal. The lone pairs of

electrons in heteroatoms or π -electrons in conjugate bonds are responsible for the formation of layer over steel materials (Karki et al., 2020; Thapa et al., 2019).

1.8 *Solanum xanthocarpum*

It is commonly known as yellow berried nightshade a green pigment herb, which is woody at the base. The average height of this plant is 2-3 m found most in dry places as a weed on roadsides and wastelands (Singh, 2017). The systemic classification of the plant species is given below.

Systemic classification

Kingdom : Plantae

Division : Angiospermae

Class : Dicotyledoneae

Order : Cucurbitales

Family : Solanaceae

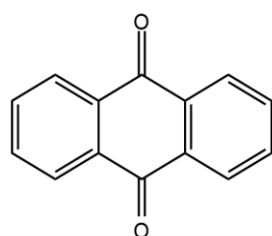
Genus : *Solanum*

Species : *xanthocarpum*

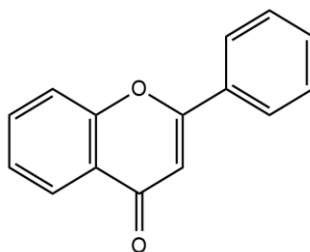


Figure 1.1: *Solanum xanthocarpum* (Leaves, Flower, Fruit, and Seeds)

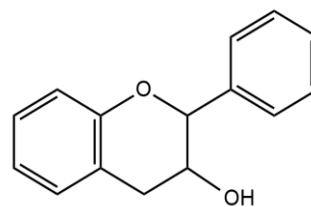
The whole part of this plant is used for medicinal purposes including fruits, roots, and stems (Parmar et al., 2010). It is reported that fruits contain several steroidal alkaloids like solanacarpine, solamargine, and other constituents like caffeic acid, diosgenin, campesterol, daucosterol, etc. The detailed study on this plant resulted in the isolation of solasonine and solasodine, β -sitosterol, and campesterol (Parmar et al., 2010). The bioactive chemical constituents found in the stem of this plant are anthroquinone, flavonoids, saponins, tannins, and alkaloids (Kumar, 2014).



anthraquinone



flavonoid



Tannin

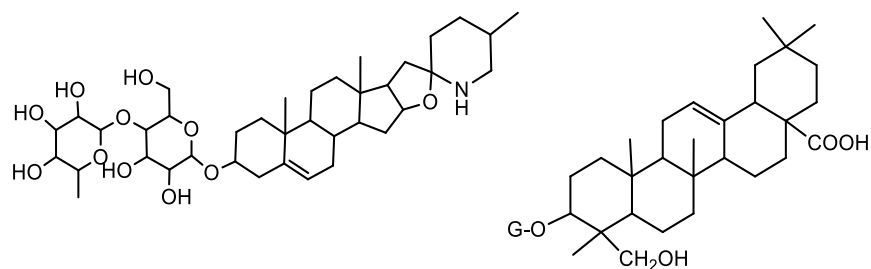


Figure 1.2: Structure of a) anthroquinone, (b) flavonoid, (c) tannin, (d) Khasianine alkaloid and (e) saponin

1.9 Corrosion Monitoring

Corrosion monitoring refers to corrosion measurements performed under different industrial and practical operating conditions. Corrosion monitoring is more complex than the monitoring of most other processes because there is no single measurement technique that will detect all of these various conditions. General corrosion rates may vary substantially, even over relatively short distances. Corrosion monitoring techniques are broadly classified into two types: direct and indirect methods. These few methods used in this experiment are explained below.

1.9.1 Weight loss method

The weight loss method is the best known and simplest of all known corrosion monitoring techniques. In this method, a sample specimen is exposed to corrosive environments for a specific period and subsequently removed for the weight loss method (Chapagain et al., 2022). This method applies to all environments solids, liquids, and gases. Similarly, corrosion deposits are observed and analyzed, visual inspection is the added advantage of this method. Since corrosion rate can be easily calculated.

1.9.2 Potentiodynamic polarization method

The polarization measurement was made to evaluate the corrosion current, corrosion potential, and Tafel slope. Potentiodynamic polarization involves the characterization of the sample by its current-potential relationship. A three-electrode corrosion system is used to polarize the electrode of interest. The current response is measured as the potential which is shifted away from the corrosion potential. When a potential, more positive than the corrosion potential is applied to the sample through an external source, otherwise known as anodic polarization, then the anodic current predominates over the cathodic current. As the anodic polarization increases, the cathodic current becomes negligible concerning the anodic current. Conversely, on the cathodic

polarization of the sample, the cathodic current predominates and the anodic current becomes negligible (Parajuli et al., 2022; Uwah et al., 2013).

Potentiodynamic polarization measurements are generally used in aqueous systems. The basic difference from the linear polarization technique is that the applied potentials for polarization are normally stepped up to levels of several hundred millivolts. When the potential more positive than E_{corr} is applied on WE through an external source anodic current predominates and as the polarization increases, the cathodic current becomes conversely, cathodic current predominates while the anodic current becomes negligible in cathodic polarization. This technique is very useful to detect localized corrosion for passivating alloys such as stainless steels, nickel-based alloys containing chromium, and other alloys. These polarization levels facilitate the determination of kinetic parameters such as the general corrosion rate and the Tafel constants. The formation of passive films and the onset of pitting corrosion can also be identified at characteristic potentials, which can assist in assessing the overall corrosion risk (Africa, 2008; Chapagain et al., 2022; Parajuli et al., 2022; Thapa et al., 2019).

1.10 Objectives of the Study

1.10.1 General objective

The major objective of this study is to develop eco-friendly corrosion inhibitors from natural products of Xanthocarpum(kantakari).

1.10.2 Specific objectives

The specific objectives of the present study are summarized as follows:

- a) Study the effect of inhibitor concentration on the inhibition efficiency by weight loss method.
- b) Study the Potentiodynamic Polarization behavior of mild steel in the presence and absence of an inhibitor.
- c) Study the correlation between the inhibition efficiency from polarization and weight loss data.
- d) Analysis of the plant extract and its component adsorbed onto the mild steel surface by FTIR.

CHAPTER 2

2. LITERATURE REVIEW

2.1 Green Inhibitors

Numerous organic compounds have been examined and are still being studied as an inhibitor to reduce corrosion. Based on these studies, concluded that organic molecules, particularly those containing N, S, and O, showed notable inhibitory efficacy. But distressingly, the majority of these substances are not only pricey but also harmful to the environment and living things.

Phytochemicals closely resemble their molecular and electronic properties with organic inhibitor molecules. They are fetching importance as they are readily available, renewable, and environmentally acceptable sources for a variety of inhibitors. They contain a variety of substances that have a high potential for inhibition that justifies their usage as corrosion inhibitors (E. Oguzie, 2007).

An intensive review work on green chemical for rust preventive surmise that natural substances rise as impressive rust inhibitors shortly due to their advantages as easy accessibility, eco-friendly on earth, and non-toxic, with the remark that the era of green corrosion inhibitors has already begun (Raja & Sethuraman, 2008), they discussed the function of inhibitor over the metal surface and application and limitations of natural corrosion inhibitor.

Extract of *Embilica officianilis* leaves (Saratha & Vasudha, 2010), *Terminalia chebula* (E. Oguzie et al., 2014), *Sapindus trifolianus* (Sanghavi et al., 1996), and *Accacia coniciann* (Haldhar et al., 2020) have all been studied for their anticorrosion properties. It is examined that the extracts of chamomile, half a bar, black cumin, and kidney bean for their ability to prevent the corrosion of steel in an acidic medium (Abdel-Gaber, 2006). These findings are very promising and attractive for corrosion scientists.

In the next experiment, Eddy et al. (2009), investigated the corrosion inhibition efficiency of ethanol extract of Musa species peel for mild steel corrosion. In the experiment, corrosion measurement experiments were monitored by hydrogen evolution and thermometric methods. The effectiveness of the inhibitor for corrosion inhibition was governed by inhibitor concentration, working temperature, immersion

time, pH of the working solution, and electrode voltage. Thermodynamic and adsorption isotherm study reflected the spontaneous adsorption of the inhibitor on the mild steel surface regulated by physical adsorption (Eddy et al., 2009).

2.2 Methanol Extract as inhibitor

The widely known fact is that almost all phytochemicals can be extracted using methanol that being so methanol extract could serve as an effective corrosion inhibitor. Methanol extract of *Azadirachta indica* as a green inhibitor was investigated using weight loss measurement and gasometric techniques as corrosion inhibitors for mild steel in acid media. They surmise that the extract could serve as an effective inhibitor, and the effectiveness rises with concentration (Okafor et al., 2007).

Punica granatum extract as an effective corrosion inhibitor to prevent the corrosion of brass in 1 N HCl was monitored by mass loss over time and at various temperatures. Based on the values of activation energy and free energy adsorption, the adsorption process was spontaneous with the physical adsorption process. Langmuir adsorption isotherms were best fitted for the whole inhibition process indicating the formation of a molecular layer of inhibitor on the metal surface (Rani & Selvaraj, 2010).

The inhibitory action of *Dodonaea viscosa* leaves extract (DVLE) on the mild steel corrosion in 1 M HCl and 0.5 M H₂SO₄ solutions has been studied by mass loss and electrochemical measurements. Obtained results reflected that DVLE worked well as a corrosion inhibitor in an acidic environment. The effectiveness of the inhibitor was found to be temperature dependent indicating the decrease in inhibition efficiency with the rise in temperature. However, on increasing the inhibitor's concentration, inhibition efficiency increased. Studies on the various thermodynamic and kinetic parameters reflected the spontaneous adsorption of the inhibitor molecules on the mild steel surface (Leelavathi & Rajalakshmi, 2013). The inhibitory impact of methanol extract of *Calotropis gigantea* leaf on mild steel corrosion in an HCl medium was investigated using weight loss measurement and electrochemical techniques. The effectiveness of the inhibitory action increased with increasing concentration. Findings of electrochemical measurement revealed that the inhibitor could work by suppressing both anodic and cathodic reactions (Desai, 2015). These inhibitors reduced the corrosion current densities by a mixed mode mechanism, thereby slowing the corrosion rate (Shyamala & Arulanantham, 2008).

2.3 Alkaloids as Inhibitor

Several researches on green corrosion inhibitors have been reported. Most of the reported findings are based on crude extract (methanol extract) and little research on alkaloid extracts has been done. The ability of *Ochrosia oppositifolia* leaf extract was investigated to inhibit corrosion of mild steel in an acidic medium. The isoreserpiline was the main alkaloid isolated from the extract (Raja, Qureshi, et al., 2013). Some recent works of literature are listed in table 1.

Table 1: Plants and their alkaloids as green corrosion inhibitors

S.N.	Plant Name	Plant Parts	Medium	Method	Efficiency, %	Reference
1.	Caulerpa racemosa	Alga alkaloids	1 M HCl	Weight loss Polarization EIS	83 80 85	(Kamal & Sethuraman, 2012)
2.	Ochrosia oppositifolia	Bark and leaves alkaloids	1 M HCl	Polarization EIS	94 89	(Raja, Fadaeinasab, et al., 2013)
3.	Neolamarckia cadamba	Bark and leaves alkaloids	1 M HCl	Polarization EIS	89 83	(Raja, Qureshi, et al., 2013)
4.	Garcinia kola	Seed alkaloids	5 M H ₂ SO ₄	Hydrogen evolution Polarization	99.4 98	(Ikeuba et al., 2013)
5.	Solanum melongena	Leaves alkaloids	Alkaline medium Sodium trioxocarbonate	Weight loss	81.1	(Ugi, 2014)
6.	Geissospermum laeve	Whole plant	1 M HCl	Polarization	92	(Faustin et al., 2015)
7.	Retama monosperma	Seed alkaloids	1 M HCl	EIS	94.4	(El Hamdani et al., 2015)
8.	Fauvolfia macrophylla		0.5 M H ₂ SO ₄ 1 M HCl	EIS Polarization	97, 93 92, 93	(Ngouné et al., 2019)
9.	Retama monosperma	Seeds alkaloids	1 M HCl	Electrochemical	94.42	(Sadik et al., 2020)
10.	Rhynchosylyus retusa	Rhizome alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	87.51 93.24	(Chapagain et al., 2022)
11.	Artemisia	Stem	1 M H ₂ SO ₄	Weight loss	92.58	(Parajuli et

	vulragis	alkaloids		Polarization	88.06	al., 2022)
12.	Solanum tubersum	Stem alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	90.79 83.22	(Parajuli et al., 2022)
13.	Alnus nepalensis	Bark alkaloids	1 M H ₂ SO ₄	Weight loss Polarization	71.94 78.48	(Dhakal et al., 2022)

There are several such publications on plant extract corrosion investigations. There are still many unknown sources of innovative and effective green corrosion inhibitors. In the future, efficient inhibitors will be made from compounds that are found in nature.

CHAPTER 3

3. MATERIALS AND METHODS

3.1 Collection of Plant and Preparation of powder

Frist, the plant was identified with the help of herbarium and named *Solanum xanthocarpum*. The stem of the plant was collected from Dang (Latitude: 28.1545° N, Longitude: 82.3235° E), Lumbini Province, Nepal. The collected sample was shade dried, and the dried sample was ground into fine powder.

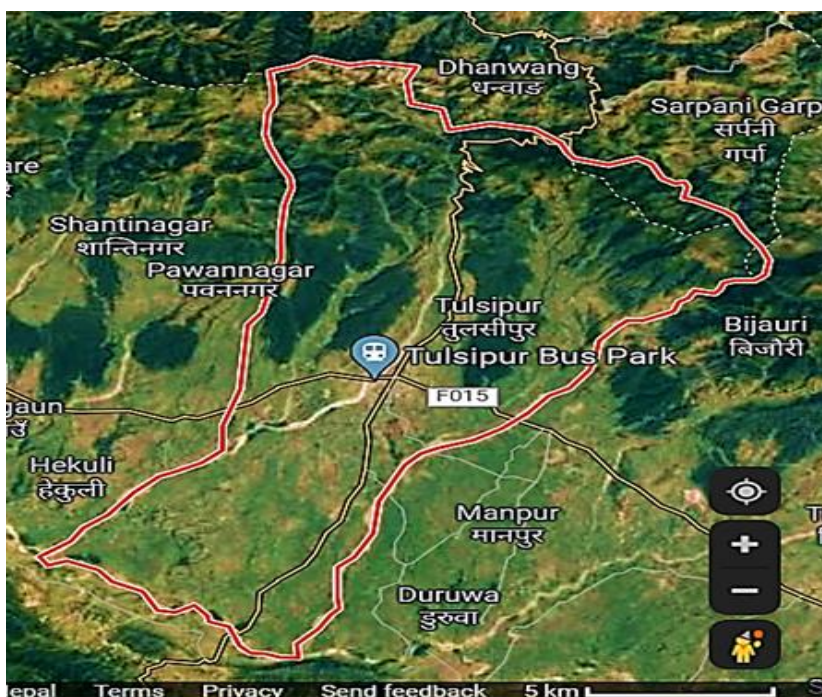


Figure 3.1: Google map of the sample collection area

3.2 Preparation of plant extract

Methanol extract of the powder was prepared through a cold percolation method for which 100 g of powdered sample was dissolved in 800 mL of methanol. The mixture was well stirred and allowed to stand for 15 days and then filtered. The filtrate was taken in a beaker and acidified with 5 % tartaric acid till the pH reached 3.0. Alkaloids got precipitated in the form of salt. The mixture was then filtered. The residue was taken and ammonium hydroxide solution was added to maintain pH 10. The alkaline solution was subjected to solvent extraction with an equal volume of dichloromethane (DCM). The DCM layer containing alkaloids was collected in a beaker and concentrated using a rotary evaporator. The concentrated solution was then dried using a water bath at 40 °C up to dryness.

3.3 Preparation of solution

3.3.1 Preparation of Caustic Medium

A stock solution of one molar sulphuric acid was prepared by diluting 55.6 mL of concentrated acid in a 1000 mL volumetric flask.

3.3.2 Inhibitor solution

Inhibitor stock solution was prepared by dissolving 1 g of alkaloids in 1 liter of one molar sulphuric acid solution and labeled as 1000 ppm. Other required concentrations of the inhibitor solutions (200, 400, 600, and 800 ppm) were prepared by diluting this stock solution.

3.4 Test for alkaloids

Chemical, as well as spectroscopic test methods, were applied to confirm the alkaloids. In the chemical test method, Mayer's test, Dragendorff's test, and Wagner's test were performed. The details of these three methods are given below.

3.4.1 Mayer's test

Mayer's reagent was prepared freshly by dissolving 1.36 g of mercuric chloride and 5 g of potassium iodide in 100 mL of water. A small amount (2 mL) of the extract was treated with Mayer's reagent (K_2HgI_4 , potassium mercuric iodide). An orange precipitate of potassium-alkaloid indicates the presence of alkaloids.

3.4.2 Dragendorff's test

For Dragendorff's Solution A: 3 g of Bismuth nitrate was taken and then dissolved in 4 N H_2SO_4 (8 mL) and for Solution B: 12 g of KI was dissolved in 18 mL distilled water. Then, solution A was mixed with solution B in a 50 mL volumetric flask with distilled water. A small amount (2 mL) of the extract was treated with Dragendorff's reagent and the appearance of an orange-red precipitate of potassium- alkaloid indicates the presence of alkaloids.

3.4.3 Wagner's test

Wagner's reagent was prepared by dissolving 2 g of iodine and 6 g of KI in 100 mL of distilled water. In a small amount (2 mL) of the extract, a few drops of Wagner's reagent were added. A reddish-brown precipitate of potassium-alkaloid indicates the presence of alkaloids.

3.5 Preparation of mild steel specimen

Mild steel coupons of A516 composed of 0.21-0.27 % of C, 0.13-0.45 % Mn, 0.79-1.30 % P, 0.035 % S, and the rest of iron were used in this study. The coupons were prepared from an MS sheet sourced from Katuwal Greel Udyog, Kathmandu, Nepal. Before each experiment, coupons were mechanically abraded with SiC paper of (100-1200) grits and stored in moisture-free desiccators. Anterior to each experiment, each sample was sonicated in ethanol and dried.

3.6 Weight Loss Measurements

Weight loss measurements were performed by immersing the MS coupons into the acid solutions in the absence and presence of different concentrations of inhibitor. Previous to each weight loss measurement, the dimension of each coupon was measured using a Vernier caliper. The initial weight of the sample was weighed by an electronic balance and subsequently immersed in the acid solution. After 0.5 h immersion, the sample was taken out, washed thoroughly with distilled water, dried, and reweighed. From the initial and final weights of the sample, the loss in weight was calculated.

Similar experiments were carried out for 1, 3, 6, and 24 h immersion time separately as well as for different concentrations of the inhibitors (200, 400, 600 and 800 ppm) for mentioned time. The temperature effect on the weight loss was studied for which immersion tests were carried out for 1 h at different temperatures (28, 38, 48, and 58 °C) separately. From these data, the corrosion rate, inhibition efficiency, and surface coverage by inhibitors are calculated using the formula mentioned in equations (1), (2), and (3).

$$\text{Corrosion rate} = \frac{K \times \Delta W}{ATD} \quad \dots (1)$$

$$\text{Inhibition efficiency (\%)} = \frac{(W_2 - W_1)}{W_2} \times 100 \quad \dots (2)$$

$$\text{Surface coverage} = \frac{(W_2 - W_1)}{W_2} \quad \dots (3)$$

Where, K =87600 (constant); ΔW = weight loss in gram; A= area in cm^2 , D = density in g/cm^3 ; T= time in an hour; W_1 and W_2 are the weight losses for mild steel in the presence and absence of inhibitor, respectively.

3.7 Electrochemical Measurement

The effectiveness of the inhibitor for mild steel corrosion and the electrochemical measurements were performed. The open circuit potential (OCP) and potentiodynamic polarization were carried out in a three-electrode system. The corrosion cell was arranged by taking MS coupon as a working electrode, graphite as a counter electrode, and saturated calomel electrode (SCE) as a reference electrode. Before polarization, the equilibrium potential was achieved by measuring OCP for 30 minutes.

3.7.1 Open circuit potential

The OPC is essential to explain the mode of inhibition by the alkaloid solutions. It was recorded for a better understanding of the corrosion behavior of the mild steel sheet in 1M H₂SO₄ solution at room temperature in the absence and presence of alkaloids as a corrosion inhibitor. The measurement was carried out using Hokuto Denko potentiostat (HA-151, Japan) in a three-electrode system. The OCP was measured in acid and different concentrations of inhibitor solutions for 30 minutes with each 2 minute interval for both as immersed and 1 h immersed conditions.

3.7.2 Potentiodynamic polarization

The potentiodynamic polarization method provides useful information about the corrosion current density and mechanism of corrosion. Evaluation of corrosion potential, current, and Tafel slopes for all solutions in both as-immersed and 1 h immersed conditions. The potentiodynamic polarization of the specimen was subjected just after the OCP recording in a three-electrode system. Both anodic and cathodic polarization in the potential window -0.8 to -0.2 V i.e. ± 300 mV from OCP with a scan rate of 60 mV/s and a maximum current of 10 mA was applied for both immersed and as-immersed conditions. Inhibition efficiency and a fraction of surface coverage were calculated using the formula in equations (4) and (5), respectively.

$$\text{Corrosion inhibition efficiency (IE, \%)} = \frac{I_{corr} - I_{corr}^*}{I_{corr}} \times 100 \quad (4)$$

$$\text{Fraction of surface coverage by an inhibitor } (\theta) = \frac{I_{corr} - I_{corr}^*}{I_{corr}} \quad (5)$$

Where, I_{corr} and I_{corr}^* are the current densities in the absence and presence of inhibitor, respectively.



Figure 3.2: Experimental set up for potentiodynamic polarization of mild steel

CHAPTER 4

4. RESULTS AND DISCUSSION

4.1 Chemical Test for Alkaloids

After successful extraction of alkaloids from the stem of *Solanum xanthocarpum*, further confirm by chemical test methods. The results obtained are in table 2. The formation of precipitate with characteristic color for each test is given in figure 4.1.

Table 2: Chemical test for alkaloids

S.N.	Experiment	Observation	Result	Inference
1.	Mayer's Test	Orange precipitate	Alkaloids presence	+Ve
2.	Dragendroff 's Test	Orange-red precipitate	Alkaloids presence	+Ve
3.	Wagner's Test	Reddish-brown precipitate	Alkaloids presence	+Ve

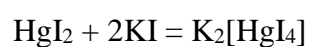
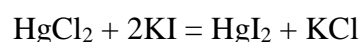


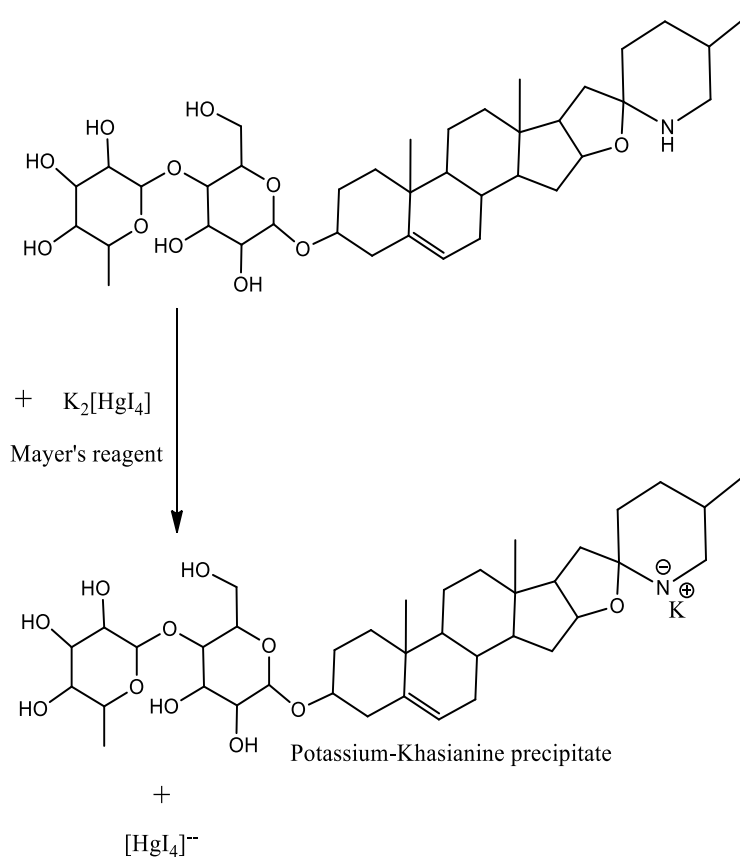
Figure 4.1: Test for alkaloids: a) Mayer's b) Dragendroff's and c) Wagner's test

The major alkaloid present in this plant is Khasianine. Reactions involved in the test for alkaloids are based on this Khasianine molecule as given below (Chapagain et al., 2022)

4.1.1 Mayer's test

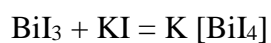
Mayer's reagent is potassium mercuric iodide (K_2HgI_4), which when added to the alkaloid solution, an orange precipitate of potassium-alkaloid was obtained indicating the presence of alkaloids. The possible reaction involved is given with the reference to Khasianine as below.

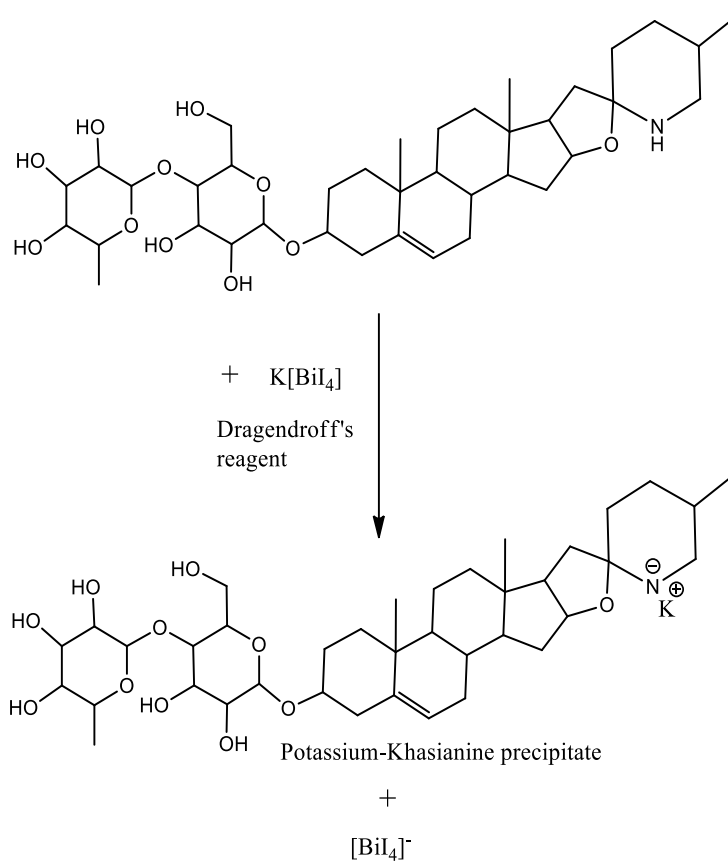




4.1.2 Dragendorff's test

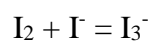
Potassium bismuth iodide is Dragendorff's reagent. When it is treated with alkaloids, the orange-red precipitate of potassium-alkaloid is obtained which indicates the presence of alkaloid. The possible reaction involved is given with the reference to Khasianine as below.

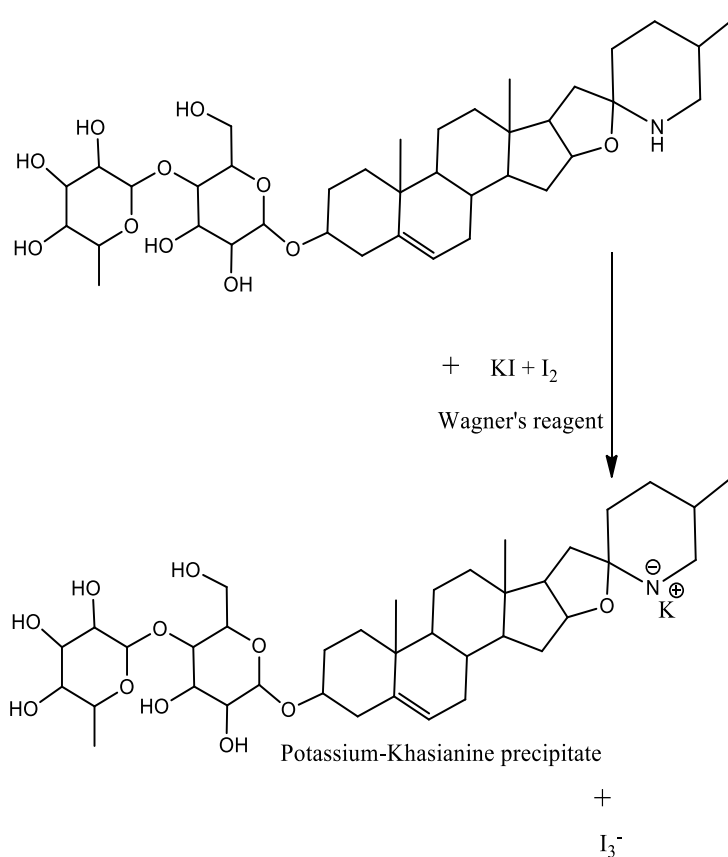




4.1.3 Wagner's test

Iodine when dissolved in potassium iodide solution then the solution contains potassium and tri-iodide ions. These ions, when treated with alkaloids the reddish-brown precipitate of potassium alkaloid is obtained which indicates the presence of alkaloids. The possible reaction involved is given with the reference to Khasianine as below.





4.2 FTIR Spectroscopic test for alkaloids

The spectrum of FTIR provides information about the molecular structure and conformation of organic molecules as well as different functional groups present in the sample. Spectra of alkaloids extracted from the stem of the *Solanum xanthocarpum* plant were documented using Perkin Elmer Spectrometer 10.6.2 version. The background correction was accomplished by using isopropanol. All the spectral data were collected from $450\text{-}4000\text{ cm}^{-1}$ cutoff range with 4 cm^{-1} resolution.

Recorded spectral lines are shown in figure 4.2 and the recognition of the absorption band is as follows. The broad peak observed in the $3500\text{-}3150\text{ cm}^{-1}$, range indicates the presence of the N-H and O-H stretching groups. As there is a combination of the O-H group and amine group, so a broad peak has appeared. The peak observed at 2957 cm^{-1} means C-H aliphatic asymmetry stretching vibration along with methyl and methylene groups. The peak observed at 1658 cm^{-1} belonging to the range ($1540\text{-}1870$) cm^{-1} determines the presence of the ketone group. The peak observed at 1065 cm^{-1} is due to the C-N stretching of amines. The peak observed at 576 cm^{-1} is due to N-H bending vibration of primary and secondary amines (Siverstein & Webster, 2006).

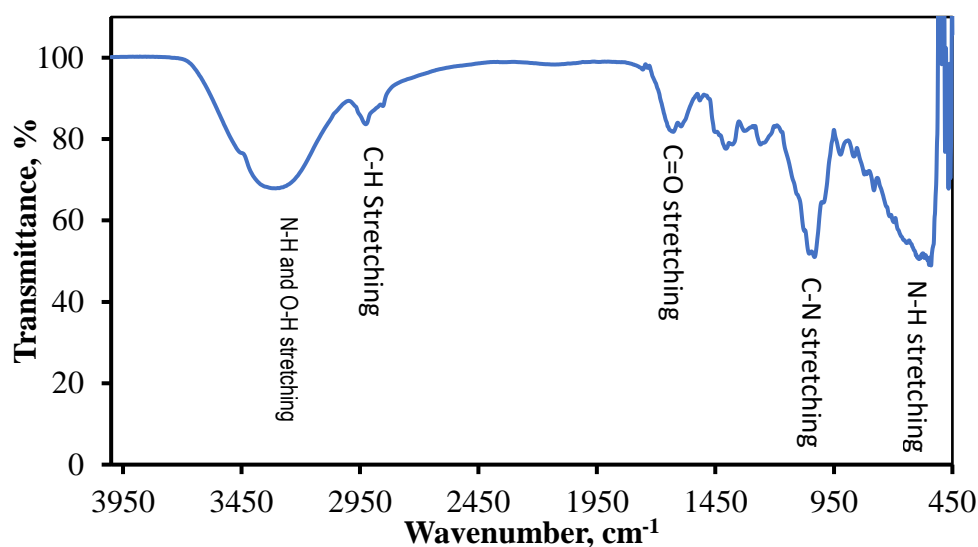


Figure 4.2: IR- spectrum of alkaloid extract of *Solanum xanthocarpum*

4.3 Weight loss Measurements

Immersion tests were performed to determine the inhibition efficiency of the alkaloids extracted from the *Solanum xanthocarpum* stem. The interaction of the MS with the surrounding aggressive environment when immersed in it leads to the formation of corrosion product. The formation of corrosion products gives rise to the decrease in the weight of MS. This decrease in weight is influenced by the nature of the surface, working temperature, and concentration of inhibitor solution.

4.3.1 Effect of immersion time on MS Weight loss

The effect of immersion time on weight loss was monitored at laboratory temperature (28 °C). The effect of immersion time on corrosion inhibition was studied by performing weight loss experiments at different time intervals (0.5, 1, 3, 6, and 24 h). The weight loss data of MS coupons immersed in acid in the absence and presence of inhibitor solutions for different immersion times are shown in table 3 and figure 4.3.

Table 3: Weight loss (g/cm^2) of the mild steel that was immersed in a caustic environment in the presence and absence of inhibitor at different immersion time

Time(hrs)	Weight loss (g/cm^2)				
	Acid	200 ppm	400 ppm	600 ppm	800 ppm
0.5	0.001203	0.000226	0.000236	0.000128	0.000153
1	0.003259	0.000601	0.000536	0.000309	0.000224
3	0.011619	0.001827	0.001941	0.001007	0.000834
6	0.019379	0.007889	0.0053	0.002787	0.001915
24	0.06024	0.03556	0.022307	0.013786	0.007887

The results obtained from this experiment showed that the presence of inhibitors in a caustic environment reduces the weight loss of MS. The weight loss in MS immersed in acid-only solution is high than that immersed in presence of inhibitor solutions. Initially, the weight loss is very small but after prolonged immersion, the weight loss of MS increased but the trend is the same. Taking an example, the weight loss of weight loss trend in this experiment manifest that inhibitors could inhibit and protect the MS surface from a caustic environment.

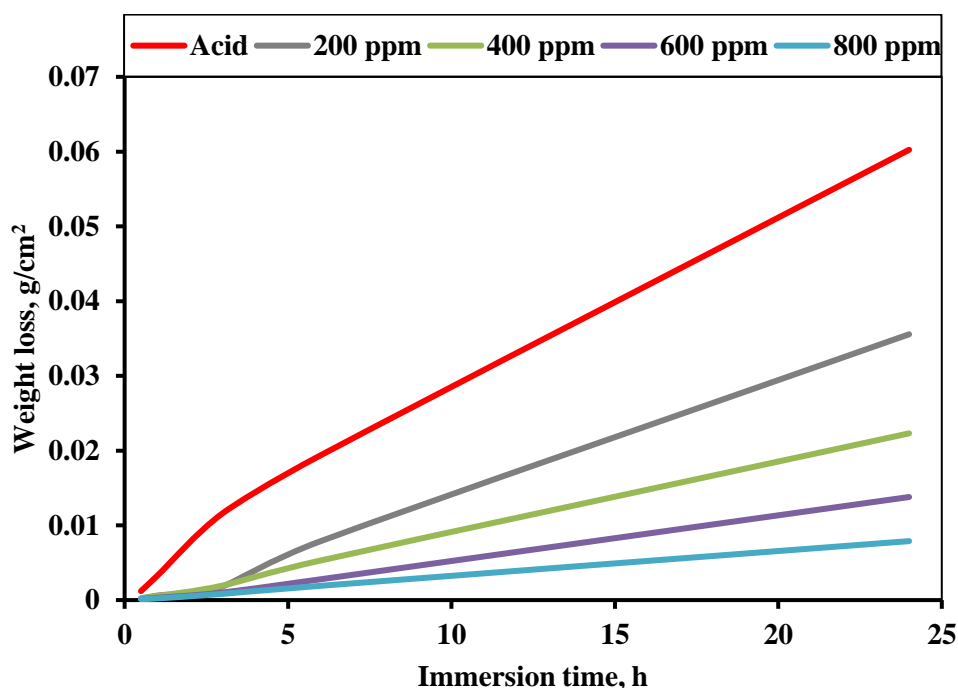


Figure 4.3: Weight loss (g/cm^2) of MS that was immersed in the acid solution in the absence and presence of inhibitor at different immersion time (h)

4.3.2 Effect of immersion time on Inhibition Efficiency

The inhibition efficiency of the inhibitor at different immersion time (h) is shown in figure 4.4. It is found that inhibition efficiency gradually increased up to 3 h immersion time, afterwards the inhibition efficiency remains almost constant for 800 ppm inhibitor solution. But, the case is different for 200 ppm inhibitor solution. In this case, the inhibition efficiency gradually decreased after 3 h immersion time. This implies that on continuous immersion, the desorption of inhibitor molecules from the MS surface as well as the formation of the Fe-inhibitor chelate complex could happen.

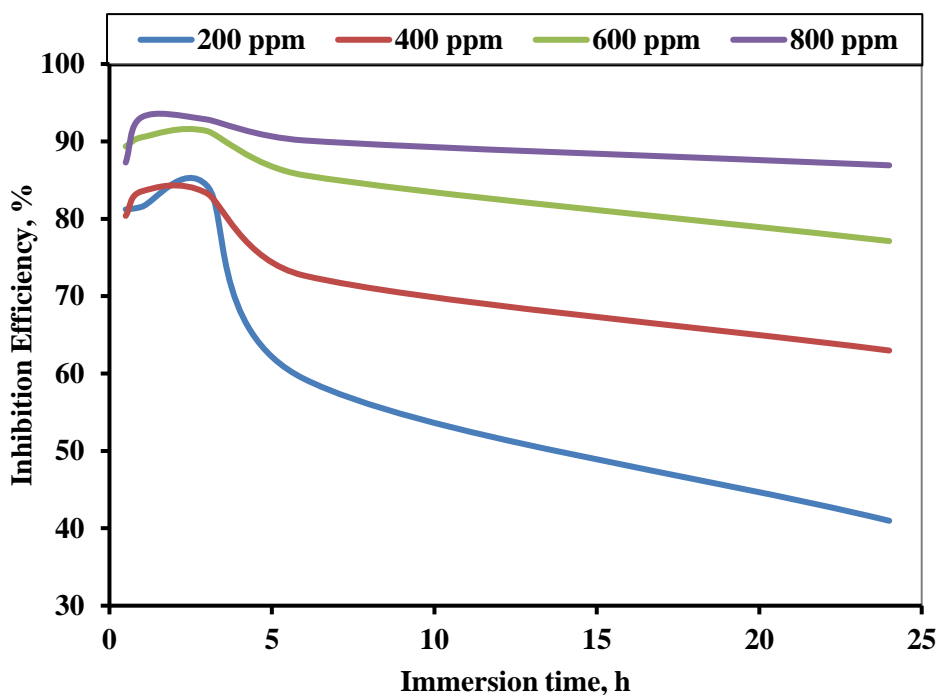


Figure 4.4: The inhibition efficiency of inhibitor in 1M H₂SO₄ solution for the corrosion of mild steel immersed at a different time interval.

4.3.3 Effect of inhibitor concentration in weight loss

Interaction of caustic medium with MS surface results in the formation of corrosion products consequently decreasing in weight of the MS. The addition of inhibitor solutions could withstand weight loss. The capability of the inhibitor solution to resist corrosion depends on its concentration. In this experiment, inhibitor solutions of different concentrations (200, 400, 600, and 800 ppm) were examined against MS corrosion. The weight loss is very high for the MS immersed in 200 ppm than the rest of all and minimum for that immersed in 800 ppm solution. This leads to conclude that the 200 ppm inhibitor concentration is inadequate to control MS corrosion for 24 h.

The loss in weight was very high for the MS immersed in the acid-only solution. Besides inhibitors, the loss in weight of MS gradually decreases with an increase in the concentration of inhibitors as shown in figure 4.5. It may be due to the adsorption of inhibitor molecules on the metal surface. The minimum weight loss was observed in the 800 ppm solution at 0.5 hour of immersion. The findings are following the immersion time effect.

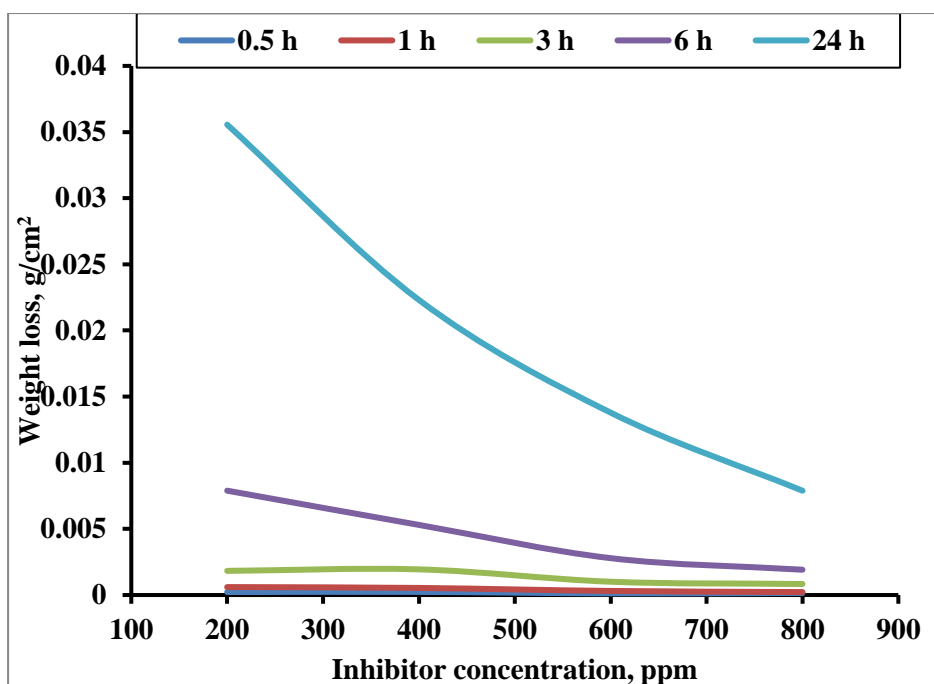


Figure 4.5: Variation of weight loss with different inhibitor concentrations at various times.

4.3.4 Variation in Inhibition Efficiency of Inhibitor concentration

The corresponding inhibition efficiency of the inhibitor apropos inhibitor concentration effect is shown in table 4 and figure 4.6. In the study, the inhibition efficiency was found higher at a higher concentration of inhibitor. The experiment was carried out in the different concentrations (0, 200, 400, 600, and 800 ppm) of inhibitor solutions in 1M H₂SO₄. The weight loss data indicates that the inhibition efficiency increases as the increase in concentration. It is because, at higher concentrations, a large number of molecules are available for adsorption onto the MS surface. The maximum inhibition efficiency is found to be 91.92% at 3 hours of immersion time in 800 ppm inhibitor concentration. This efficiency didn't remain constant on increasing immersion time. The minimum efficiency of 800 ppm inhibitor concentration was found to be 80.39% at 24 hour.

Table 4: Table showing the effect of inhibitor concentration on corrosion inhibition

Time (hrs)	Inhibition Efficiency %			
	200 ppm	400 ppm	600 ppm	800 ppm
0.5	81.21	80.38	89.35	89.58
1	81.55	83.55	90.53	93.14
3	84.28	83.29	91.34	92.82
6	59.29	72.65	85.62	90.12
24	40.97	62.97	77.11	88.37

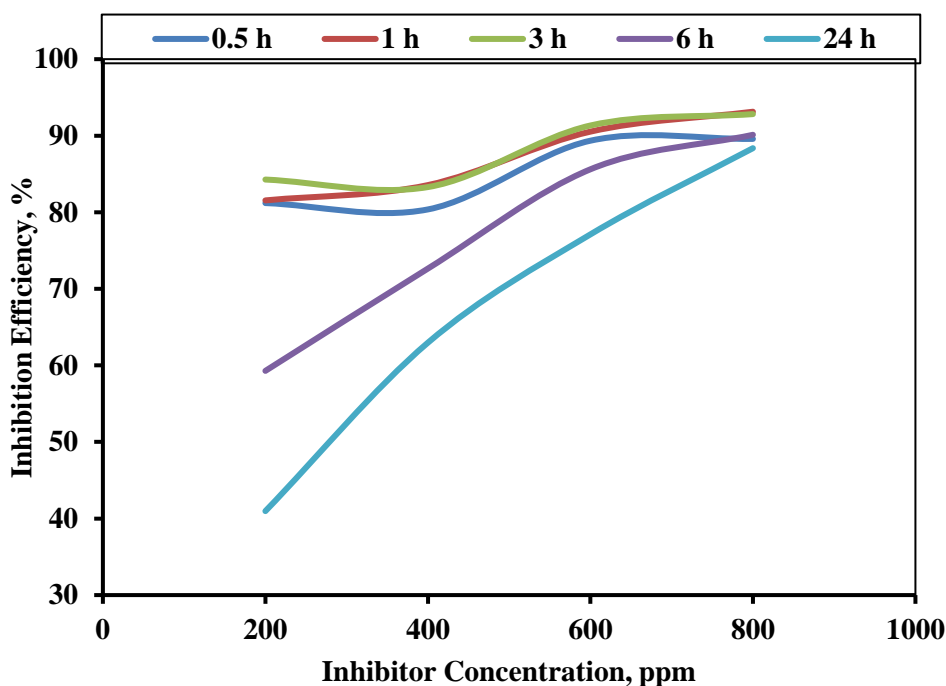


Figure 4.6: Variation of inhibition efficiency with the concentration of inhibitor solutions.

4.3.5 Effect of temperature on weight loss

Temperature greatly influences the corrosion rate as well as inhibitor action. The temperature effect on the inhibition process was studied by varying the working temperature. The weight loss of 1 h immersed MS sample in inhibitor solutions of different concentrations (200, 400, 600 and 800 ppm) was measured at different working temperatures (28, 38, 48, and 58 °C). The corresponding inhibition efficiency was measured by taking references to the weight loss of the 1 h immersed MS sample in an acid-only solution. All the observed data are tabulated in table 5.

Table 5: Variation in weight loss of the MS sample immersed for 1 h at different temperatures at different concentrations of inhibitor.

Temperature °C	Weight loss (g/cm ²)				
	Acid	200 ppm	400 ppm	600 ppm	800 ppm
28	0.003259	0.000601	0.000536	0.000309	0.000224
38	0.007701	0.002708	0.001603	0.000649	0.000624
48	0.018307	0.010639	0.004664	0.003032	0.002435
58	0.031575	0.030034	0.027119	0.013676	0.009221

It is found that the weight loss in acid-only solution is very high in comparison to others. The weight loss of MS was also found to be temperature dependent. From table 4, it is clear that weight loss is very high for the acid-only solution and very low for 800 ppm inhibitor immersed for 1 h at 28 °C. All the data recorded in this experiment are tabulated in table (2).

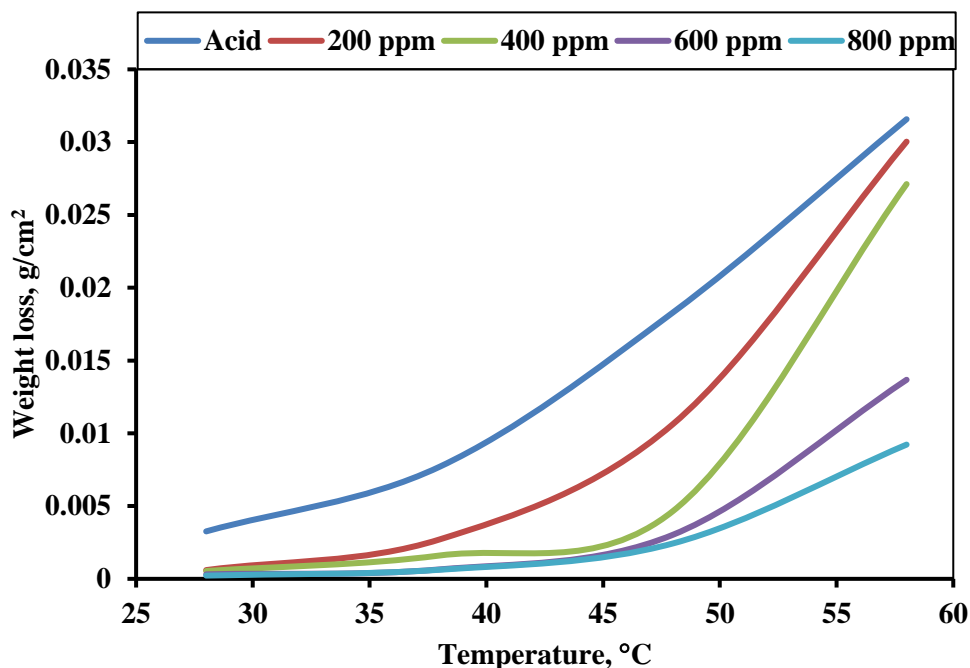


Figure 4.7: Variation of weight loss of MS with temperature in the presence and absence of inhibitor.

The variation of weight of MS in different concentrations of inhibitor with temperature is shown in figure 4.7. In the graph it is clear that the weight loss of MS increased gradually with increasing temperature, it is because the hydrogen evolution reaction takes place so rapidly at increasing activation energy with higher temperature.

4.3.6 Effect of temperature on Inhibition Efficiency

The efficiency of the inhibitor also varied with the temperature. The effect of temperature on corrosion inhibition with and without inhibitors was studied with different temperatures at a constant immersion time(1 h). Thus obtained data are tabulated in table 4. As in the table, it is clear that the inhibition efficiency is maximum at 28 °C. Above the 38 °C the inhibition efficiency at 200 ppm is almost negligible but at higher concentrations, it showed sufficient inhibition. Whenever it

has lower efficiency at normal temperature, it is stable up to higher temperatures also. It was very interesting that the methanol extract of *xanthocarpum* was not decomposed even at 50 °C temperature. So it can be claimed that the 800 ppm methanol extract prepared from *Xanthocarpum* can work at higher temperatures up to 48-50 °C. After these results, it can be concluded that the structural deformation of molecules may occur more than that of the desorption of molecules from the MS surface. This deformation leads to the decrease in inhibition efficiency of inhibitors for MS in the acidic medium above 58 °C.

Table 6: Inhibition efficiency (%) of the inhibitor of different concentrations on MS at different temperatures

Temperature °C	Inhibition Efficiency %			
	200 ppm	400 ppm	600 ppm	800 ppm
28	81.55	83.55	90.53	93.14
38	64.84	79.18	91.58	91.89
48	41.89	74.52	83.44	86.70
58	4.88	14.11	56.69	70.80

The inhibition efficiency dependence on temperature was studied and shown in the figure below. The maximum efficiency of 97.27 % at 800 ppm inhibitor at 38 °C temperature was achieved.

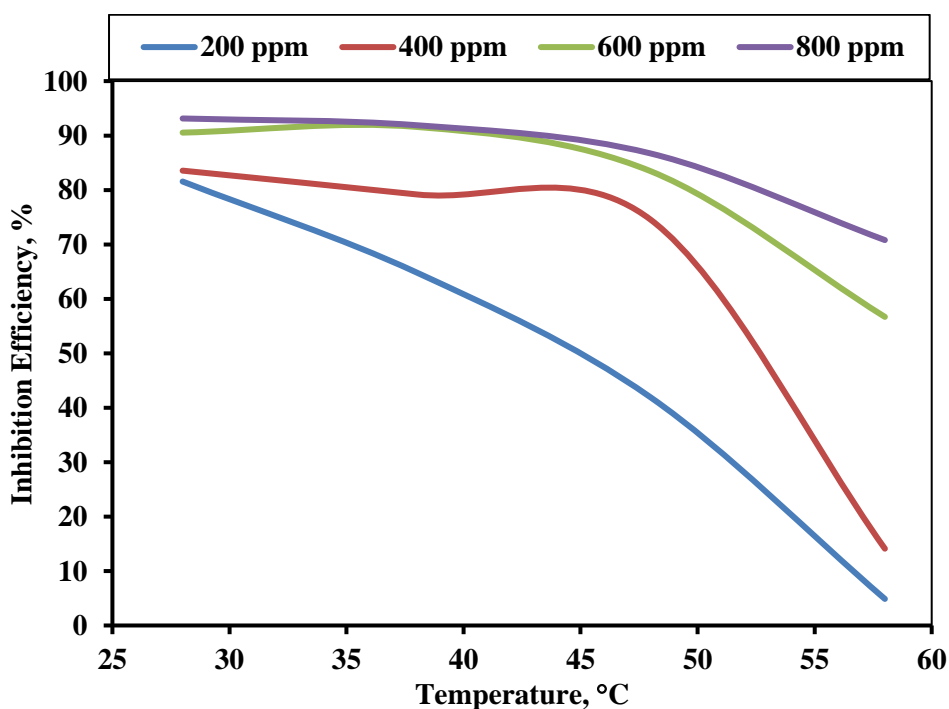


Figure 4.8: Effect of temperature on the Inhibition efficiency of the alkaloids in 1M H₂SO₄ for MS corrosion

4.4 Adsorption Isotherm

Adsorption isotherm in this corrosion inhibition process gives information regarding the number of alkaloids adsorbed on the MS surface concerning concentration at the working temperature. Adsorption isotherms were studied here by immersing MS coupons in inhibitor solutions of different concentrations at laboratory temperature. When MS coupons are immersed in the solution containing inhibitor, bulky organic molecules of inhibitors (alkaloids) are adsorbed on the MS surface. These adsorbed alkaloids blockage the active sites and the reaction between the metal surface and acid molecule was restricted. Alkaloids restrict both anodic and cathodic reaction progress (Ijuo et al., 2016; Shukla & Ebenso, 2011).

A very interesting phenomenon occurs in the adsorption of alkaloids on the MS surface. When MS is immersed in inhibitor solution, firstly, water molecules of the aqueous solution get adsorbed on the MS surface. Consequently, these adsorbed molecules are replaced by inhibitor molecules i.e. quasi-substitution process accelerated (Karki et al., 2018; Oli et al., 2021). The rate of corrosion decreased only when water molecules on the MS surface are completely replaced by inhibitors and the surface is covered. To identify the adsorption process, interactive nature of the molecules, the free energy of the adsorption, and thickness of the adsorptive layer, three adsorption isotherms: Langmuir, Freundlich, and Tempkin adsorption isotherms were determined (Andoor et al., 2021; Dhakal et al., 2022).

4.4.1 Langmuir adsorption isotherm

The linear form of the Langmuir adsorption isotherm equation (equation 6) was evaluated in this experiment. It could give information on whether the adsorption process is monolayer or multilayer.

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \quad \dots (6)$$

The linear relation between the fraction of covered surface (θ) and molar concentration (C_{inh}) should be established. Khasaine molecules are taken as a reference to calculate the molar concentration of the inhibitor solution (Oli et al., 2021). If the slope of the straight line obtained by plotting versus C_{inh} in equation 6 is

unity then it indicates the monolayer adsorption. And the value of the adsorption constant is obtained from an intercept.

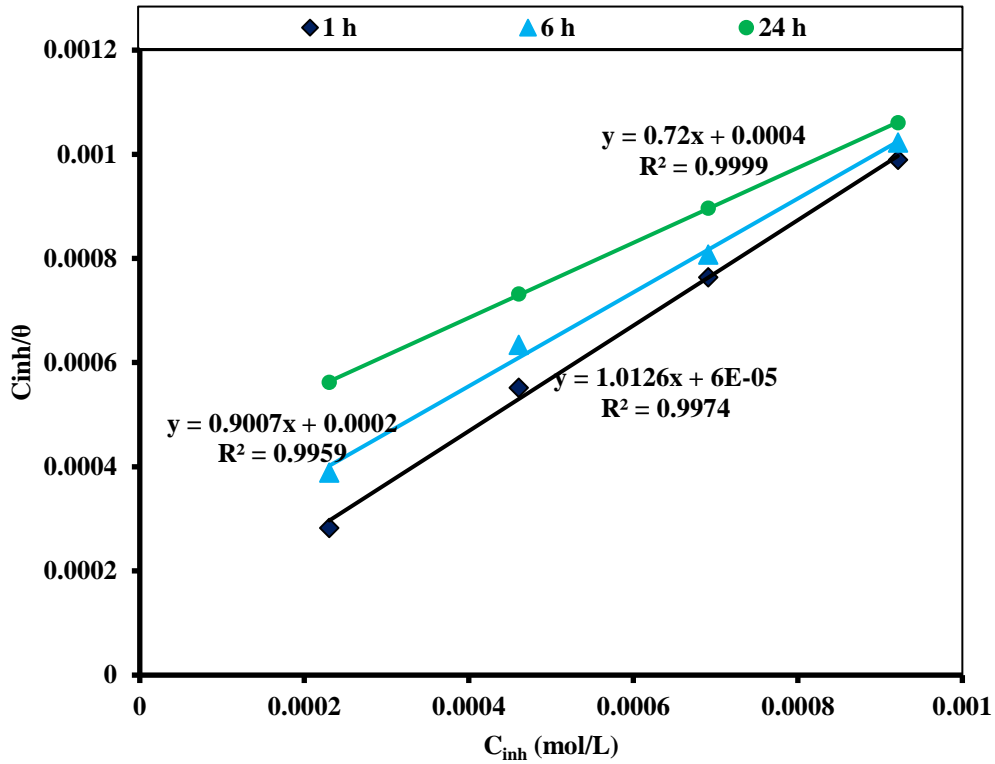


Figure 4.9: Langmuir adsorption isotherm plot for MS in 1 M H_2SO_4 with different concentrations of inhibitor.

Langmuir adsorption isotherm was studied by immersing MS coupons in inhibitor solutions of different concentrations for 1, 6, and 24 h intervals as shown in figure 4.9. The R^2 values for all the conditions are obtained by plotting C_{inh}/θ versus C_{inh} and all are nearly equal to unity indicating the adsorption process strongly follows the Langmuir model. The free energy of the adsorption was calculated using equation 7 and tabulated in table 6.

$$\text{Free energy of the adsorption } (\Delta G) = -RT \ln(55.5 K_{ads}) \quad \dots (7)$$

4.4.2 Freundlich adsorption isotherm

The inspection of the linear form of Freundlich adsorption isotherm for corrosion inhibition study is to determine the adsorption easiness. It can be studied to know how easily the adsorption of inhibitor molecules on the MS surface is. The Freundlich adsorption isotherm can be determined by using the following formula (Chapagain et al., 2022; Parajuli et al., 2022),

$$\ln \theta = \frac{1}{n} \ln C + \ln K \quad \dots (8)$$

This equation (8) is in the form of the equation of a straight line. A graph obtained by plotting $\ln \theta$ versus $\ln C$ gave the linear correlation coefficient (R^2) values of straight lines. The details are given in table 6.

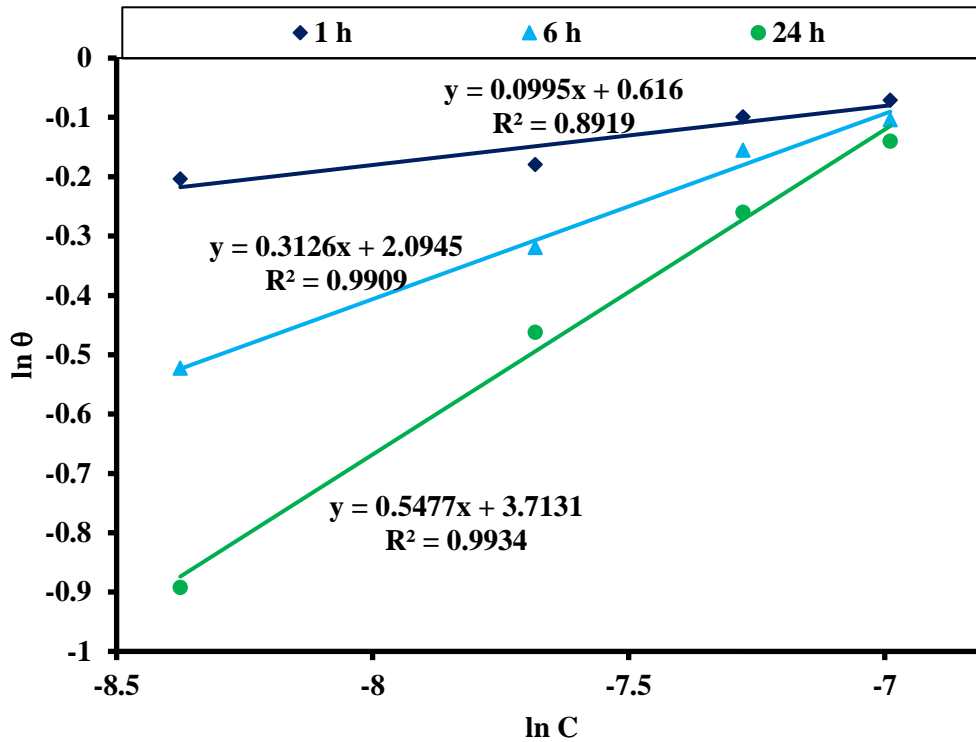


Figure 4.10: Freundlich adsorption isotherm plot for MS in 1 M H_2SO_4 solution.

4.4.3 Temkin adsorption isotherms

Temkin adsorption isotherm was evaluated in this experiment to evaluate the interaction of alkaloids with the MS surface. It helps to explain the corrosion inhibition mechanism and the nature of interaction taking place in the adsorbed layer. A linear form of the equation (9) was used to evaluate the isotherm by plotting θ against $\ln C$ (Chapagain et al., 2022),

$$\theta = -\frac{1}{2a} \ln C - \frac{1}{2a} \ln K \quad \dots (9)$$

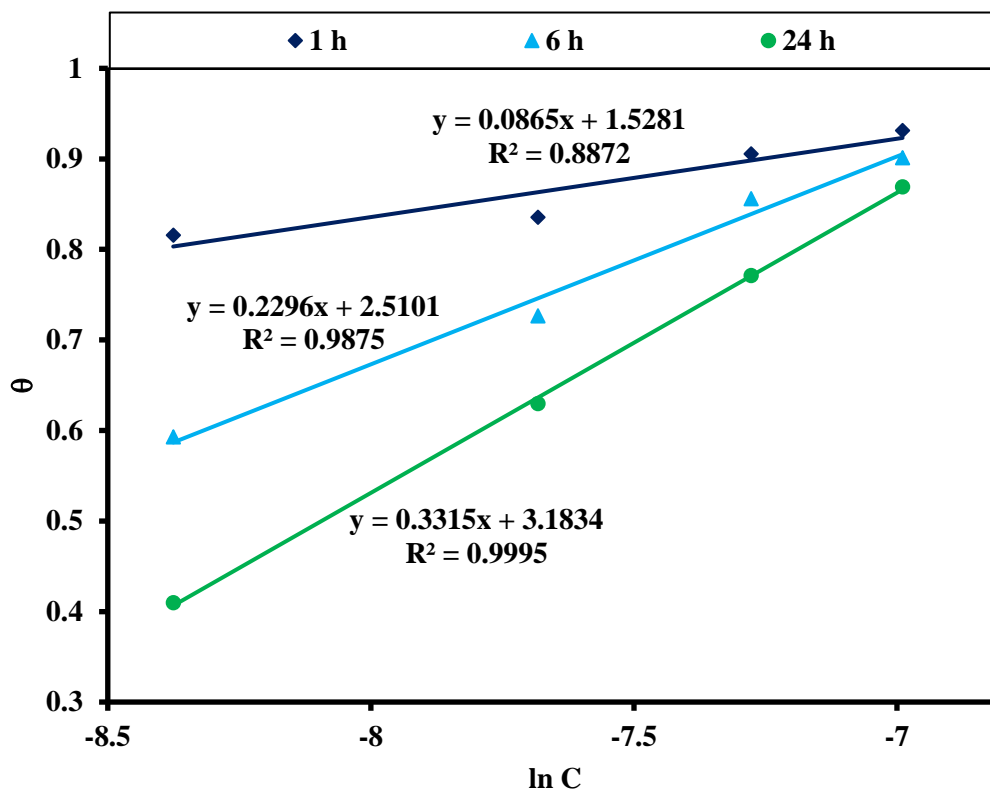


Figure 4.11: Temkin adsorption isotherm plot for MS in 1 M H₂SO₄ with different concentrations of inhibitor.

From the slope of the straight line in figure 4.11, the molecular interaction parameter (a) was calculated and tabulated in table 6.

The values of slope and intercept have their usual significances. The R² value gives the correlation between the measured values and hence helps to check the linearity range to check whether the measured values are acceptable. The R² values obtained from all three adsorption isotherms are listed in the table (6). From the findings, it is clear that the adsorption of inhibitor on MS surface obeys the Langmuir adsorption model for every immersion time. For Freundlich and Temkin adsorption isotherm, the correlation coefficient deviated for small immersion time however obeys for prolonged immersion time.

Table 7: Different parameters obtained from three different adsorption isotherms

Isotherm	Graph	Immersion time (h)	Slope	Intercept	R ²	K _{ads}	-ΔG (kJ/mol)
Langmuir	C/θ vs C	1	1.0126	0.00006	0.9974	16666	34.37
		6	0.9007	0.0002	0.9959	5000	31.36
		24	0.72	0.0004	0.9999	2500	29.63
Freundlich	ln θ vs ln C	1	0.0995	0.616	0.8919		
		6	0.3126	2.0945	0.9909		

		24	0.5477	3.7131	0.9934		
Temkin	Θ vs $\ln C$	1	0.0865	1.5281	0.8872		
		6	0.2296	2.5101	0.9875		
		24	0.3315	3.1834	0.9995		

4.5 Activation energy and Corrosion Kinetics

The activation energy of the reaction in the presence and absence of inhibitor in an electrochemical cell can be explained by rearranging the Arrhenius equation. The activation energy of the reaction is related to corrosion rate (Karki et al., 2018; Oli et al., 2021),

$$\log(CR) = \log A - \frac{Ea}{2.303RT} \quad \dots (10)$$

Where A is the Arrhenius pre- exponential constant, T is the absolute temperature. Equation (10) reveals that the activation energy of the reaction is equal to the slope of the Arrhenius plot i.e. a plot obtained between logarithms of corrosion rate with $\frac{1}{2.303RT}$ along axes.

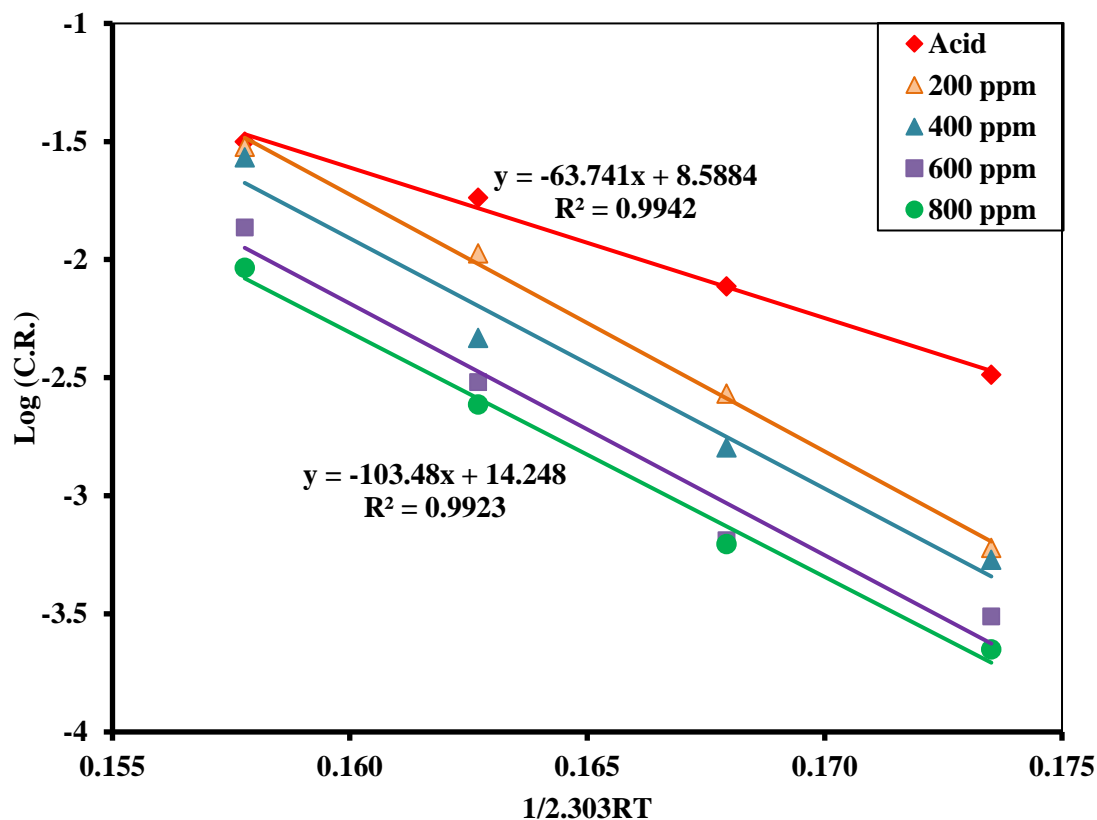


Figure 4.12: Arrhenius plot for MS in 1M H₂SO₄ with and without inhibitor

4.6 Thermodynamics of Corrosion and Mode of Inhibition

Enthalpy and entropy of the system can be calculated by using the transition state equation, an alternative form of the Arrhenius equation (Ijuo et al., 2016; Karki et al., 2018; Oli et al., 2021),

$$\log\left(\frac{CR}{T}\right) = \log\frac{R}{hN} + \frac{\Delta S^\circ}{2.303 R} - \frac{\Delta H^\circ}{2.303RT} \quad \dots (11)$$

Where, h is plank's constant, 6.6261×10^{-34} Js, and N is Avogadro's number, 6.0225×10^{23} mol⁻¹.

Enthalpy of activation (ΔH°) is obtained as the slope of a straight line by plotting the $\log \frac{CR}{T}$ versus $\frac{1}{2.303RT}$ in the equation and the entropy of activation (ΔS°) can be calculated from its intercept.

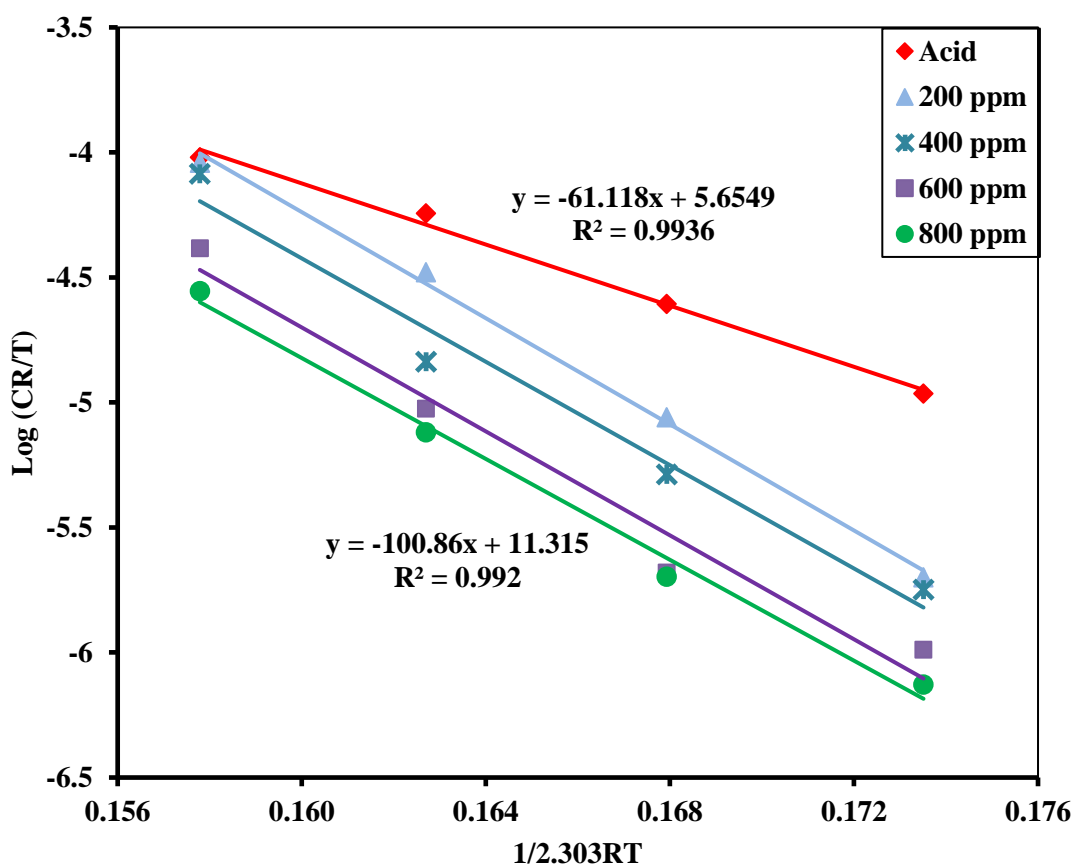


Figure 4.13: Transition state plot for MS in 1M H₂SO₄ with and without inhibitor.

Table 8: Activation parameters of the MS dissolution in 1 M H₂SO₄ without and with inhibitor.

Electrolyte	Log(A)	Ea(KJ/mol)	ΔH° (KJ/mol)	Ea- ΔH°	ΔS° (J/mol/K)
Acid	8.5884	63.74	61.12	2.62	-89.304
200 ppm	15.671	108.72	106.1	2.62	46.298
400 ppm	15.044	105.96	103.34	2.62	34.312
600 ppm	14.861	106.55	103.92	2.63	30.808
800 ppm	14.248	103.48	100.86	2.62	19.071

4.7 Electrochemical measurements

4.7.1 Information obtained from OCP

OCP measurement is one of the simplest concepts for indirect corrosion monitoring. OCP is the mixed potential achieved as a result of the redox reaction. It is also known as electrode potential when no net current flow through the external circuit of the electrochemical cell. It is not mandatory but generally measured in three electrodes system by using a potentiostat as a voltage between the working electrode (metal itself) and the reference electrode (SCE). The variation of OCP with time can be measured by determining the voltage difference between an MS immersed in a sample solution and as an appropriate reference electrode along with the counter electrode (Bhattacharai, 2008).

The variation of OCP of mild steel immersed in 1M H₂SO₄ and inhibitor solution was studied by monitoring changes in corrosion potential (ϕ_{corr}) with time. The OCP changes of mild steel were measured for 30 min at room temperature for both immersed and as-immersed conditions.

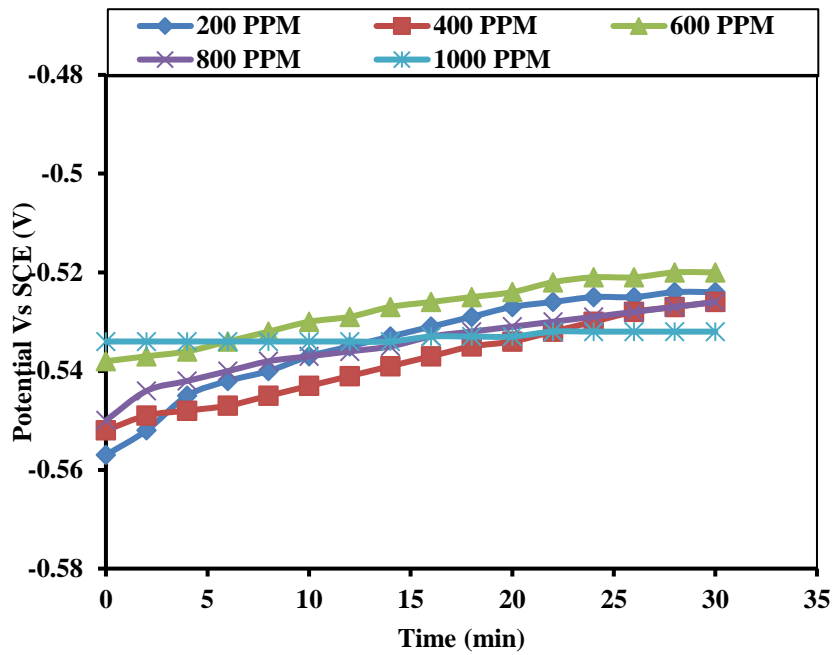


Figure 4.14: Variation of OCP with the time of immersion of mild steel in different concentrations of inhibitor in 1M H_2SO_4 measured for as-immersed condition

OCP recorded for an as immersed sample and the immersed sample is shown in figures 4.14 and 4.15, respectively. Initially, the potential shifted to more positive in both immersed and as-immersed conditions indicating that the positive charge is accumulated on the MS surface. The shifting of potential from OCP to more positive indicates the formation of a protective layer by inhibitor molecules in acid solution on the MS surface, (i.e. Passivation) that limits the interaction of aggressive ions with the MS surface (Chapagain et al., 2022). The OCP shifting is less than 50 mV indicating that alkaloids protect MS by inhibiting both anodic and cathodic reactions i.e. mixed inhibition mechanism.

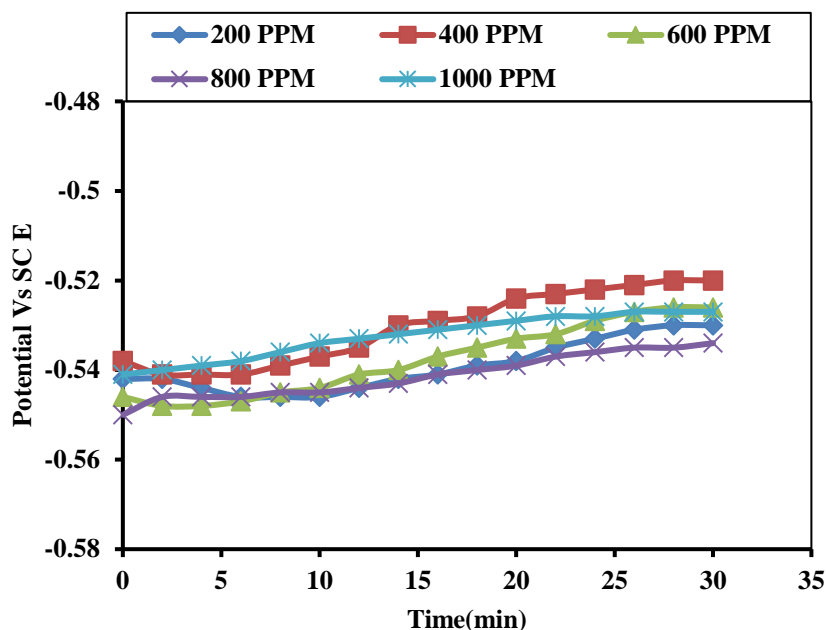


Figure 4.15: Variation of OCP with the time of immersion of mild steel in different concentrations of inhibitor in 1M H₂SO₄ measured after 1 h immersion in solutions.

4.7.2 Polarization measurement of as-immersed MS sample

The polarization measurement of the MS sample in an as-immersed condition was carried out in a corroding medium in the presence and absence of an inhibitor. In the polarization measurement, different inhibitor concentrations (200, 400, 600, 800 and 1000 ppm) were used and 300 mV potential was applied for both anodic and cathodic directions. The corrosion current density in acid-only solution was found to be 0.0477 mA/cm² which gradually decreased with the addition of different concentrations of the inhibitors. It decreased up to 0.006 mA/cm² for 1000 ppm inhibitor solution. The current density decreasing trend is very interesting. It decreases slightly for a lower concentration of inhibitors (200 and 400 ppm), but notably for higher concentrations of inhibitor (600, 800, and 1000 ppm) solutions. The data obtained by polarization measurement for as immersed samples are tabulated in table 8.

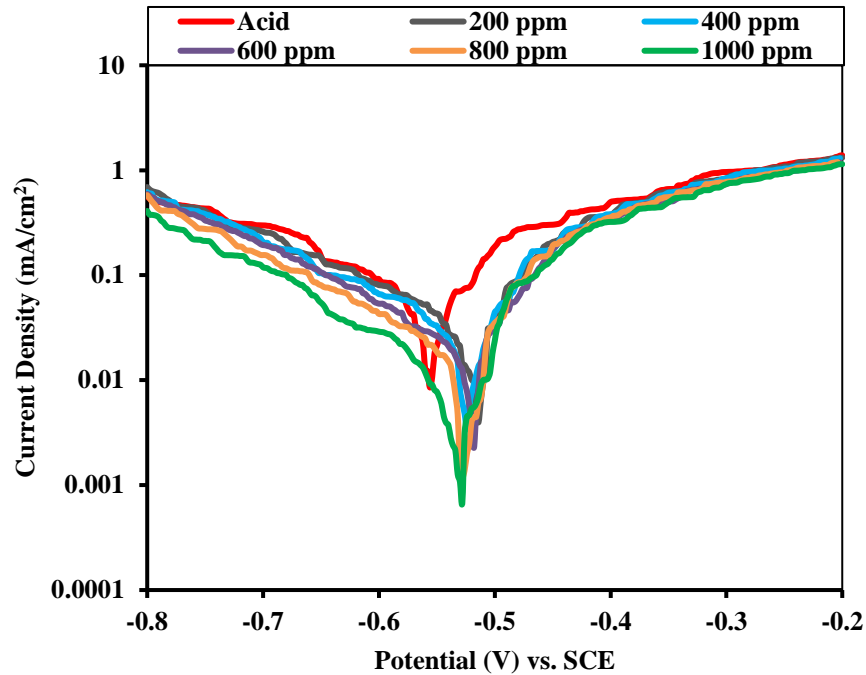


Figure 4.16: Potentiodynamic polarization curves for mild steel in 1M H_2SO_4 containing different concentrations of inhibitor measured at as-immersed conditions.

The polarization curves (Figure 4.16) indicate the decrease in current density on the addition of inhibitor. It means the MS surface is covered with inhibitor molecules ensuing less chance of interaction with corrosive media. This implies that the inhibitor molecules blockade the reaction pathway.

Table 9: Table showing OCP, current density, anodic and cathodic slope, and inhibition efficiency for as-immersed sample.

Medium	OCP	I _{corr}	Anodic Slope	Cathodic slope	Efficiency (%)
Acid	0.552	0.0477	12.83	5.46	
200 ppm	0.521	0.0318	10.87	5.19	33.33
400 ppm	0.518	0.0209	21.15	5.7	56.18
600 ppm	0.521	0.016	13.78	6.52	66.46
800 ppm	0.526	0.0144	17.06	7.3	69.81
1000 ppm	0.532	0.006	15.01	4.15	87.42

4.7.3 Polarization measurement of 1h-immersed MS sample

Measurement of corrosion current density and the OCP of the MS samples dipped in acid only and inhibitor solutions were carried out in the potential window of -0.8 to -0.2 V. It is found that the current density for the MS dipped in acid only solution is very high 0.102 mA/cm² that decreased drastically for MS dipped in inhibitor solutions. The inhibitor efficiency, cathodic and anodic slope, OCP and corrosion current densities obtained by potentiodynamic polarization measurement of MS in 1 h immersed condition are tabulated in table 9.

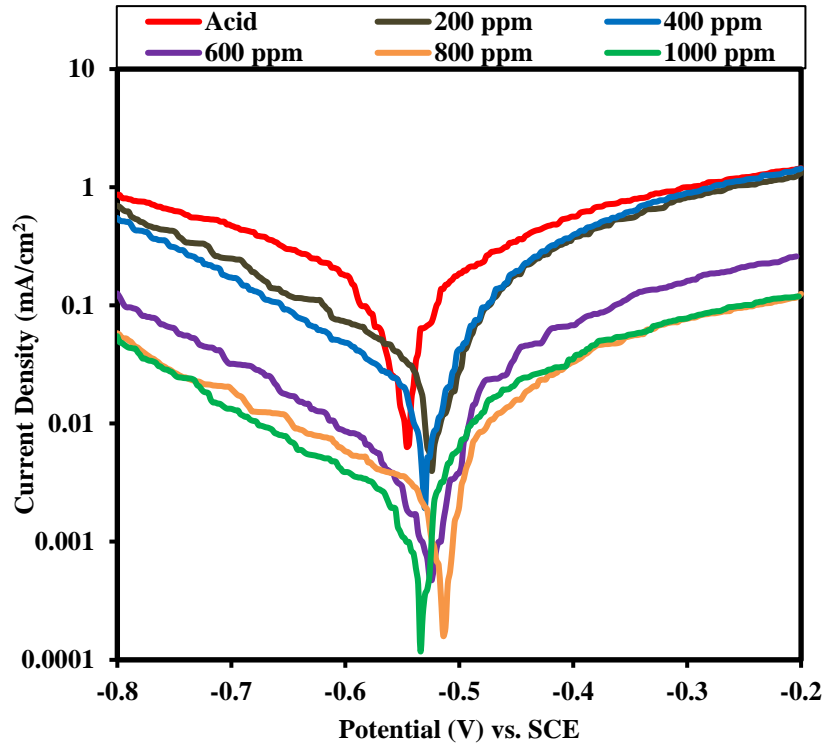


Figure 4.17: Potentiodynamic polarization curves for mild steel in 1M H_2SO_4 containing different concentrations of inhibitor immersed for 1 h condition.

The decrease in the current density in presence of an inhibitor is due to the resistance of inhibitor solution toward corrosion reaction. Hence like in the as-immersed condition, the current density decrease is due to coverage of MS surface by inhibitor molecules. The nature of anodic and cathodic slopes has not been changed for acid only and inhibitor solutions that indicate the reaction pathway for the corrosion inhibition have not been changed.

Table 10: Table showing the OCP, current density, anodic and cathodic slope, and inhibition efficiency for immersed sample.

Medium	OCP	I _{corr}	Anodic slope	Cathodic slope	Efficiency (%)
Acid	0.545	0.102	15.42	4.29	
200 ppm	0.52	0.027	10.65	5.6	73.53
400 ppm	0.529	0.017	14.19	5.9	83.33
600 ppm	0.526	0.0029	25.26	6.32	97.16
800 ppm	0.52	0.0025	18.5	4.9	97.55
1000 ppm	0.537	0.0019	12.5	4.8	98.14

4.7.4 Inhibitor efficiency from polarization measurement

The inhibition efficiency of the inhibitor in varying concentrations was measured for both as-immersed and immersed conditions by polarization method. The inhibition efficiency is very small for a 200 ppm inhibitor solution in an as-immersed condition that speeds up and works effectively from a 600 ppm inhibitor solution. It is found that inhibition efficiency increases with an increase in the concentration of the inhibitor solution. This is due to the increase in the fraction of surfaces covered by inhibitor molecules. The maximum efficiency was found as 87.42 % and 98.14 % at 1000 ppm concentration of the inhibitor solution in 1M H₂SO₄ for as-immersed and immersed conditions respectively.

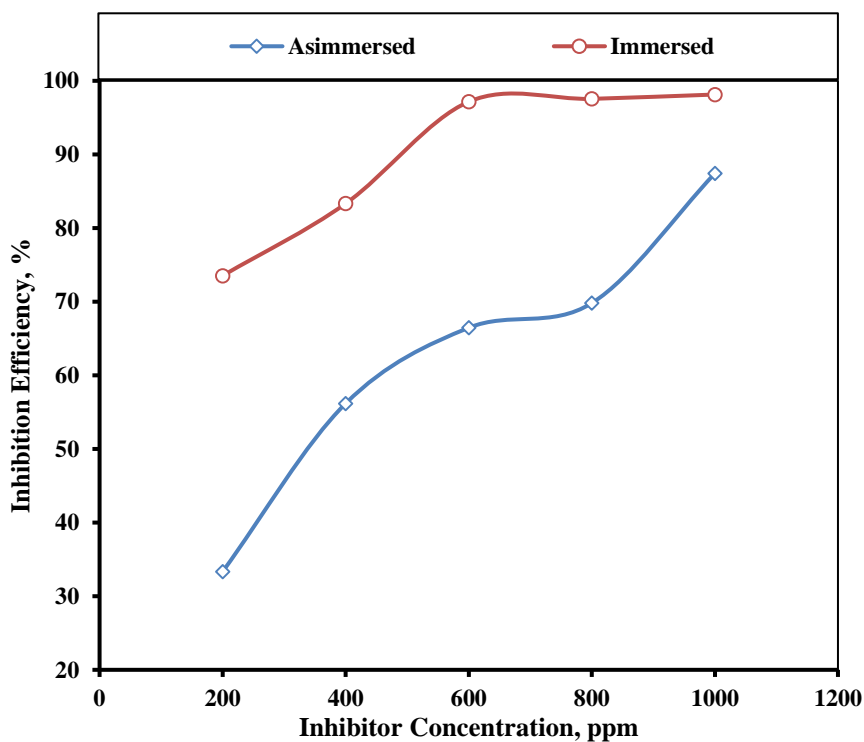


Figure 4.18: Inhibition efficiency of inhibitor obtained from the polarization of as-immersed and 1 h immersed MS sample in 1M H₂SO₄ in the presence and absence of inhibitor.

4.8 Surface Morphological Study

An optical microscopic image of the polished MS as well MS coupons immersed in the acid and 1000 ppm green inhibitor solutions have been captured at Professor Yadav's Laboratory, Central Department of Chemistry. All the images were captured by high definition optical microscope (RXLR-4, Radical Scientific, India). The optical image of MS surfaces that are dipped in acid and the presence of green inhibitors in different

magnifications are shown in figure 4.19. The formation of corrosion products (small pits and rusts) is observed in acid than in presence of inhibitor, while no corrosion products seem in polished (undipped) MS coupons. This indicates that the alkaloids form a protective layer over the MS surface and hence protect it from corrosion (Chapagain et al., 2022; Parajuli et al., 2022).

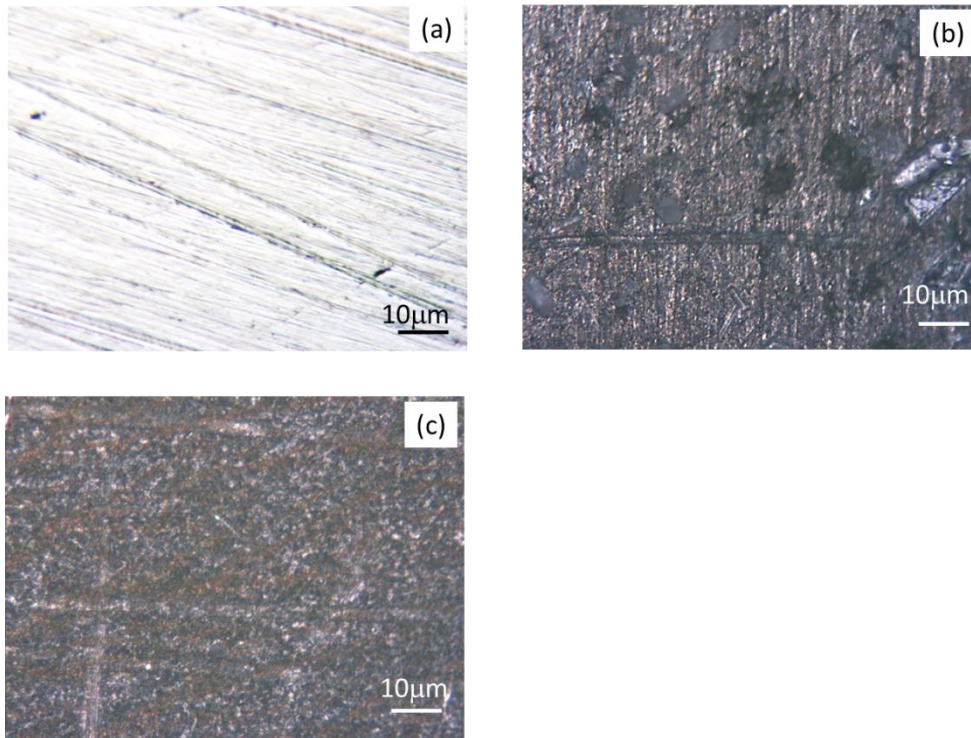
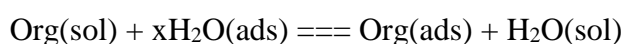


Figure 4.19: Optical microscopic images of (a) Polished MS, (b) dipped in Acid only, and (c) dipped in inhibitor solution

4.9 Mechanism of Corrosion

The mechanism behind the corrosion inhibition can be explained based on the above results and discussion. Generally, an inhibitor works on an adsorption mechanism. But the adsorption process in the corrosion inhibition mechanism is not simple. The energy of activation in acid-only solution is 63.74 kJ/mol which increases drastically in presence of alkaloids and reached (108.72 kJ/mol in 200ppm, 105.96 kJ/mol in 400 ppm, and 103.48 kJ/mol in 1000 ppm inhibitor solution), reveals that the inhibitors altered the reaction route. But, the similar trend in the polarization slope reflected the reaction pathway has not been changed. So, it concluded that the inhibitor molecules adsorbed on the MS surface form a protective layer that increases activation energy and reduces the corrosion rate by blocking both anodic and cathodic

reactions simultaneously. The free energy of the adsorption was found to be intermediate between physical and chemical adsorption energy revealing the physical adsorption followed by chemical adsorption between MS and alkaloids. Corrosion inhibition by alkaloid molecules is due to the adsorption of inhibitor molecules on the MS surface by the replacement of water molecules, called the quasi-substitution process.



Where, Org(sol) and Org(ads) represent the solvated and adsorbed organic (alkaloid) molecules, respectively. Similarly, H₂O (ads) represents the adsorbed water molecules on the MS surface, and x represents the size ratio i.e. many water molecules that are replaced by one organic molecule (Karki et al., 2021).

OCP gives the information where there is a positively or negatively charged MS surface. The OCP of MS is recorded at around 0.49 V, which is more positive than PZC value. It implies that the surface of the metal is positively charged in the inhibitor solution (Dhakal et al., 2022; Karki et al., 2021). In a positively charged surface, there could be an interaction of SO₄²⁻ ions. And the surface becomes negatively charged. Alkaloids contain mainly nitrogen as heteroelement in the ring, which when dissolved in an acidic solution gets protonated. The protonated alkaloid molecules interact with sulfate ions by the electrostatic force of attraction. The protonated alkaloids return to their neutral form after releasing H₂ molecules (Dhakal et al., 2022; Karki et al., 2020).

Then, the electron pair of HOMO with a high electron density of alkaloids especially the lone pair of nitrogen is shared with the vacant d-orbital of iron forming a coordinate covalent bond, which brings the accumulation of extra negative charge on the metal surface. To relieve the charge, electrons are returned to LUMO with high orbital density especially to antibonding π* orbital of the organic molecule to form a feedback bond. This retrodonation strengthens the bond and inhibitors are adsorbed chemically (Chapagain et al., 2022; Dhakal et al., 2022; Karki et al., 2020).

CHAPTER 5

5. CONCLUSION

In this research work, a green inhibitor for mild steel in 1M H₂SO₄ was successfully prepared from the extract of *Solanum xanthocarpum*. The corrosion of mild steel was studied in the presence and absence of inhibitors by using the weight loss method and electrochemical (OCP measurement & potentiodynamic polarization) method at different concentrations (200 ppm, 400 ppm, 600 ppm and 800 ppm) of inhibitors at different periods as well at varied temperature. In the process, alkaloids are successfully extracted by methanol extract using the liquid-liquid extraction method and characterized by phytochemical and spectroscopic methods. From the data and results obtained, the following statements can be inferred,

1. Alkaloids were extracted from methanol extract using the solvent extraction method.
2. *Solanum xanthocarpum* acts as a good and efficient inhibitor for the corrosion of mild steel in 1M H₂SO₄ by forming the protective layer on the surface of the mild steel coupon. The OCP measurement showed that the inhibitor is a mixed type.
3. Potentiodynamic polarization measurements showed that corrosion current density significantly decreases with an increase in inhibitor concentration.
4. The inhibition efficiency increases with an increase in inhibitor concentration in all the cases. The polarization experiment revealed that maximum inhibition efficiency is found to be 94.44 % in 1000 ppm for as-dipped conditions and 46.75 % in 1000 ppm for the immersed condition of 1 hour.
5. Weight loss measurement results showed that the inhibition efficiency of the plant extract, first increased with time, and after a certain time inhibition decreased to time. The maximum efficiency was found to be 97.27 % at 800 ppm concentration in immersion time 1 hour at 38 °C.
6. Both weight loss and polarization experiments showed that inhibition efficiency increases with an increase in concentration.
7. FTIR results confirmed the presence of N-H, O-H, C-H stretching, and C-H bending group, providing the inhibitive property.

Hence, it was concluded that the *Solanum xanthocarpum* can be used as an excellent, eco-friendly, green corrosion inhibitor for mild steel in 1M H₂SO₄ solution.

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