CORROSION INHIBITION BY ALKALOIDS EXTRACT OF *CISSAMPELOS PAREIRA*(Batule Lahara) STEM FOR MILD STEEL IN 1 M HCl SOLUTION

A DISSERTATION WORK SUBMITTED FOR THE PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE MASTER OF SCIENCE DEGREE IN CHEMISTRY

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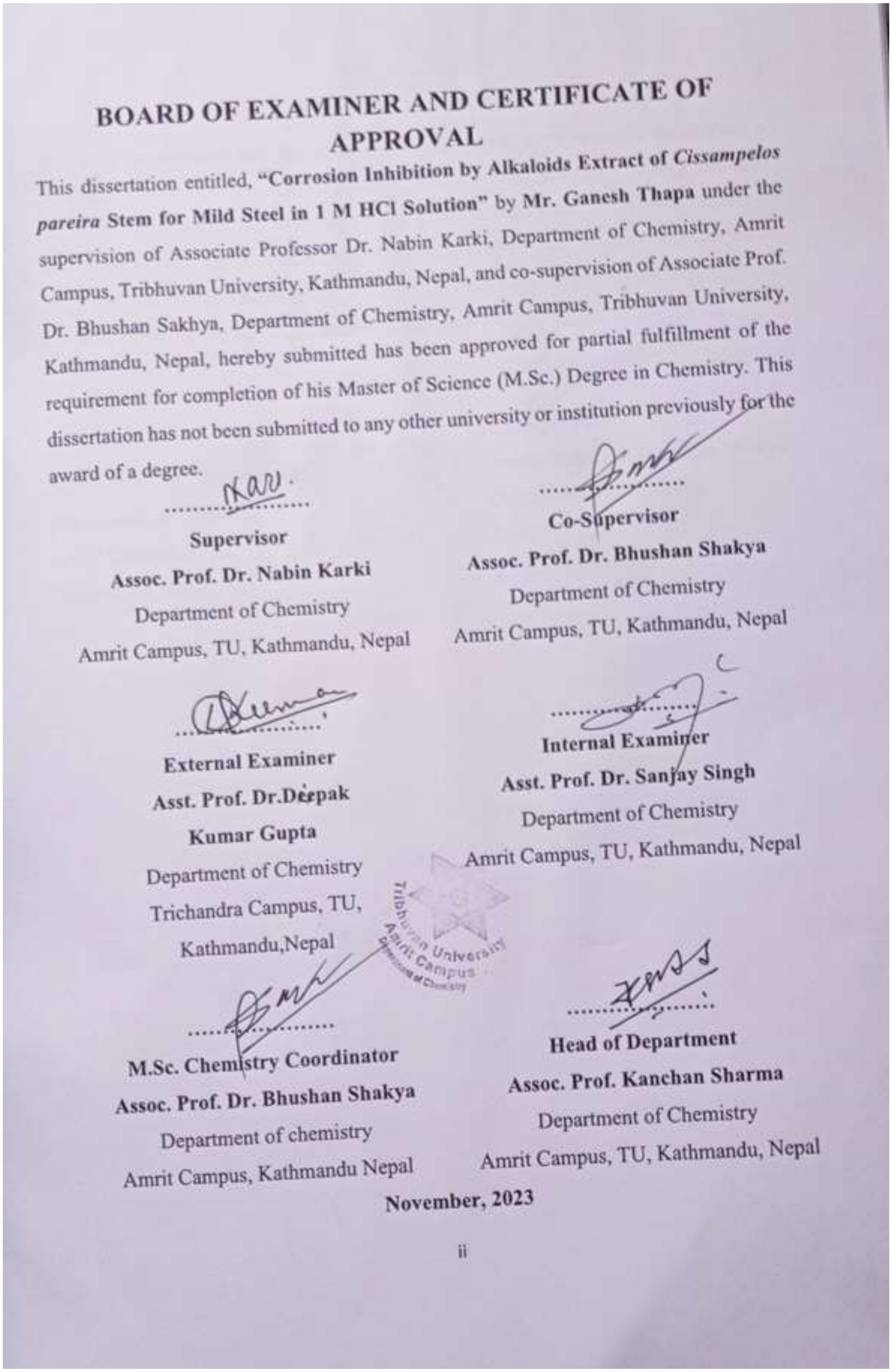

BOARD OF EXAMINER AND CERTIFICATE OF APPROVAL

This dissertation entitled, **"Corrosion Inhibition by Alkaloids Extract of *Cissampelos pareira* Stem for Mild Steel in 1 M HCl Solution"** by **Mr. Ganesh Thapa** under the supervision of Associate Professor Dr. Nabin Karki, Department of Chemistry, Amrit Campus, Tribhuvan University, Kathmandu, Nepal, and co-supervision of Associate Prof. Dr. Bhushan Sakhya, Department of Chemistry, Amrit Campus, Tribhuvan University, Kathmandu, Nepal, hereby submitted has been approved for partial fulfillment of the requirement for completion of his 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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LETTER OF RECOMMENDATION

This is to recommend that the dissertation work entitled, **"Corrosion Inhibition by Alkaloids Extract of *Cissampelos pareira* Stem for Mild Steel in 1 M HCl Solution"** has been carried out by **Mr. Ganesh Thapa** as partial fulfillment of the requirements for Master of Science Degree in Chemistry. This is his original work and has been carried out under my guidance and supervision. To the best of my knowledge, this research work has not been submitted for any other degree at this institute.

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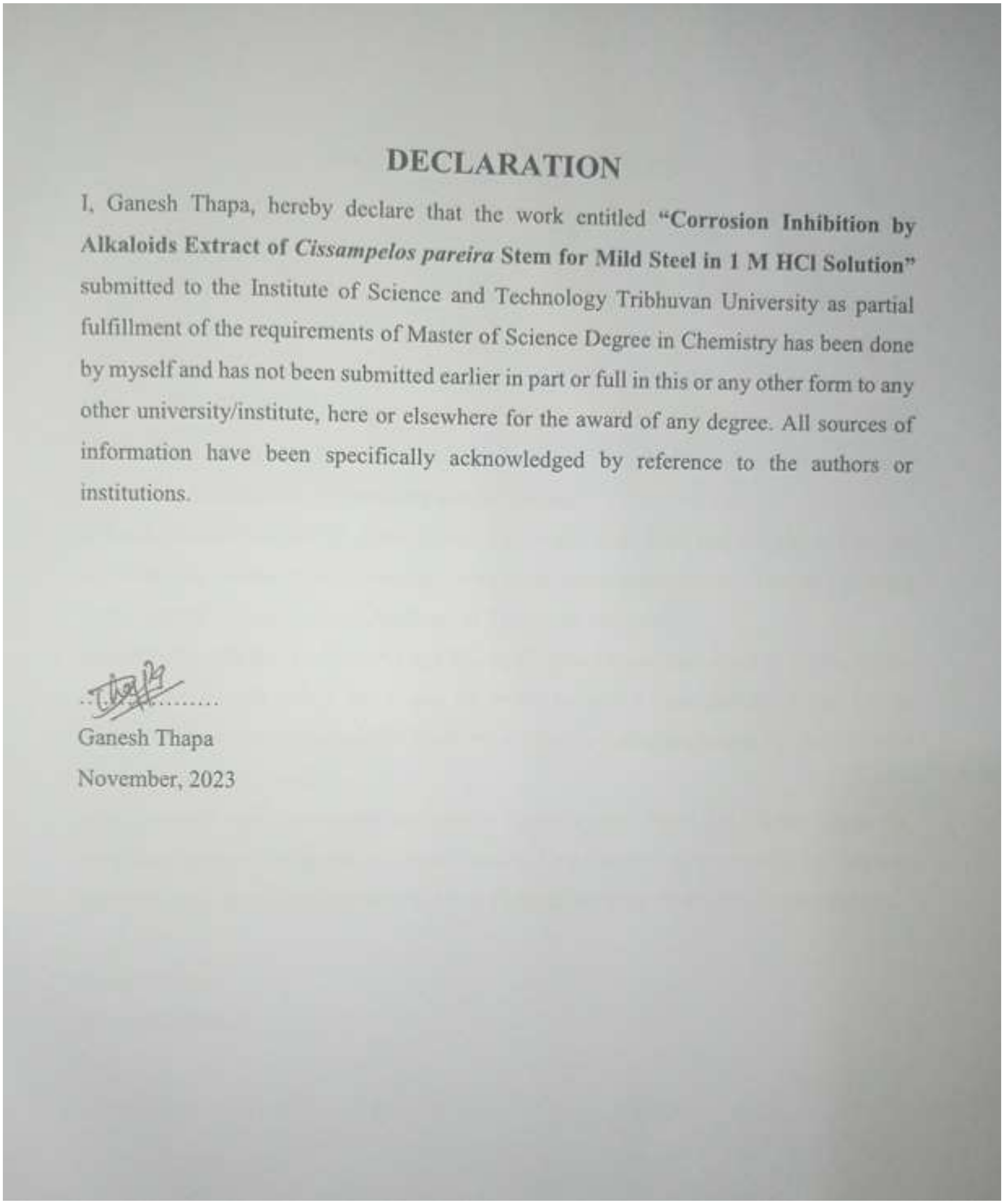

DECLARATION

I, Ganesh Thapa, hereby declare that the work entitled **"Corrosion Inhibition by Alkaloids Extract of *Cissampelos pareira* Stem for Mild Steel in 1 M HCl Solution"** submitted to the Institute of Science and Technology Tribhuvan University as partial fulfillment of 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

The use of an inhibitor is one of the effective alternatives for protective metals as opposed to corrosion. The use of inorganic inhibitors like chromates, borates, etc. are found to be toxic and their uses are substituted by the use of organic inhibitors like quinoline derivatives, thiourea derivatives, etc. although they were found to be highly expensive and hazardous, so the introduction of green organic inhibitor was essential because of cheap, environmentally friendly, and non-toxic. An organic compound with nitrogen contained as a hetero atom in a ring is considered to be an alkaloid, which can be a good alternative for corrosion inhibition by forming a protective layer to cease the contact of metal surface with acid (1 M HCl).

In this research, alkaloids of *Cissampelos pareira* (CP) were extracted by solvent extraction technique. The chemical test method and FT-IR analysis technique were used to confirm the presence of alkaloids. Corrosion inhibition efficiency (IE) was studied by the weight loss method, and electrochemical methods such as potentiodynamic polarization and electrochemical impedance Spectroscopy (EIS). Corrosion inhibition Efficiency increases with an increase in the concentration of alkaloids. It was 96.97% in 20 ppm and 98.88% in 100 ppm. Inhibition efficiency increases with the increase in time of immersion up to 18 hours. IE decreases with an increase in temperature. The adsorption of inhibitor on metal follows Langmuir adsorption and shows adsorption in mixed with dominating chemical adsorption as value range between -39.74 to 41.4 kJ/mol.

Thus, alkaloids extracted from *Cissampelos pareira* can be used as an effective inhibitor.

***Keywords**: Green inhibitor, weight loss method, Potentiodynamic polarization, Electrochemical impedance spectroscopy (EIS), Adsorption isotherm*

शोधसार

धातुलाई क्षयीकरणबाट सुरक्षित गर्नको लागि अवरोधकको प्रयोगलाई उत्तम विकल्पको रुपमा लिइन्छ । क्रोमेट, बोरेट आदि अकार्वनिक अवरोधकहरु विषालु देखिएकोले यसको प्रयोगलाई क्विनोलीन, थायोयूरिया यौगिक जस्ता कार्वनिक यौगिकहरुले विस्थापन गर्न थालिएको छ, तर यी यौगिकहरु पनि महंगा र हानिकारक नै छन् । त्यसैले सस्तो, वातावरणमैत्री, गैर विषाक्त, हरित अवरोधकको प्रयोग महत्वपूर्ण हुन्छ । नाइट्रोजन परमाणु रिङमा भएको कार्वनिक यौगिकहरुलाई अल्कल्वाइड भनिन्छ जसले धातुको सतहमा सुरक्षा तह बनाएर अम्ल र धातुको सम्पर्कमा बाधा उत्पन्न गर्न सक्ने भएकोले यो हरित अवरोधकको रुपमा प्रयोग गर्न सकिने यौगिकको राम्रो विकल्प हुन सक्छ ।

यो प्रयोगमा (अनुसन्धान कार्यमा) बाटुले लहरा (*Cissampelos pereira*) को विरुवाबाट अल्कल्वाइडको एक्स्ट्रयाक्ट घोलक एक्स्ट्रयाक्सन तरिकाबाट निकालियो । रसायनिक परीक्षण विधि तथा FT-IR विश्लेषण प्रविधिबाट एक्स्ट्रयाक्टमा अल्कल्वाइड भएको पुष्टि गरियो । तौल घटाउने विधि तथा पोटेन्सोडाइनामिक पोलाराइजेशन, इलेक्ट्रोकेमिकल इम्पिडेन्स स्पेक्ट्रोस्कोपी जस्ता इलेक्ट्रोकेमिकल प्रकृयाको प्रयोग गरि अल्कल्वाइडको क्षयीकरण अवरोधक कार्य क्षमताको अध्ययन गरियो । एसिडमा अल्कल्वाइडको गाढापनको बृद्धिसँगै यसको क्षयीकरण अवरोधक कार्य क्षमतामा बृद्धि भएको पाइयो । अल्कल्वाइडको अवरोधक कार्य क्षमता एकदमै कम गाढापनमा पनि उल्लेख्य पाइयो र यो २० ppm मा नै ९६.९७% र १०० ppm मा ९८.८८% पाइयो । धातुलाई डुवाइएको समय अनुसार १८ घण्टासम्म अवरोधक कार्य क्षमतामा बृद्धि पाइयो भने तापक्रमको बृद्धि सँग यो घटेको भेटियो । अवशोषण ल्याङ्गमुयर एडशोर्पसन आइसोथर्म अनुसार भएको पाइयो र अवशोषण स्वतन्त्र शक्ति (फ्रि इनर्जी) को मानले –३९.७४ देखि ४१.४० kJ/mol भएको आधारमा यो अवशोषण रसायनिक अवशोषण प्रबल भएको मिश्रित अवशोषण भएको देखाउँछ ।

निष्कर्षमा बाटुले लहराबाट निकालिएको अल्कल्वाइड एक्स्ट्रयाक्ट प्रभावकारी अवरोधकको रुपमा प्रयोग गर्न सकिन्छ ।

शब्द कुञ्जिका: *हरित अवरोधक, तौल घटाउने विधि, पोटेन्सोडाइनामिक पोलाराइजेशन, इलेक्ट्रोकेमिकल इम्पिडेन्स स्पेक्ट्रोस्कोपी र एडशोर्पसन आइसोथम*

LIST OF ABBREVIATIONS

GDP	Gross Domestic Product
IUPAC	International Union of Pure and Applied Chemistry
PHMSA	Pipelines and Hazardous Materials Safety Administration
EGIG	European Gas Pipeline Occurrence Data Organization
OGP	oil and gas pipelines
CPEx	*Cissampelos pareira extract*
CAGR	Compound Annual Growth Rate
W.L	Weight Loss
Pol	Polarization
EIS	Electrochemical Impendence Spectra
mL	Milli-liter
g	Gram
h	Hours
IE	Inhibition Efficiency
MS	Mild Steel
OCP	Open Circuit Potential
θ	Fraction of Surface Coverage
C_{inh}	Corrosion Inhibitors
CR	Corrosion Rate
E_a	Activation Energy
ϕ_{corr}	Corrosion Potential
FT-IR	Fourier Transform Infrared Spectroscopy
I_{corr}	Corrosion current
mV	Millivolt
mV/s	Milivolt per second
ppm	Parts per million
C.E	Counter Electrode
SCE	Saturated Calomel Electrode

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CHAPTER 1

INTRODUCTION

1.1. Introduction of Corrosion

The word corrosion was derived at the beginning of the 19th century from the lower Latin word “Corrosion” which means “Act of gnawing” and the word “corrode” was acquired from Latin word “Corroder” that reflect “to gnaw away”. Peneck defines corrosion as wear and tear by mechanically transported materials(Gregory 2016). According to a general definition, metal corrosion is a normal and inevitable process that occurs when specific elements in the environment interact with desired metal qualities (Umoren, 2016). It is a persistent issue that is frequently challenging to fully resolve. Complete elimination of corrosion would not be practicable or attainable but prevention could be done by using various technique. When metallic materials (metals and alloys) deteriorate unintentionally due to electrochemical and chemical interactions with their corrosive surroundings, it compromises the qualities of the metals and alloys that need to be conserved. This definition of corrosion is widely accepted by scientists worldwide (Heusler *et al*., 1989), in contrast to the very broad definition of corrosion recommended by the IUPAC (Bhattarai, 2010), which includes both metallic and polymeric materials like plastics, rubbers, etc., as well as ceramics like concrete, bricks, etc. defined as “an irreversible interfacial reaction of a material (metal, ceramic, polymer) with its environment which results in consumption of the material or in dissolution into the material of a component of the environment”.

Corrosion is an electrochemical process where the transfer of electron take place from metal to less active site (cathodic site) whereas the metal ion itself get migrate at anodic site in a solution during this process so the byproduct H_2 get evolved at cathodic site and metal get oxidized in anodic site(Karki, *et al*., 2021). A half-cell reaction is one of the two electrodes in a galvanic call or a simple battery occur as (McCafferty, 2010)

Anodic reaction:

An electron gets loss in anodic reaction

Metal (M) $\longrightarrow$ $M^{n+} + ne^-$ (1.1)

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

Cathodic reaction

The cathodic reaction goes according to the environment, the reactions are;

i. $2H^+ (aq) + 2e^- \longrightarrow H_2 (g)$ (1.2)

ii. $2H_2O + 2e^- \longrightarrow H_2 + 2OH^-$ (1.3)

Hydrogen evolution from neutral water:

iii. $\frac{1}{2} O_2 + H_2O + 2e^- \longrightarrow 2OH^-$ (1.4)

Oxygen reduction in neutral or basic solution

iv. Metal (M^{n+})+ $ne^- \longrightarrow$ Metal (M) (1.5)

Metal get reduce

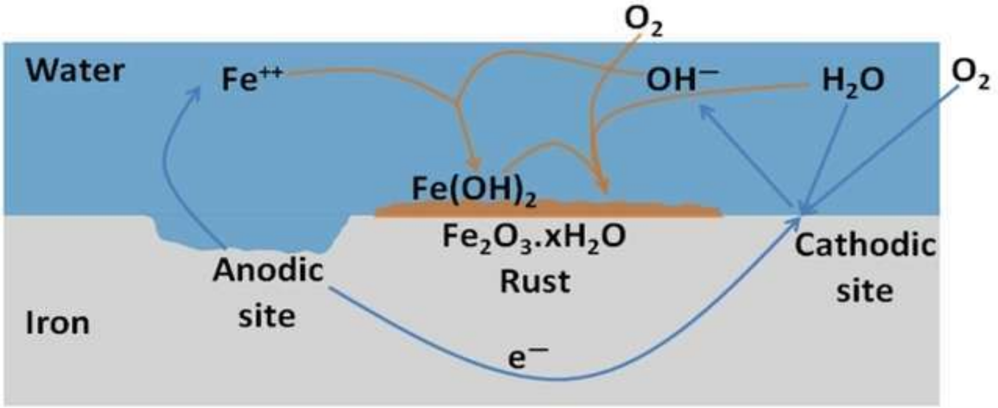


Figure 1: A schematic representation of the corrosion on a metal surface in an acidic solution showing both the anodic and cathodic site.

1.2. Economic Importance of Corrosion Study

One of the foremost vital chapters within the subject of chemical designing for scholastics, industrialists, and researchers is the think about of corrosion science (Karki *et al.*, 2022). Corrosion can be an irreversible factor that can cause catastrophic economic disasters, property damage, equipment loss, human deaths, and many other naturally occurring accidents and events, while at the same time shortening the lifespan of hardware (Regmi *et al.*, 2021) . Thus, the corrosion study is essential and be described in following points

a. Prevent Economic Losses:

Corrosion causes critical financial misfortunes to different monetary segments ,the whole yearly taken a toll of corrosion within the oil and gas generation industry is evaluated at $1,372 billion, counting $589 million in pipeline costs and surface establishment, $463 million per year in downsizing channeling costs and an extra $320 million in corrosion-related capital costs (Cao *et al*., 2017;Nik *et al*., 2012)**,** the overall cost of dropping from corrosion has been evaluate separately. It has been calculated that the US loses up to of 137.9 billion dollars per year. Corrosion-related hardship to worldwide GDP is anticipated to reach 3.4%, or more than $75 trillion per year. Inversion of disintegration within the Joined together States was US$9.2 billion in 1977, US$90.4 billion in 1999, US$1.1 trillion in 2016 in China was almost $310 billion USA in 2015 (Kadhim *et al*., 2021). As portion of India, a bordering nation, INR 1.67 trillion per year makes an disintegration chance, proportionate to 4.2% of GDP (Koch *et al*., 2016) also by a report of NACE predicted that the universal corrosion cost was US$2.5 trillion per year. Further, Corrosion cost in our bordering country, China and India, was 6.1%,4.7% and 3.4% from an agriculture, industries and services respectively was also reported (Karki, *et al*., 2021**;** NACE International, 2016**).** Realistic delivery of the right corrosion safety improvements will reduce these woes by 15-35%, or $400-900 billion (Gong *et al*., 2019;Haque *et al.*, 2017)**.**

As the full budge of Nepal in financial year 2079/80,was 17 Kharab, 93 Arabs and 83crore rupee, 4.2% was Nepal corrosion cost (75 Arab 90 crore) which can be minimized upto 15-35% of corrosion fetched by utilizing appropriate mastery which is about almost 11 Arab 3 crore to 25 Arab 74.42 crore rupees can be spared (GON, 2023; Dhakal *et al*., 2022)

Table 1: Cost of corrosion on various sectors on the basis of neighboring country data.

S.N.	Ministry of (Sectors)	Budget Percentage	GDP Cost in NRs Arabs	Corrosion Cost percentage	Corrosion Cost NRs in Arab
1.	Agricultural	23.9%	428 .73	6.1%	26.15
2.	Industries,	14.3%	256.52	4.7%	12.06
3.	Services	61.8%	1108 .59	3.4%	37.69
	Total	100%	1793.84		75.90

(*Economy Survey 2079/2080, 2022*)

b. Prevent Human losses:

Concurring to the Perilous Materials and Pipeline Security Organization (PHMSA) and the European Gas Pipeline Occurrence Data Organization (EGPODO), corrosion contributes 17% and 25% individually to client and are root cause of oil and gas pipelines failures (OGP) within the period of 2009-2018 and the period of 2007-2016 (Biezma *et al*., 2020).

***Table 2**: List of accident occur due to corrosion*

Date	Location	Human loss due to corrosion
November 1984	San Jaranico, Mexico	650
August 2000	Carlsbad, New Mexico	12
September 2010	San Bruno, California	8 (58 injured)
November 2013	Qingdao, China	62
July 2014	Kaohsiung, Tiwan	32

c. Conservation of precious materials and equipment safety: It is essential to conserved metal and various industrial tools and equipment by using proper technique which has been practices from our ancestor although they were unknown about scientific explanation (Karki *et al*., 2021).

1.3. Mild Steel

Mild steel contains a little sum of carbon, too known as low carbon steel, with composition C 0.21-0.27%, Mn 0.13-0.45%, P 0.79-1.30%, S 0.035 % and rest of is iron (Kamal & Sethuraman, 2012). Mild steel is commonly utilized in different businesses counting development, transportation, pipelines, mining, chemical preparing, atomic control, fossil fuel generation, oil refineries and metals plants (Shukla *et al*., 2011) due to its many physical properties (malleable quality, high hardness) and low cost (Ahamad *et al*., 2010).

Steel generation has expanded strongly in later a long time, coming to more than 1662 million tons in 2014, an increment of 1.2% compared to 2013. By 2050, the request for steel is anticipated to extend by 1.5 times, numerous times the current level to meet the wants of the developing populace (Mousa *et al*., 2016), but it is thought that numerous reasons counting expanded transportation, crude fabric and labor costs have driven the lessening of South Africa's steel generation capacity from 9.7 million tons in 2006 to 6.6 million tons in 2014. In 2019, 1870 million tons of steel was produced worldwide, of which only 1545 million tons was used (Pelser *et al*., 2022).

A quarter of the world's yearly steel generation, or around 150 million tons, is accepted to be annihilated each year by corrosion (Parajuli *et al*., 2022). MS corrosion is one of the genuine issues that industry must confront (face) an unforgiving environment of corrosive and pickling prepare. HCl, H_2SO_4 and acids are utilized for descaling, corrosive pickling, chemical cleaning of steel structures also boilers cleaning (Bhardwaj e*t al*., 2021). Corrosion leads to critical remediation costs, substitution of different parts and hardware, misfortune of life and natural issues (Singh *et al*., 2016).

1.4. Corrosion control and corrosion protection using inhibitor

The surface was the most sensitive part in an engineering but corrosive environment degrade the properties of such metal surfaces, so the scientist had to face many problem regarding corrosion and wide research had been carried out from past few decade (Akande *et al*., 2019). The corrosion process creates quickly after the defensive obstruction is broken and is went with by a number of responses that alter the creation and properties of the metal surface and the nearby environment by the arrangement of oxides or dispersion of metal cations within the network either by a alter in pH or by an electrochemical potential (Sivakumar & Srikanth, 2020)**.** As the corrosion control is essential and can be prevented by using various technique like protective coating and lining, biocides, anodic protection ,cathodic protection, corrosion inhibitors and selection of materials (Liu *et al*., 2020).

Protective Coating: The anti-oxidant property of a carbon steel can be uplifted by this coating and two system coating or sometime 3 system coating have practice especially in an paint. Also some composite coating has been practice to protect the corrosive surface in an engineering fields (Bierwagen 1996;Akande *et al*., 2019).

Anodic Protection : This is an another corrosion minimizing method, where, metal to be protect was forcefully made more anodic than in normal state by passing an external current which finally formed an uniform metal oxide over desire metal (Munro & Shim, 2001).

Cathodic Protection (CP): Cathodic protection is one of the corrosions controlling technique, where metals to be protect was forcefully made as more cathodic than in at normal state by connecting more reactive metals (Al^{+3}, Mg^{+2}, Zn^{+2}) as anode. The most commonly used electrolyte (expose environment) were soil, water, Caustic soda, $CaCl_2$, etc. and more practically used as cathodic protection are in an area of aluminum pipelines, steel uses concretes, underground tanks, dams etc. (Bushma, 1990; Durham, 2005).

Although, those protective methods can minimize the corrosion rate of metal exposed to certain environment, but had high chance to losses others metal while preventing the one, so these methods can be overcome by the uses of inhibitors.

Corrosion Inhibitor**:** Corrosion inhibitors are characterized as materials that are joined in little concentrations with the environment, successfully decreasing the corrosion rate of metals in contact with it (Raja *et al*., 2016). The utilization of inhibitors is one of them mostly practiced options to secure metals and amalgams from corrosion, particularly in acidic arrangements from few decades ago.

Inorganic inhibitors incorporate phosphates, silicates, borates, tungsten, chromates, dichromates, molybdates (are also anodic inhibitor) (Marciales *et al*., 2018;Tamalmani, 2020), arsenates, in acidic media acts as an ideal inhibitor but such types of inhibitors were found to highly carcinogenic, water insolubility as well highly hazardous towards aquatic and terrestrial living being (Karki *et al*., 2020). Thus concept of an organic inhibitor place forwards (Marzorati *et al*., 2019) .Organic inhibitors incorporate nitrogen inhibitors such as acetylenic alcohols, fragrant compounds containing aldehydes, iminium salts, nitriles salts, triazoles salts (Chevalier *et al*., 2014), thiocyanates salts, pyridines salts and theirs respective subsidiaries , subordinates of quinoline, thiourea subsidiaries, quaternary salts, etc. species. Thiosemicarbazide salts and derivatives, quinoline subordinates, thiourea subsidiaries, benzotriazole (Tamalmani 2020), nitriles salts and derivatives, iminium-based salts and nitrogen complexes have been effectively treated by shaping lean films on metal surfaces and decreasing misfortune metals and H_2 generation (Williams, 1992;Marciales *et al*., 2018;Chevalier *et al*., 2014;Tamalmani, 2020) .Those inhibitor are more compatible than inorganic inhibitors but they are highly expensive to synthesis in laboratory, time consuming and some of them are non eco-friendly. So, keeping the principle of green chemistry as well as sustainable development principle these both inhibitors need to replace by further inhibitor i.e. Green organic inhibitors was introduced.

Since the choice of inhibitor is exceptionally vital to stifle the rate of corrosion, the inhibitor has less of a characteristic as recommended by Paloule. A few of them are (i) Capacity to secure metal surfaces, (ii) Profoundly dynamic for utilize in little amounts (ppm), (iii) Low fetched compound(s), (iv) Dormant properties so as not to influence prepare, (v) Ease of dealing with and capacity, (vi) Superior with low harmfulness, (vii) No contamination, (viii) It should act as an emulsifier and frothing specialist. These characteristics created within the inhibitor are influenced by (i) chain length, (ii) atomic measure, (iii) official, aromatics/conjugates, (iv) bond quality substrate, (v) cross-linking capacity and (vi) medium solubility (Kadhim *et al*., 2021)**.**

1.5. Green corrosion inhibitor

The synthetic organic inhibitor compounds which are especially Nitrogen and Sulphur containing compound, use in oil, gases and petroleum product are found to be highly toxic, water insoluble along with highly expensive, difficult to degradable and need to be immediate replaceable (Marzorati *et al*., 2019; Ngouné *et a*l., 2019) .Thus, development of highly acceptable ecofriendly corrosion inhibitor is need to be developed because of which green corrosion inhibitor is kept in priority rather than synthetic inhibitors (Regmi *et al*., 2021) also, green inhibitor components have almost show similar properties with organic inhibitors because most of them are alkaloids rich species and along with containing others phytochemical like flavonoids, terpenoids, saponins etc. The natural product must be well studied. Variety of faunas found in our surrounding are considered being main source of green corrosion inhibitor. Thus the extract from various part of fauna reported to be acts better corrosion inhibitor especially in acidic medium for mild steel, iron, copper and aluminum (Gupta *et al*., 2020)**.** The efficiency of these organic corrosion inhibitors is related to the presence of polar functions containing in the trend of O < N < S < P atoms which are centers for the developing of the adsorption process (Jmiai *et al*., 2018;Parajuli *et al*., 2022). These heteroatoms containing organic compounds along with π bond able to form a new bond with vacant d orbital of metal which prevent the corrosion of metal by forming a passivating layer over metal by plant extract. Materials (Thapa Magar *et al*., 2023; Karki *et al*., 2021)

1.6. Plant selected for study: *Cissampelos pareira*

The imperative agroforestry plant *Cissampelos pareira* of the Menispermaceae family is commonly known as Ambastha or Laghu Patha in conventional Indian pharmaceutical, and in South America as 'parreira' whereas batule pate / badal pate lahara in Nepal (Parmar *et al.*, 2011*)* The systematic classification of the plant is

Kingdom – Plantae

Division – *Magnoliophyta*

Class – Magnoliopsida

Order – Ranunculales

Family – Menispermaceae

Genus – *Cissampelos*

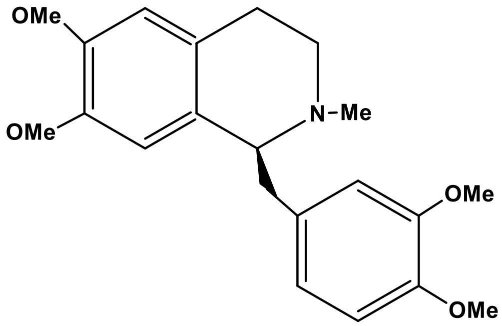
Species*: pareira*

Figure1*: Cissampelos pareira* (a) leaves,

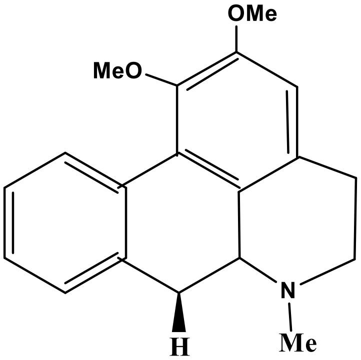
It is perennial, erect and climbing herbs which is usually 2-5 m above the ground and distributed in the tropical and subtropical regions found up to 2100 m high. It has plentiful record in its orthodox wield in Ayurveda, Traditional Chinese's Medicines and Western Herbalism (Singh *et al.*, 2010) Traditionally it was ply breaks, untimely conveyances, measles, headaches and body harms, fevers, hacks and colds) are served with this mending vegetation. The underground portion is essentially utilized for distinctive therapeutic purposes. The root stick of this herb is utilized to treat colic, aggravation, corrosive reflux, loose bowels, wounds, fistulas, shivering, hemorrhoids, snake bites, breast torment, skin rashes, and lactation clutters.

In the context of Nepal; its root powder used for Headache, ulcer (Kumari et al., 2021), root juices against stomachache , juice of aerial part for fever treatment and induced abortion (Dangol & Gurung, 1991), whole plant juice for Hemorrhage treatment (Balami, 2004); root and leaves for Cough, sores and disorder (Gautam, 2013)

To date, approximately 54 compounds have been reported from *C. pareira* with a major focus on isoquinoline alkaloids(Bisbenzylisoquinoline, Benzylisoquinoline, Tropoisoquinoline) (Bafna & Mishra, 2021), Protoberberine, Azafluoranthene (Kumari *et al.*, 2021). The aerial parts contain the alkaloids like Laudanosine, Corytuberine, Nuciferine, Bulbocarpine, nor-N-magnoflorine and non-alkaloids includes Amentoflavone, Kaempferol-3-O-β-D-glycopyranoside, Cissampeloflavone (Ramírez *et al.*, 2003). The modern pharmacological studies indicated that this plant exhibited diverse biological activities such as anti-leukemic, antinociceptive and antiarthritic, anthelmintic (Shukla e*t al*., 2012) , curariform (basu, 1970), cardio-protective anti-dengue (Sood *et al*., 2015) antioxidant and immunomodulator (Kumari *et al*., 2021), and chemo-modulatory activities (Gupta *et al*., 2007).



Laudanosine(Benzylisoquinoline)



Nuciferine(Aporphine)

1.7. Objectives of study

1.7.1. General Objective

The general objective is to study the corrosion inhibition efficiency of alkaloids extract from *Cissampelos pareira* stem as green inhibitors for mild steel in acidic medium.

1.7.2. Specific Objectives

The specific objectives of the study are:

- To extract alkaloids from *Cissampelos pareira* and confirm presence of different functional groups by FT-IR.
- To study corrosion inhibition efficiency of extract varying concentration, temperature and time of immersion by weight loss method.
- To study corrosion inhibition efficiency of alkaloids extract using electrochemical methods such as potentio-dynamic polarization, EIS.
- To study the adsorption isotherm, thermodynamic and kinetic parameters of inhibition process.

CHAPTER 2

LITERATURE REVIEW

The problems posed by conventional and synthetic inhibitors which were highly toxics, expensive, non-biodegradable, carcinogenic should be controlled and must minimize in utilization so the explorers and researcher were highly focused to develop biodegradable, non-toxic, inexpensive inhibitors, which can be solved by using natural product extract as nature itself is a major source for solving many problems, are also environmentally friendly (Faustin *et al*., 2015). Due to their eco-friendliness, the market for plants that have been harvested and organic green corrosion inhibitors is anticipated to grow by 7.0% by 2026 (Hossain *et al*., 2021).

In world the global market for corrosion inhibitors was $7.2 billion in 2019 and is projected to increase at a 3.6% Compound Annual Growth Rate (CAGR) to $9.6 billion by 2026, according to a report. As different plants sources for corrosion control have been discovered to date, the use of an appropriate extraction procedure and extraction solvent is important for extraction from the natural product (Tamalmani & Husin, 2020).

The first natural product extracts used in corrosion studies date back to about 1930 from *Chelidonium majus* (Celidoine) and other plant extract had been studied in H_2SO_4 (Sanyal, 1981). Various parts of plants or whole plant extract can be used as corrosion inhibitor for mild steel at acidic condition (HCl, H_2SO_4).

The plant extract obtained from *Poinciana pulcherrina* show similar IE of 94% when studied by polarization as well as by EIS method, but when studied on *Cassia occidentais* extract shows highest IE 93% by polarization method and least when study by weight loss method 87%, also in *Datura stramonium* has IE=91% when study pol, 90% EIS method and , 86% by weight loss method (Zucchi & Omar, 1985) in 1 N HCl at 1 gL^{-1} so these plants shows good corrosion inhibitor properties. Similarly extract from *Rosmarinus officinalis* oil studied by EIS show the inhibition efficiency 73% at 298 K in 2 M H_3PO_4 at 10 gL^{-1} (Bendahou *et al*., 2006).The leaf extract of *Carica papaya* show the inhibition efficiency 93.88% at 2 g/L in 1 M H_2SO_4 (Okafor & Ebenso, 2007). Also *Piper guinensis* plant extract show IE of 93.76% when studied by weight loss method at 0.5 gL^{-1} in 2.5 M

H_2SO_4 (Ebenso *et al.*, 2008), when the peel of *Musa acuminate* was studies I.E. was found to be 71.05% at 4000 ppm in 2.5 M H_2SO_4 (Eddy & Odiongenyi, 2008) and also shows physical adsorption following Langmuir adsorption. While studying medicinal plant *Alove vera* shows the IE was 75% at 0.4 gL^{-1} in 2.5 M H_2SO_4 (Eddy & Odoemelam, 2009) also *Ananas comosus* show the IE of 72% when studies by weight loss method at 0.5 g/L in 1 M HCl at 333K and follows chemical adsorption (Ekanem *et al.*, 2010) .When *Ocimum gratissimum* plant extract was studied by gasometrical method show the IE= 97.37% at 0.5 g/L in 2.5 M H_2SO_4 shows physical adsorption (Eddy *et al*., 2010) but when *Ananas sativum* was studied by WL show IE 96.09% at 0.5 g/L in 0.1 M HCl at 333 K shows chemo-sorption (Ating *et al*., 2010).

When seed of *Areca catechu* extracts was studied, highest inhibition efficiency was found by EIS method i.e. 97.61% and almost same efficiency was found when studied by weight loss method 96.97% at 500 ppm in 1 M HCl also shows physical adsorption (Kumar *et al.*, 2011), likewise fruit extract of *Cordia dicotona* shows IE 75.86% in 0.1 N HCl and IE 92.81% in 0.1 N H_2SO_4 when in both studied by weight loss method and follows Langmuir adsorption isotherm (Khandelwal *et al.*, 2011), also when extract from *Schinopsis lorentzii* was studied by EIS found to be IE=38.60% at 2000 ppm in 1 M HCl show physical adsorption but follow Temkin adsorption isotherms (Gerengi, 2012) ,but extract from *Parthenium hysterophorus* reported that highest IE found by polarization method with IE 83.85% and least by weight loss method having IE=78.99% at 1100 ppm in 1 M HCl and follows Langmuir adsorption isotherm (Ji *et al*., 2012). When juices of *Apricot* plant was studied by weight loss method IE was found to be 75% at 40 g/L in 1 M H_3PO_4 shows physical adsorption (Yaro *et al*., 2013), also leaf extract of *Acalypha torta* show IE 89% by in 1000 ppm at 1M HCl (Krishnegowda *et al*., 2013), and extract from *Tagete erecta* shows IE 98.07% by EIS method, 96.10% by pol method at 1 g/L in 0.5 M H_2SO_4 (Mourya *et al*., 2014), When leaf extract of *Aquilaria crassna* in Methanol extract was studied shows high IE 80.28% by WL method and least when studied by EIS method having IE 76.36% in 1 M HCl at 300 ppm (Helen *et al*., 2014), Watermelon rind when studied with weight loss method IE was found to be 83.35% in 1 M HCl and 77.34% in 0.5 M H_2SO_4 at 2 g/L (Odewunmi *et al*., 2015). Similarly leaf extract of *Psidium guajava* leaf shows highest inhibition efficiency 87% by WL method , least IE 74% by pol method, 82% by EIS method at 1200 mg/L in 1 M H_3PO_4 (Victoria *et al*., 2015), *Rhus verniciflua* had IE=91% by WL method, 93.01% by pol, 89.12% by EIS methods at 500 ppm in 1 M H_2SO_4 (Prabakaran *et al*., 2016), When *Canna indica* flower was studied, highest IE was found to be 95.52% by WL, and least efficiency 77.79% by EIS method and by polarization it was reported to be 83.47% at 0.7 g/L in 1 M HCl (Verma *et al*., 2016) , Similarly when leaf extract of *Bauhinia tomentosa* shows IE 93.47% by WL method, 94.56% by EIS method in 1 M HCl at 700 ppm (Perumal *et al*., 2017) all these plants show physical adsorption. *Butea monosperma* show least IE 87.45% by WL method and maximum Efficiency 97.94% by polarization method at 500 ppm in 0.5 M H_2SO_4 (Saxena *et al*., 2017) also when study extract from papaya seed show I.E. 90% by EIS in 3 M H_2SO_4 and while studying watermelon wastes seed I.E was found to be 83.67% by EIS at 200 ppm and in its peel I.E was found to be 72.42% by EIS at 200 ppm, from cashew extract I.E was found to highest i.e. 93% by EIS method and 80% when studied by polarization method at 800 mg/L in 1 M HCl (Tamalmani & Husin, 2020) while studying lychee plant I.E was found to be 98% in 0.5 M HCl, *Aloe vera* shows I.E 98% in 1 M H_2SO_4 (Hossain *et al*., 2021) consider to be better green inhibitors. Also the leaves extract of *Neolomarckia cadamba* shows I.E 88% at 300 mg/L, *Euphorbia falcate* shows I.E 93% at 300 mg/L, and *Silybum marianum* shows I.E=95% at 800 mg/L, Neem shows I.E=81% at 250 mg/L (Bhardwaj *et al*., 2021) also acts as better green inhibitors.

Other plants extract like bamboo leaf by using ethanol solvent when studied by polarization and weight loss method in 1 M HCl shows IE 90.3%, 87.4% for 0.5 M H_2SO_4 and IE 79.2%,78.8% respectively (Li *et al*., 2012), from *Sida acuta* (I.E=85% by WL at 0.5 g/L in 1 M H_2SO_4)(Umoren *et al.*, 2016) , *Myristica fragrans (*I.E=87.81% EIS, 83.27% W.L in 0.5 M H_2SO_4 at 500 mg/L) (Haldhar *et al.*, 2018), *Zizyphus lotus*(I.E=92.56% W.L, 91.72% pol, 90.57% EIS in 1 M HCl) (Jmiai *et al.*, 2018), rice straw (I.E= 85% W.L, 53% pol, in 3.5%NaCl) (Othman *et al*., 2019), *Gongronema latifolium* (I.E=95.4% W.L) (Alexander *et al.*, 2019), *Mimuaps elangi* leaf(IE=98.50% W.L, 81.71% pol, 75.53% EIS at 20 ppm in 1 N HCl) (Sivakumar *et al.*, 2020), Lemon Balm (I.E= 94.04% EIS immersed for 12 h at 800 ppm in 1 M HCl) (Asadi *et al*., 2019), *Litchi chenesis*(I.E= 97.8% W.L, 95.7% pol, 97.8% EIS, at 3 g/L in 0.5 M H_2SO_4) (Singh *et al*., 2019) , *Euphorbia royleana*(IE=99.6% by W.L at 100% extract concentration in 1 M HCl) (Thapa *et al*., 2019) also reported as better green inhibitor in acidic medium.

The history of using plant extract as corrosion green inhibitor was old in the world but while seen in the context of Nepal this practice wasn't older. Some the Nepalese plant which were practice in methanol extract were *Artemesia vulgaris*(IE=67.81%) , *Lantana camara* (IE=97.33%pol) (Shrestha *et al.*, 2019), *Jatropha curcas(*IE= 92.05% W.L, 91.34% pol) (Gupta *et al*., 2020), *Equisetum hyemale* (IE= 92.74% pol, 86.28% EIS, 82% W.L) (Karki., *et al.*, 2021), *Soria robusta (*I.E= 71.4% W.L, 75% pol) (Bajgai *et al.*, 2022).

The research work on plant extract as a corrosion inhibitor has significant limitations due to a limited explore on phytochemical of plants. The limited explore on phytochemical may be affected by variables such as solvent-to-solids proportion, solvent type of distinctive extremity, extraction time and temperature can also influence the properties of the extricate through dissolvable extraction, as well as the composition of the extricate, chemical and physical properties of the sample. (Dai & Mumper, 2010) so the chosen of proper solvent is essential in both phytochemical investigation and corrosion study. As most of research on corrosion had been studied by using ethanol as solvent but due to cost effective methanol was also chosen in few cases. Plant extract using methanol was *J gendarussa* (IE=91.3% pol, 86% EIS) (Satapathy *et al*., 2009).

2.1. Alkaloid Extract

The organic compound that consist of at least one Nitrogen atom as a hetero atom in plants was consider to be an alkaloids, whereas attached oxygen atom may also present in ring as the functional group (Chapagain *et al.*, 2022). Literatures shows that most successful isolated compound that works as corrosion inhibitor is alkaloid. Because an alkaloid has almost similar properties with synthetic organic inhibitor. To overcome the drawbacks seen in organic inhibitor study of flora was essential therefore, plant are views as the naturally synthesized rich sources of chemical components (Lebrini *et al.*, 2010). The flora rich in

Phosphorus was difficult to gather although it has higher inhibition efficiency so the alkaloids like papaverine, strychnine, quinine and nicotine were mostly studied as corrosion inhibitors in acid medium (Lebrini, *et al*., 2011). The practice of using alkaloids extract from plants as corrosion green inhibitor was not too old in developed countries but while seen in the context of Nepal this practice was started from a few years ago.

S.N	**Plants/ Parts use**	**Conc.**	**Extraction Solvent**	**IE%**	**Methods**	**Literature**
1.	*Oxandra asbeckii* (leaf)	100 mg/L	DCM (Acid Base extraction)	88% (1M HCl) 92%(1M HCl)	Polarization EIS	(Lebrini, *et al*., 2011)
2.	*Palicourea guianensis*(Leaf)	100 mg/L	DCM (Acid Base extraction)	90% 89%	Polarization EIS	(Lebrini, *et al*., 2011a)
3	*Aspidosperma album* (Bark)	100 mg/L		90%(1M HCl) 88%(1M HCl)	EIS Polarization	(Lebrini, *et al*, 2011b)
4.	*Caulerpa racemose* (Algae)	100 ppm 25 ppm 250 ppm	Methanol	83%(1M HCl) 80%(1M HCl) 85%(1MHCl)	Weight loss Polarization EIS	(Kamal & Sethuraman, 2012)
5.	*Neolamarokia cadamba* (leaf/Bark)	5 mg/L	DCM (Acid Base extraction)	82% (bark,1MHCl) 84%(leaf) 91%(bark) 88%(leaf)	EIS EIS Polarization Polarization	(Raja *et al*., 2013)

6.	*Garcima kola* (Seed)	10 mg/L	Ethanol	99.4% (H_2SO_4) 98% (H_2SO_4)	H_2 evolution Polarization	(Ikeuba *et al.*, 2013)
7.	*Aniba rosaeodora* (Wood)	200 mg/L	DCM (Acid Base extraction)	91% (1 M HCl) 95.3% (1M HCl)	Polarization EIS	(Chevalier *et al.*, 2014)
8.	*Solanum melongena* (Leaf)	3 g/L	Ethanol	81.1% (Na_2CO_3)	Weight loss	(Ugi, 2014)
9.	*Geissospermum laeve*(Bark)	100 mg/L	DCM (Acid Base extraction)	90% (HCl) 90% (HCl)	Polarization EIS	(Faustin *et al.*, 2015)
10.	*Rauvolfia macrophylla* (Bark)	200 mg/L	Alk+ Isolated Comp	97% (1M HCl) 92% (1M HCl)	Polarization EIS	(Ngouné *et al.*, 2019)
11.	*Eucalyplus globulus*	1.09 g/L		87.65% (0.1M NaOH +0.5M NaCl)	Polarization	(Etteyeb & Nóvoa, 2016)
12.	*Berberis Aristata*	1000 ppm		96.0% (H_2SO_4) 98.18% (H_2SO_4)	Weight loss Polarization	(Karki *et al.*, 2020)
13.	*Rhynchostylis retusa* (Rhizome)	1000 ppm	Methanol	87.51% (H_2SO_4) 93.24% (H_2SO_4)	Weight loss Polarization	(Chapagain *et al.*, 2022)

14.	*Solanum tubersom* (Stem)	1000 ppm	Methanol	90.79% (H_2SO_4) 91.10% (H_2SO_4)	Weight loss Polarization	(Parajuli *et al*., 2022)
15.	*Artemisia vulgoris (*Stem)	1000 ppm	Methanol	92.58% (H_2SO_4) 92.18% (H_2SO_4)	Weight loss Polarization	(Parajuli *et al*., 2022)
16.	*Alnus nepalensis (*Bark)	1000 ppm	Methanol	<70% (H_2SO_4) 90% (H_2SO_4)	Weight loss Polarization	(Dhakal, 2022)
17.	*Coriaria nepalensis* (Stem)	1000 ppm	Methanol	96.41% (H_2SO_4) 97.03% (H_2SO_4)	Weight loss Polarization	(Oli *et al*., 2022)
18.	*Acacia catechu* (Bark)	1000 ppm	Methanol	93.96% (H_2SO_4) 98.54% (H_2SO_4)	Weight loss Polarization	(Karki *et al*., 2022)
19.	*Ageratina adenophora* (Stem)	1000 ppm	Methanol	96.95% (H_2SO_4) 92.53% 91.23%	Weight loss EIS Polarization	(Thapa Magar *et al*., 2023)

The other phytochemical extract like Terpenoid from *Salvia officinalis* L.(Khiya *et al*., 2019), flavonoid Gallocatechin and catechin present in *Musa paradisica* (banana) peel (Tiwari *et al*., 2018) , volatiles oils from Olive pomace(Bouknana *et al*., 2014)   Mustard seed (Umoren, 2016) , Tannin from *Xanthium strumarium* Leaves (Bhardwaj *et al.*, 2021) also acts as corrosion inhibitor.

CHAPTER 3

MATERIALS AND METHODS

3.1. Study Area

Firstly, the plant was recognized by the Herbarium specialist as Cissampelos pareira. The stem of the plant was collected from Sainamaina-4 Municipility, Rupandehi District (Latitude: 27°41'1.644''N, Longitude: 83°20'7.74''E), Lumbini Province, Nepal. The collected sample was washed with tap water to remove dust particles and shade dried nearly for 1 month and the dried sample was grinded into fine powder by using grinder (AMEET)in Amrit Campus

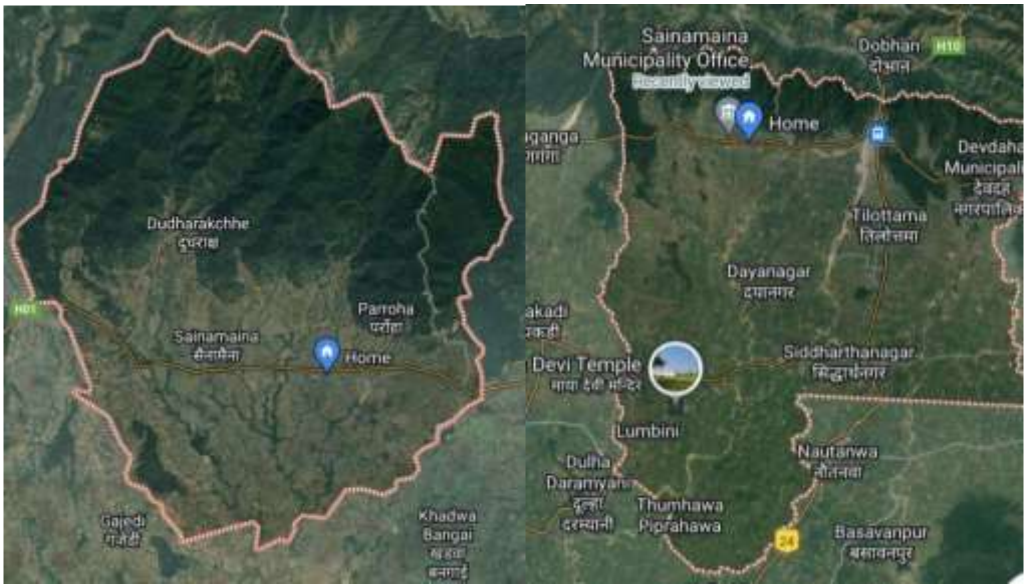


Figure 2:(a) Sainaminana Municipality;(b) Rupandehi District

3.2. Preparation of Alkaloids extract

First, 200 g of grind powder was soaked in 750 mL of hexane for 24 h to remove fats and non-polar phytochemicals, and then filtered. The residue may contain alkaloids in the form of salts. So, it is treated with 25 mL conc. ammonia to recover alkaloids. It was then soaked for 1 week using 750 mL of methanol and shaking frequently. After filtration, the filtrate was concentrated with a rotary evaporator (IKA RV 10) and treated with 5% 2 N HCl to convert the alkaloids to their salts and left to stand for one day at pH 2.5 and then treated using DCM till the colorless solution was observed. Salts of acid-soluble alkaloids will be present in the acid layer (water layer) while other pigments and phytochemicals have been separated in the DCM layer. The water layer was washed with DCM 3 times. Then, 25% NH_4OH was added to the water layer to maintain the pH at 10.5 as measured with a HAANA pH meter. It is then extracted with DCM in a separating funnel. Then, the top layer of DCM was collected and evaporated over a 25°C water bath. Finally obtained extract was dried and weighted (2 g), collected and utilized for further steps.

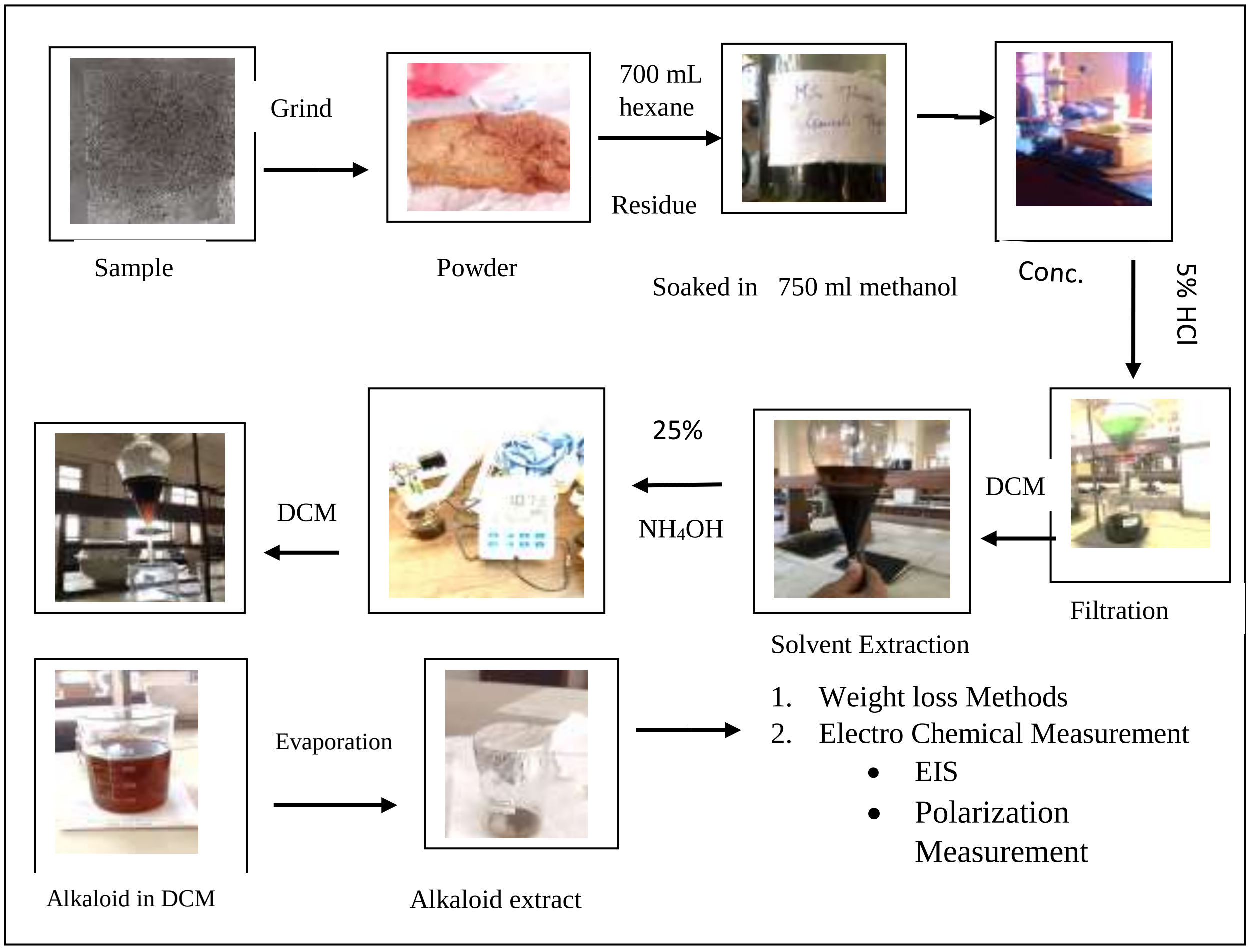


Figure 3: Process to obtain alkaloid Extract

3.3. Preparation of solution

3.3.1. Preparation of Acidic solution

A stock solution of one molar hydrochloric acid was prepared by diluting of 182 mL (11M) 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 Hydrochloric acid solution and labeled as 1000 ppm. Other required concentrations of the inhibitor solutions (20, 40, 60, 80 and 100 ppm) were prepared by diluting this stock solution with 1 M HCl.

3.4. Test for alkaloids

Both chemical and Spectroscopy method had been studied for the test of alkaloids. In chemical method mainly Mayer`s test, Dragendorff`s test and Wagner`s test was studied whereas in Spectroscopy method FT-IR Spectroscopy (PerkinElmer spectrum) was used.

3.4.1Mayer's test

At first freshly mayer reagent was prepared by dissolving 5 g potassium iodide and 1.36 g mercuric chloride in 100 mL distilled water. Then about 2 mL alkaloid extract solution was taken in test-tube and add 5 mL mayer`s reagent which gives orange precipitate of potassium-alkaloid confirm the presence of alkaloids.

3.4.2. Dragendroff's test

Initially, dragendroff reagent was prepared by mixing solution A and solution B. Solution A was prepared by dissolving 3 g bismuth nitrate in 8 mL 4 N H_2SO_4, similarly solution B was prepared by dissolving 12 g KI in 18 mL distilled water. Then, about 2 mL of the alkaloid extract solution was treated with Dragendorff reagent gives an orange-red precipitate of potassium- alkaloid indicates the presence of alkaloids.

3.4.3. Wagner's test

At the beginning, wagner`s reagent was prepared by dissolving 6 g KI and 2 g iodine in 100 mL distilled water. Then about 2 mL of the alkaloid extract solution treated with a few drops of Wagner's reagent give reddish-brown precipitate of potassium-alkaloid indicates the presence of alkaloids.

3.4.4. Methods to test others phytochemical

The presence of other phytochemical were test by following the given procedure.(Sharma et al., 2020)

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Test for Flavonoids

Ammonia test: 5 mL of dil. NH_4OH was added in 2 mL extract and few drops of Conc H_2SO_4 from side of tube was added gives yellow colour indicating presence of flavonoids.

Shinoda Test: When 2 mL of extract was treated with 1 mL of 50% methanol solution in tiny Mg chunk was added and 2 mL con. HCl was further added which give red colour indicating presence of flavonoids.

Test for Terpenoids

2 mL of $CHCl_3$ was added in 2 mL extract and 3 mL of Conc H_2SO_4 from side of tube was added slowly that gives reddish brown color confirming presence of Terpenoids

Test for saponins

0.1 g of extract was dissolved in 20 mL distilled water. By taking 10 mL filtrated it was shake vigorously which form froth formation that indicate presence of saponins.

Test for Polyphenols

3 drops of 5% $FeCl_3$ was added in 2 mL extract and shake which give violet color confirming presence of polyphenols.

Test for Glycosides

3 drop of molish reagent was added in 2 mL extract followed by adding few drops of conc H_2SO_4 from side of tube gives violets rings at the two junctions of two liquids proof the presence of glycosides.

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 Bhaktapur, Nepal. Before each experiment, 3x3 cm (weight loss) and 2x2 cm (Polarization and EIS) coupons were mechanically polished with SiC paper of 80,150,320,600,1000,1500 grits and stored in desiccators by wrapped in filter papers with labeling. During each experiment, each metal sample was washed with hexane, sonicated in absolute ethanol and dried.

3.6. Weight Loss Measurements

The inhibition efficiency was studied by the weight loss methods. In this method, at first each metal coupon dimensional had been measured and recorded by using digital Vernier caliper. Then the weight of each coupon also had been recorded using 4 digits digital beam balance (Phoenix, ASN224). Then, two metal coupons one in acid solution with and without inhibitor were immersed in same condition. After immersion for particular time, metal coupons were taken out from the solutions, corrosion products were removed with the aid of brush, washed with acetone and kept overnight in a desiccator. Next day weight of coupon was measured again. By, this method the inhibition efficiency under varying concentration, immersed time effect and temperature effect was been studied.

Experiments were carried out to measure weight loss per unit surface area per unit time for acid without and with inhibitor of different concentrations viz 20 ppm, 40 ppm, 60 ppm, 80 ppm and 100 ppm. Similarly, weight loss per unit surface area per unit time were measured in acid without and with inhibitor of different concentration for 6 h immersion of metal coupons varying temperature at 298 K, 308 K, 318 K and 328 K.

After determination of weight loss, the corrosion rate, inhibition efficiency, and surface coverage by inhibitors can be calculated using the formula mentioned in equations

(1), (2), and (3).

Corrosion rate(C.R.) = $\frac{K\times\Delta W}{ATD}$ (Dhakal *et al.*,2022) … (1)

Inhibition efficiency (IE%) = $\frac{(W_2-W_1)}{W_2}\times 100$ (Bajgai *et al.*, 2022) … (2)

Surface coverage(θ) = $\frac{(W_2-W_1)}{W_2}$ (Karki *et al.*, 2022) …(3)

Where K=87600 (constant); ΔW= weight loss (g/cm^{2}h)

; 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 using three-electrode system with metal sample as working electrode, platinum as counter electrode (CE) and a saturated calomel electrode (SCE) coupled to a fine Luggin capillaries as reference electrode. Before each electrochemical test, metal sample was immersed in acid solution with and without inhibitor of different concentration (20, 40, 60, 80, 100 ppm) until a stable open circuit potential (OCP) was attained. Then, potentio-dynamic polarization and electrochemical impedance spectroscopy were carried out to calculates inhibition efficiency.

3.7.1. Potentiodynamic polarization

The potentio-dynamic polarization method provides useful information about the corrosion current density and mechanism of corrosion. The potentio-dynamic 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 ±300 mV from OCP with a scan rate of 1 mV/s and a maximum current of 10 mA was applied. Inhibition efficiency and a fraction of surface coverage were calculated using the formula in equations (4) and (5) respectively. Evaluation of corrosion potential, current, and Tafel slopes for all solutions in 0.5 h immersed conditions(Karki, et al., 2021).

Corrosion inhibition efficiency (IE %) = $\frac{I_{corr}-I^{*}_{corr}}{I_{corr}} \times 100$ (Karki *et al.*, 2021) (4)

Fraction of surface coverage by an inhibitor (θ) = $\frac{I_{corr}-I^{*}_{corr}}{I_{corr}}$ (5)

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

3.7.1 Electrochemical Impedance Spectroscopy:

The electrochemical impedance measurements will be carried out using sinusoidal voltage of 10 mV peak to peak at frequencies between 100 kHz to 0.01 Hz at OCP. Data obtained will be fitted using a proper circuit in Z view software 3.2b version. Real impedance is plotted against imaginary impedance to obtain Nyquist plot as shown in figure. The value of charge transfer resistance (R_{ct}) will be obtained from the diameter of Nyquist plot and corrosion inhibition efficiency is calculated by the equation **[6].**

$$\mathbf{IE\%} = \frac{\mathbf{R_{ct}-R^0_{ct}}}{\mathbf{R_{ct}}} \times \mathbf{100\%} \qquad \text{(Karki } \textit{et al.}\text{, 2021)} \qquad \textbf{[6]}$$

Where R_{ct} and R^o_{ct} are the charge transfer resistance on inhibitor solution and Acidic solution respectively

3.7.2. FT-IR analysis

The FT IR was performed in Amrit Lab by using an using Perkin Elmer Spectrometer 10.6.2 version where the alkaloids powders was examined with background corrected using propanol and obtained plots were described in results section.

CHAPTER 4

RESULTS AND DISCUSSION

4.1. Chemical test for alkaloids and other phytochemical analysis

Plant consist of various types of chemical constituents called phytoconstituent which help to perform various secondary function. The phytoconstituents were separated into various class and gives specific test with specific reagent. Thus, this qualitative analysis phenomena known as phytochemical screening.

Table 3: Result of Phytochemical screening of *Cissampelos pareira*

S.N	Test For Phytochemical	Name of Test	Result
1	Alkaloids	Dragondroff Test Wagner Test	+ Ve + Ve
2	Flavonoids	Ammonia Test Shinoda Test	- Ve - Ve
3	Polyphenols	$FeCl_3$ Test	- Ve
4	Terpenoids	Chloroform Test	- Ve
5	Saponin	Froth formation Test	- Ve
6	Glycosides	Ring Test	- Ve

After the successful experiment for the confirming the alkaloid, extracted from *Cissampelos pareira* stem gives the very distinct colour precipation as shown in given below.

Figure 4: Chemical Test of Alkaloid Extract

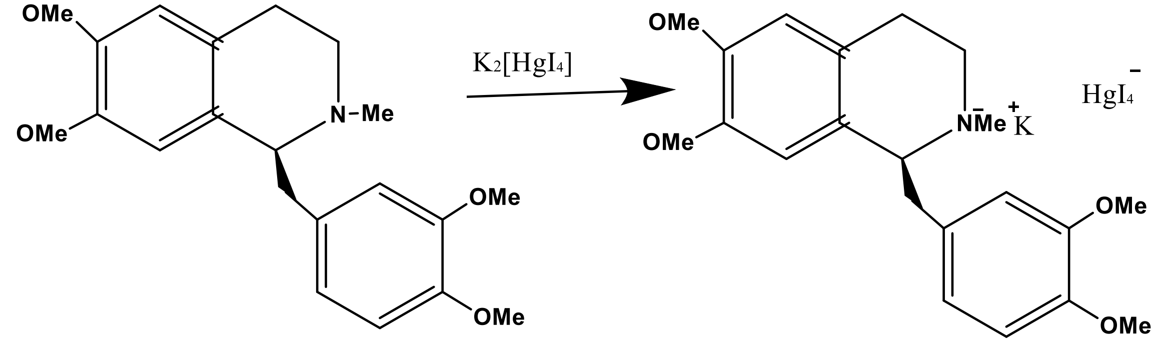
The alkaloids found in this plant were Laudanosine (benzylisoquinoline) and Nuciferine (Aporphine) alkaloids has been reported so the chemical reaction occurs with Dragendroff reagent , Wagner and Mayers regents to give respective precipitation.(Kumari et al., 2021)

4.1.1. Mayer`s reagent test with Alkaloids

About 2 mL alkaloid extract solution was taken in test tube and add 5 mL mayer`s reagent which gives orange precipitate of potassium-alkaloid confirm the presence of alkaloids. The reaction involved with alkaloids taking Laudanosine alkaloids as reference.

$$HgCl_2 + 2KI \longrightarrow HgI_2 + KCl$$

$$HgI_2 + 2KI \longrightarrow K_2[HgI_4]$$



Laudanosine(Benzylisoquinoline)

potassium alkaloids salt

4.1.2. Dragendroff reagent test with Alkaloids

About 2 mL of the alkaloid extract solution was treated with Dragendorff's reagent gives an orange-red precipitate of potassium- alkaloid indicates the presence of alkaloids. The reaction involved in this test was given below by taking a laudanosine alkaloids as reference.

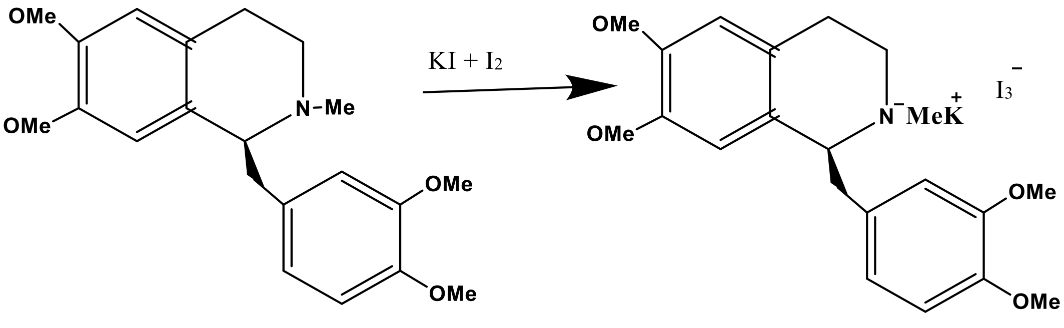
$$Bi(NO_3)_3 + 3KI \longrightarrow BiI_3 + 3KNO_3$$

$$BiI_3 + KI \longrightarrow K[BiI_4]$$

4.1.3. Wagner reagent test with Alkaloids

About 2 mL of the alkaloid extract solution treated with a few drops of Wagner's reagent give reddish-brown precipitate of potassium-alkaloid indicates the presence of alkaloids. The reaction involved in this test was given below by taking a Laudanosine alkaloids as reference.

$$I_2 + KIs \longrightarrow KI_3$$



Laudanosine(Benzylisoquinoline)

potassium alkaloids salt

4.2. FT- IR analysis

As we know that the FT-IR spectrum give the information about the presence of organic compound either it was heteroatom, aromatic ring or presence of multiple bond and functional group in that compounds. The adsorption of organic molecules presents in an extract of *Cissampelos pareira* plant over mild steel as inhibitor can be detected using FTIR spectrum as the chemical constituents were found to be different. Spectra of alkaloids extracted from the stem of the *C.pareira* was analysis using Perkin Elmer Spectrometer 10.6.2 version with correcting background using isopropanol .

The board peak seen near the region of 3198-3484 cm $^{-1}$ was due to combination of both O-H stretching and N-H stretching which may be secondary amine (Oli et al., 2022). Similarly, the sharp peak at 2927 cm $^{-1}$ and 2854 cm $^{-1}$ was of aliphatic CH group. NH bending was seen near the wavelength of 1604 cm $^{-1}$ and 1506 cm $^{-1}$, C=O stretching has be seen near 1664cm $^{-1}$ or it may be C-N stretching (Kumari et al., 2021). C-O-H bending peak was seen near the 1440 cm $^{-1}$ and at 1265 cm $^{-1}$ and 1225 is of C-O stretching and secondary C-N stretching of aromatic amine. Symmetry C-O-C stretching was seen near the 1028 cm $^{-1\cdot}$,whereas absorbance at 950 cm $^{-1}$ shows OH bending ,at 838 cm $^{-1}$,521 cm $^{-1}$ shows C-H bending (Karki et al., 2021).

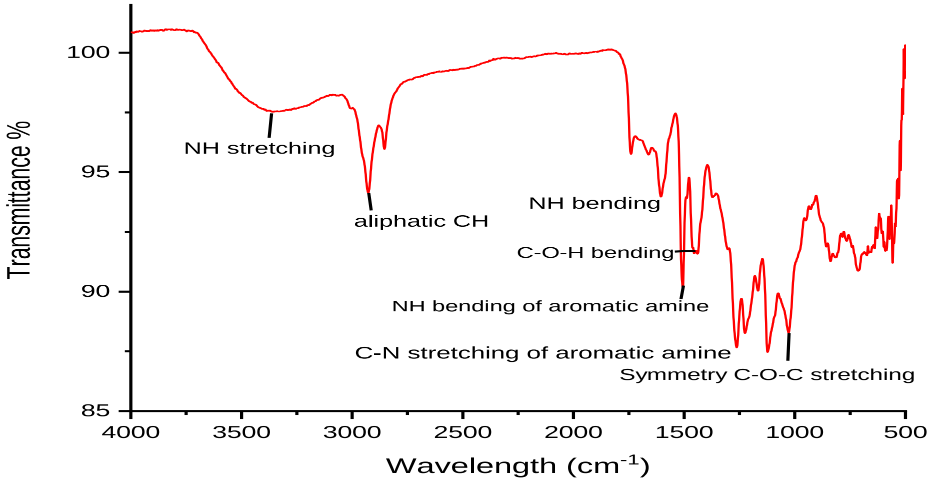


Figure 5: FT-IR Spectrum

Table 4:Various functional group stretching and bending in respective Wavelength

Wavelength cm $^{-1}$	Functional Group stretching and bending
3198-3484	OH /NH stretching
2927,2854	Aliphatic CH group
1604-1506	NH bending
1664	C=O stretching or C-N stretching
1440	C-O-H bending
1265	C-O stretching
1225	C-N stretching of aromatic amine
1028	Symmetry C-O-C stretching
950	OH bending
838, 521	CH bending

4.3. Weight loss methods

During weigh loss method the when the MS coupons were immersed in an either acidic medium or inhibition medium then there will be formation corrosive product. Thus, when the amount of corrosive product is high there will be loss of metal coupon weight, the amount of weight loss will determine whether which inhibitor solution was best. The amount of weight loss was calculated by difference in an initial weight of metal coupons before immerse and after immersed measured by using a 4-digit digital beam balance. In this method we are studying 3 main parameters i.e. concentration effect, temperature effect and immerse time effect.

4.3.1. Effect of immersion time on MS Weight loss

The effect of immersion time on MS coupons were done under room temperature i.e at 25˚C. The effect of immersion time on IE% was calculated at different time intervals i.e. 0.5 h, 1 h, 3 h, 6 h, 18 h, and 24 h by using weight loss methods. The amount of weight loss was shown in given below table at different time interval.

Table 5: Weight loss and inhibition efficiency calculated at different time and concentration

Concentration (ppm)	time (h)	surface area (cm^2)	weight loss(g)	weight loss/surface area time (g/cm^2)	corrosion rate(mm/year) C.R = $\frac{K \times \Delta W}{ATD}$	IE (%) = $\frac{(W_2-W_1)}{W_2} \times$ 100%
0	0.5	20.21	0.0181	0.0009	0.20	
	1	20.09	0.0384	0.0019	0.21	
	3	20.59	0.1089	0.0053	0.20	
	6	20.31	0.1565	0.0077	0.14	
	18	20.77	0.5356	0.0258	0.16	
	24	19.35	0.7144	0.0369	0.17	
20	0.5	21.69	0.0044	0.0002	0.05	77.39
	1	20.76	0.0053	0.0003	0.03	86.59
	3	21.10	0.0086	0.0004	0.02	92.23
	6	20.44	0.0115	0.0005	0.01	92.67
	18	20.59	0.0194	0.0009	0.01	96.33
	24	19.48	0.0315	0.0016	0.01	95.62
40	0.5	21.06	0.0031	0.0002	0.03	83.22
	1	20.32	0.0046	0.0002	0.03	88.17
	3	20.62	0.0082	0.0004	0.01	92.48
	6	21.05	0.0116	0.0005	0.01	92.84
	18	20.87	0.0190	0.0009	0.01	96.46
	24	20.70	0.0243	0.0011	0.01	96.82
60	0.5	20.81	0.0030	0.0001	0.03	83.56
	1	20.37	0.0038	0.0001	0.02	90.25
	3	20.83	0.0065	0.0003	0.01	94.09
	6	20.36	0.0077	0.0004	0.01	95.07
	18	19.88	0.0127	0.0006	0.01	97.52
	24	20.09	0.0239	0.0012	0.01	96.77
80	0.5	19.74	0.0027	0.0001	0.03	84.75
	1	20.24	0.0033	0.0001	0.02	91.33
	3	20.20	0.0033	0.0001	0.01	94.71
	6	19.61	0.0061	0.0003	0.01	95.91
	18	21.14	0.0092	0.0004	0.002	98.29
	24	19.10	0.0193	0.0010	0.004	97.26
100	0.5	21.10	0.0023	0.0001	0.02	87.85
	1	21.20	0.0032	0.0001	0.01	92.05
	3	21.47	0.0051	0.0002	0.01	95.47
	6	20.75	0.0061	0.0003	0.01	96.16
	18	19.83	0.0046	0.0002	0.001	99.09
	24	19.10	0.0193	0.0010	0.004	97.42

Table 6:Weight loss (g/cm^{2}h) of MS that was immersed in the acid solution in the absence and presence of inhibitor at different immersion time (h) which was obtained from table 5.

Hours	Acid	20 ppm	40 ppm	60 ppm	80 ppm	100 ppm
0.5	8.97 x 10^{-4}	2.03 x 10^{-4}	1.51 x 10^{-4}	1.48 x 10^{-4}	1.37 x 10^{-4}	1.09 x 10^{-4}
1	1.91 x 10^{-3}	2.57 x 10^{-4}	2.26 x 10^{-4}	1.87 x 10^{-4}	1.66 x 10^{-4}	1.63 x 10^{-4}
3	5.29 x 10^{-3}	4.18 x 10^{-4}	3.98 x 10^{-4}	3.12 x 10^{-4}	2.39 x 10^{-4}	1.93 x 10^{-4}
6	7.71 x 10^{-3}	5.52 x 10^{-4}	5.65 x 10^{-4}	3.80 x 10^{-4}	3.15 x 10^{-4}	2.96 x 10^{-4}
18	2.58 x 10^{-2}	9.12 x 10^{-4}	9.45 x 10^{-4}	6.39 x 10^{-4}	4.38 x 10^{-4}	2.34 x 10^{-4}
24	3.69 x 10^{-2}	1.62 x 10^{-3}	1.17 x 10^{-3}	1.19 x 10^{-3}	1.01 x 10^{-3}	9.51 x 10^{-4}

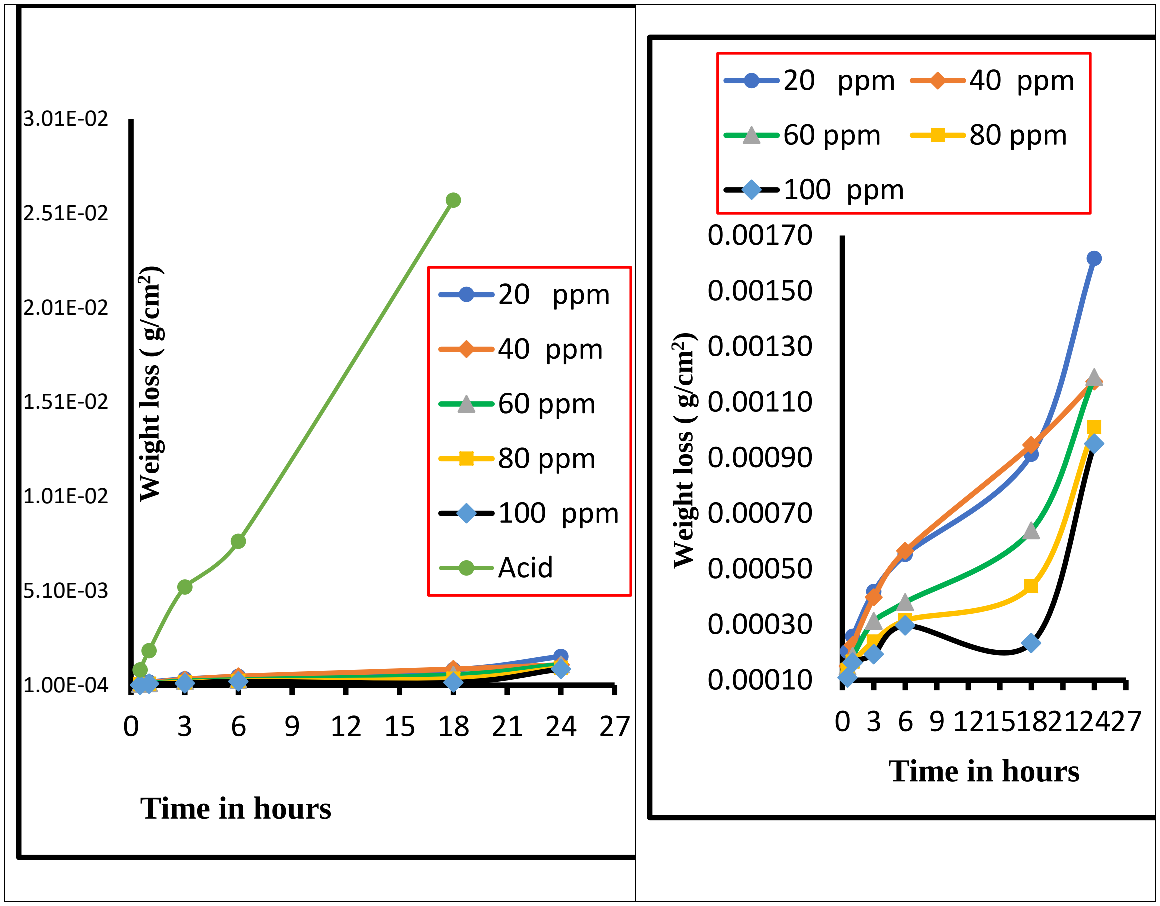


Figure 6: Comparative study of Weight loss (g/cm^2) of MS that was immersed in the acid solution in the absence and presence of inhibitor at different immersion time (h) (a): Acids and inhibitors (b) Between inhibitors

The result obtained from this experiment show that the amount of weight loss of MS was found to increase with increase acid without or with inhibitor of all concentration. It was found that the weight loss in acidic medium was very higher as compare to an inhibitor solution as in figure 6(a). As prolonged immersed in solution there will be higher contact with corrosive medium as compare to lesser time due which weigh loss were more with time as seen in graph below figure 6 (a) and (b). The trend of graphs was found to be almost same and similar in pattern.

4.3.2. Effect of immersion time on Inhibition Efficiency

An effect of immersion time on Inhibition efficiency was shown in given figure 7, below. It shows that inhibition efficiency was goes on rising to 18 h whereas after 18 hours the inhibition efficiency goes on slightly decrease this may be due to desorption of the alkaloids used in as inhibitor solution. The alkaloids may formed chelate complex with Fe^{++} and which decrease the availability of inhibitor molecules after long time which results the decrease in IE with increase in time. This experiment also show that the inhibition efficiency was maximum i.e. 99.09% at 100 ppm immersed at 18 hours whereas minimum is 77.39% at 20 ppm immersed at 0.5 h. But in this experiment the optimum time was taken as 6 hours with efficiency 92.67% and 96.16% at 20 ppm and 100 ppm respectively.

Likewise, when inhibition efficiency was studied by various researcher in the context of nepali plant, the best IE % was obtained at only higher concentration solution (1000 ppm)(Dhakal *et al.*, 2022; Karki *et al*., 2020; Karki, et al., 2021) but in this research activity 50 times dilution inhibitor solution (at 20 ppm) also shows IE 92.63% at optimum time as compared to that of 1000 ppm inhibitor solution.

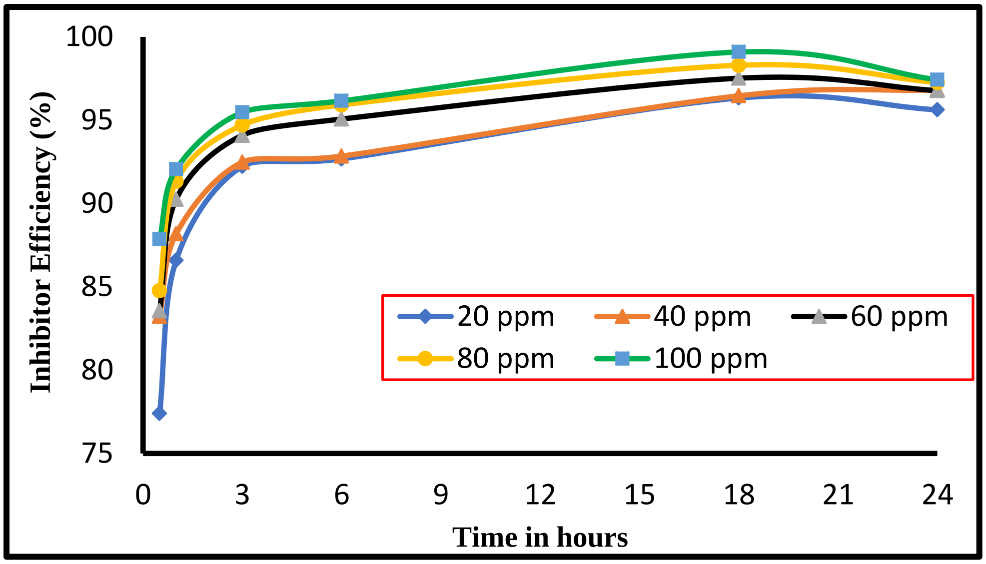


Figure 7:Inhibition efficiency of inhibitor in 1 M HCl solution for the corrosion of mild steel immersed at a different time interval

4.3.3. Effect of inhibitor concentration in weight loss

As we know the weight loss of MS will occur in contact with corrosive medium forming a corrosive product. The higher amount of corrosive product indicates the higher in weight loss. The formation of corrosive product can be somehow minimized using an inhibitor. Thus, the weight loss of MS was indirectly proportional to the concentration of the inhibitor used. So, in an experiment's inhibitor in acid varying concentration (20, 40, 60, 80 and 100 ppm) were used. The maximum weight loss was seen in lower concentration i.e. 20 ppm whereas minimum weight loss occurs at higher concentration.The loss of MS was seen higher at an acidic solution whereas negligible weight loss acid with inhibitor which is illustrated in figure 8a. In the figure 8b, the comparative study of weight loss of MS per unit area at varying concentration is shown where less weight loss is observed with increase in concentration of inhibitor solution. Decrease in weight loss of MS with increase in concentration is due to the adsorption of inhibitor molecules on MS surface to form barrier film on MS surface to minimize the contact with corrosive medium. In 100 ppm inhibitor solution when MS immersed for 18 h and 3 h, shows lesser weight loss comparative to 6 h and 1 h immersion respectively. This is because of adsorption of alkaloids molecules follow by desorption due to repulsion between alkaloids molecules and slightly developed of weak vander wall force with metal surface.

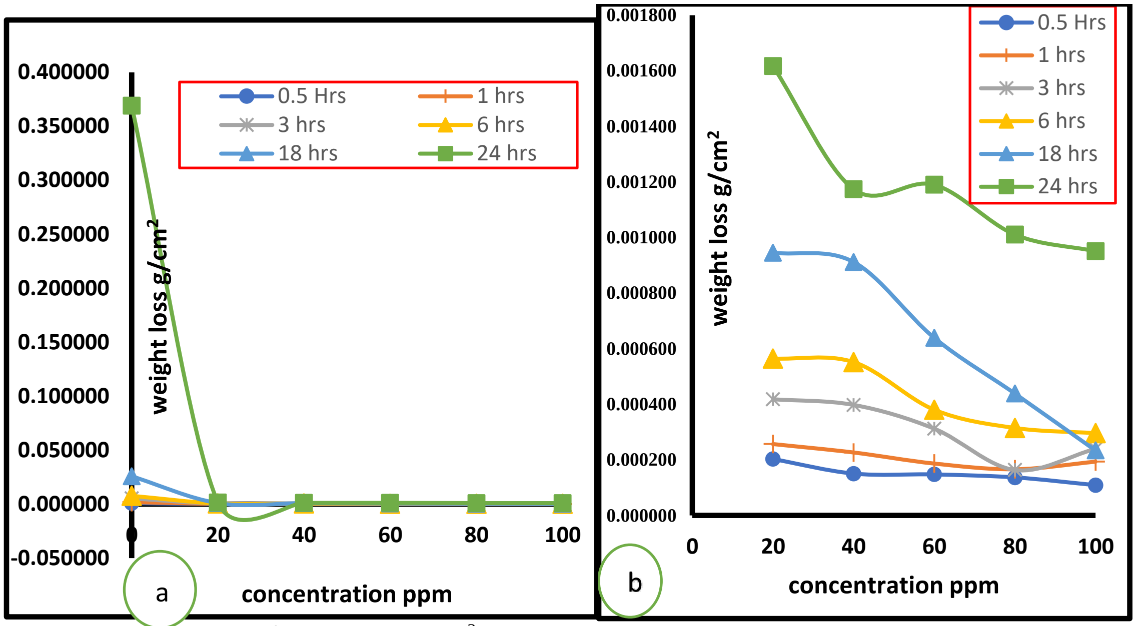


Figure 8: Variation of weight loss(g/cm^2)(a)acids and inhibitor at various times (b) between various inhibitors at various times.

4.3.4. Variation in Inhibition Efficiency of Inhibitor concentration

In this study, the IE% was found to be higher at higher concentration. At higher concentration of inhibitor (100 ppm) an inhibition was found to maximum this is due to fact that more molecules were available to adsorbed on MS surface. At optimum time 6 h and at 3 h the efficiency was nearly same due that the molecule gets enough time for almost complete adsorption over MS surface. The maximum inhibition was found at 99.08% for 18 h, whereas at optimum time (6 h) it was 96.16% for 100 ppm whereas minimum for 100 ppm was 87.85% at 0.5 h immersed time periods.

Table 7:Variation of inhibition efficiency with the concentration of inhibitor solutions which was obtained from **table 5**

	Efficiency Vs Concentration					
	1 hrs	**3 hrs**	**6 hrs**	**18 hrs**	**0.5 hrs**	**24 hrs**
20	86.59	92.23	92.68	96.33	77.39	95.62
40	88.17	92.48	92.84	96.46	83.23	96.82
60	90.25	94.10	95.07	97.52	83.56	96.78
80	91.33	94.71	95.92	98.30	84.76	97.26
100	92.05	95.47	96.16	99.09	87.86	97.43

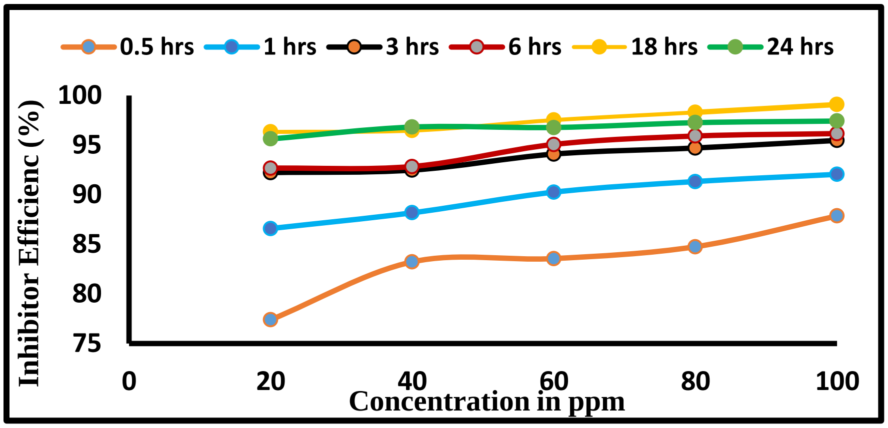


Figure 9:*Variation of inhibition efficiency with the concentration of inhibitor solutions*.

Likewise, when inhibition efficiency was studied by various researcher in the context of nepali plant, the best IE % was obtained at only higher concentration solution (1000 ppm) but in this research activity 50 times dilution solution inhibitor solution (20 ppm) also shows IE 92.63% at optimum time as compared to that of 1000 ppm inhibitor solution.

4.3.5. Effect of temperature on weight loss

Another factor which affect the weight loss of MS was temperature. Increase in weight loss may directly affect the corrosion rate and IE%. The effect of weight loss had been studies

Table 8: Variation of weight loss(g/cm^2) of MS with temperature in the presence and absence of inhibitor taken from the table 9

Concentration	298 K	308 K	318 K	328 K
Acid	0.008	0.0200	0.0300	0.0150
20 ppm	0.0006	0.0012	0.0040	0.0090
40 ppm	0.0006	0.0010	0.0030	0.0060
60 ppm	0.0004	0.0007	0.0020	0.0050
80 ppm	0.0003	0.0009	0.0020	0.0040
100 ppm	0.0003	0.0008	0.0020	0.0030

Table 9:Weight loss and inhibition efficiency calculated at different temperature and concentration at 6 hours immersion time.

Temperature	Concentration (ppm)	surface area (cm^2)	weight loss (g)	weight loss/surface area time (g/cm^2)	corrosion rate (mm/year) = $\frac{K \times \Delta W}{ATD}$	IE (%) = $\frac{(W_2-W_1)}{W_2} \times 100\%$
298 K	0	20.31	0.1566	0.0077	0.14	-
	20	20.44	0.0115	0.0006	0.01	92.67
	40	21.05	0.0116	0.0006	0.01	92.84
	60	20.36	0.0077	0.0004	0.007	95.07
	80	19.61	0.0062	0.0003	0.006	95.91
	100	20.75	0.0061	0.0003	0.005	96.16
308 K	0	20.11	0.3907	0.0194	0.24	-
	20	20.86	0.0300	0.0014	0.02	92.58
	40	20.40	0.0232	0.0011	0.01	94.15
	60	20.58	0.0154	0.0007	0.009	96.15
	80	20.22	0.0181	0.0009	0.01	95.39
	100	19.56	0.0159	0.0008	0.01	95.80
318 K	0	20.90	0.6737	0.0322	0.40	-
	20	20.01	0.0728	0.0036	0.05	88.71
	40	20.43	0.0550	0.0027	0.03	91.64
	60	20.54	0.0468	0.0023	0.03	92.94
	80	20.29	0.0380	0.0019	0.02	94.18
	100	19.63	0.0355	0.0018	0.02	94.39
328 K	0	20.31	0.3128	0.0154	0.20	-
	20	19.82	0.1840	0.0093	0.11	39.72
	40	21.05	0.1158	0.0055	0.07	64.28
	60	20.33	0.1077	0.0053	0.06	65.58
	80	20.19	0.0882	0.0044	0.05	71.62
	100	19.45	0.0633	0.0033	0.04	78.87

at varying condition i.e. 298 K, 308K, 318 K, 328 K. The weight loss obtained at different temperature at optimum time (6 hours) in varying concentration was given at above table 9 and line graph 10.

These above data figure 10 show that the weight loss (g/cm^2) of MS goes on increasing with increase in temperature. This is due to fact that desorption or decomposition of inhibitor molecules occur from MS surface at higher temperature.

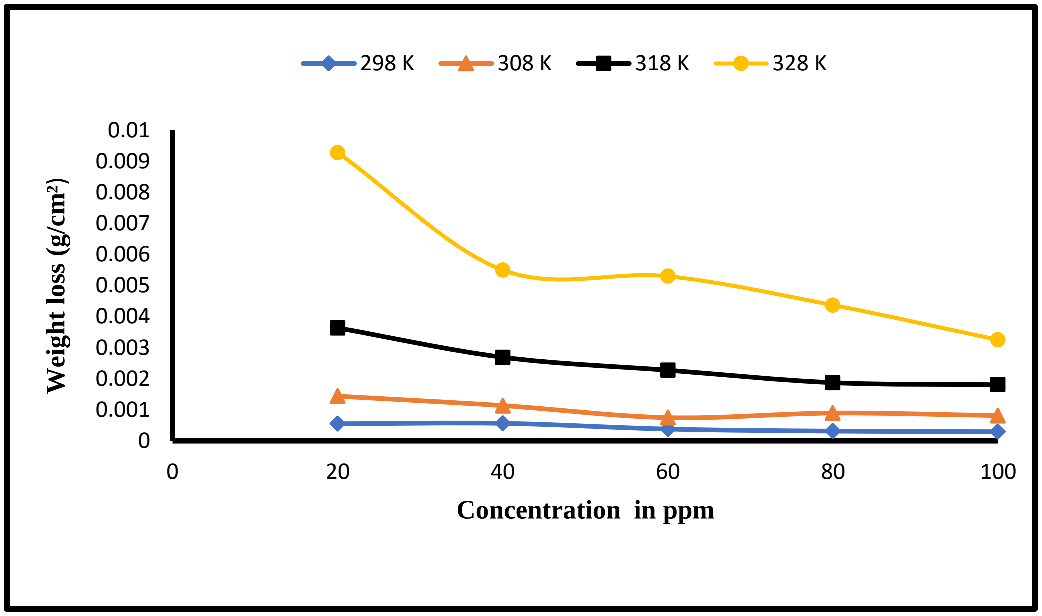


Figure 10: Variation of weight loss of MS with temperature in the presence and absence of inhibitor.

The Fig 11: shows the efficiency of inhibitor efficiency and concentration at different temperature when immersed for 6 h. It was found that the I.E % was increased with increase in concentration at every temperature (except at 60 ppm, 80 ppm at 308 K). IE% was slightly decrease (negligible change) with increase in temperature for respective inhibitor concentration because physical adsorption process start to dissociate with increase in temperature whereas chemical adsorption is still dominating till 318 K which was also support by ΔG but the drastic change was seen at 328 K due to fact that chemical adsorption as well start to dissolution at higher temperature but still, higher concentration inhibitor solution (100 ppm) can use as good inhibitor at high temperature as IE% was found to be 78.87%.

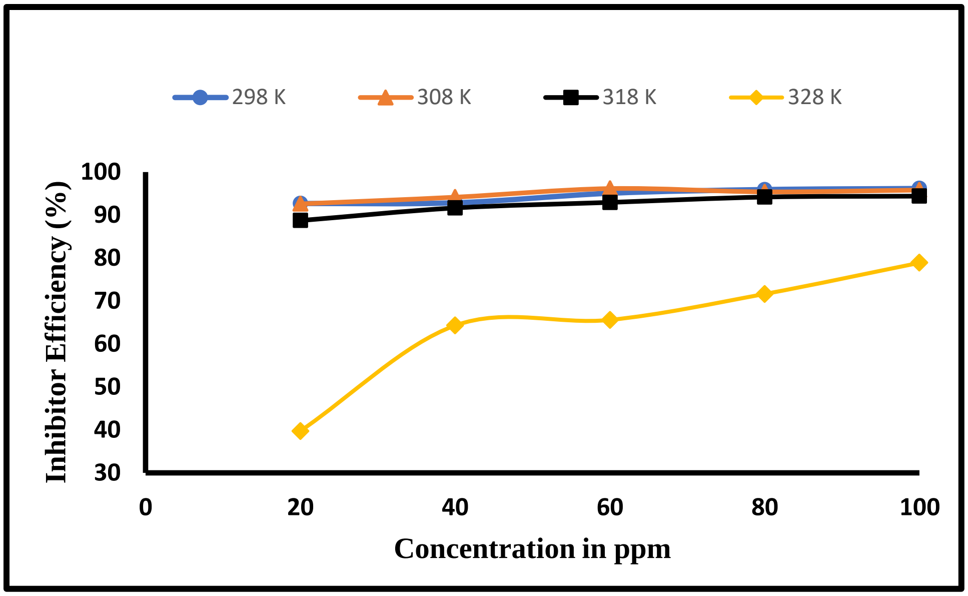


Figure 11: Efficiency Vs Concentration at varying temperature

4.4. Adsorption Isotherm

In this experiment the adsorption isotherms provide an important information that how the molecules were adsorbed over the MS surface when they were immersed at different inhibition concentration at ordinary lab temperature. The adsorption of molecules depend upon various factors like temperature, adsorbed molecular structure/size, and chemical composition and electro chemical potential at metal/solution surface (Karki *et al.*, 2020) To study the adsorption isotherm, at first MS were immersed at varying concentration inhibitor solution at a laboratory temperature. Initially, the water molecules were adsorbed over its surface with increases with time these molecules were slowly replaced by the alkaloids bulky molecules, and blocked the active site of MS which automatically reduce the corrosion rate. Thus, the phenomena in which the water molecule may adsorbed and de-adsorbed at metal/solution interface know as quasi substitution process. Various adsorption isotherm such as Langmuir adsorption, Tempking, Freundlich were tested to know the Layer of adsorbed molecules, nature of adsorption, and free energy but the best fitting was given by the Langmuir adsorption isotherm (Karki *et al*., 2020).

The linear graph for Langmuir Adsorption isotherm was given by the below equation, where $\frac{C_{inh}}{\theta}$ plotted in Y axis and C_{inh} in X axis. When the value of slope and Linear correlation coefficient (R^2) is equivalent to 1 or little slope value deviated from 1 tell there was some interaction between adsorbed molecule and metal surface also due to unequal adsorption of inhibitor molecules occur on both cathodic and anodic site. Langmuir adsorption provides a valuable information i.e. weather the formed layer is monolayer or multiple layers.

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh}$$

The K_{ads} value can be calculated by knowing the value of intercept, and by putting the value of K_{ads} in a below relation, we can get value of ΔG (Karki *et al.*, 2020).

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

Table 10: Calculating value of Free energy of adsorption

S.N.	**Hours**	**Slope**	**Intercept**	K_{ads}	ΔG **(j/mol)**	$\Delta G(\frac{kj}{mol})$
1.	1	1.06	6.00×10^{-06}	$1.67\times10^{+05}$	$-3.97\times 10^{+04}$	-39.74
2.	6	1.02	4.00×10^{-06}	$2.50\times10^{+05}$	$-4.07\times10^{+04}$	-40.75
3.	18	1.00	3.00×10^{-06}	$3.33\times10^{+05}$	$-4.15\times10^{+04}$	-41.46

The calculated value of ΔG was found to -39.7427, -40.7474 and -41.4602 kJ/mol when calculated at 3 different time periods. The last two values were greater than threshold value i.e more than -40 kJ/mol which indicate chemical adsorption, but initial value was lies in a range between -20 kJ/mol to -40 kJ/mol which indicate it is mixed adsorption with domination of chemical adsorption.

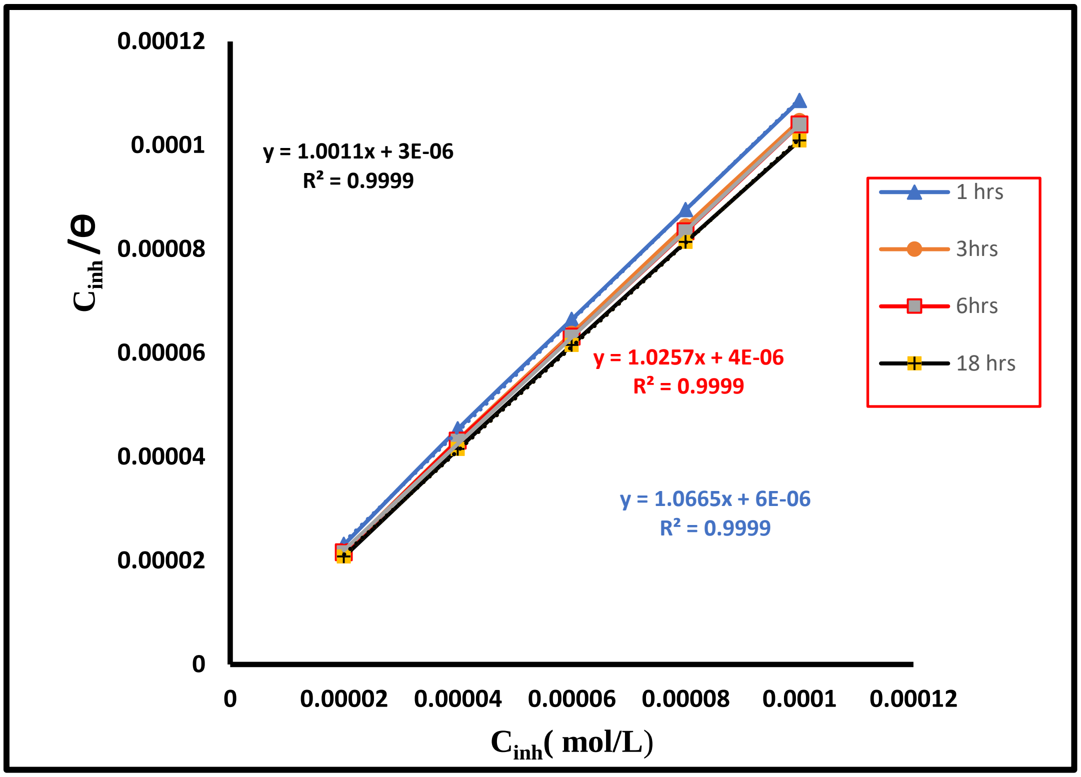


Figure 12:Langmuir adsorption Isotherm of corrosion of MS in 1 M HCl at different concentration

4.4.1. Activation energy and Corrosion Kinetics

As the inhibitor play the significant role in corrosion inhibition by blocking the site, thus by rearranging Arrhenius equation helps to predicts the activation energy of that reaction by assistant following equation (Karki *et al.*, 2020).

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

Where A is Arrhenius constant, R is rate constant, E_a activation Energy, CR, corrosion rate and T is Absolute temperature. The above linear equation tells us that E_a is obtained from a slope of straight line and intercept give log A value. The increased value of E_a with increase in concentration suggest that the adsorption mechanism is physical-adsorption mechanism whereas decrease value of E_a with increase in concentration suggest the mechanism is chemo-adsorption mechanism (Ekanem *et al.*, 2010).

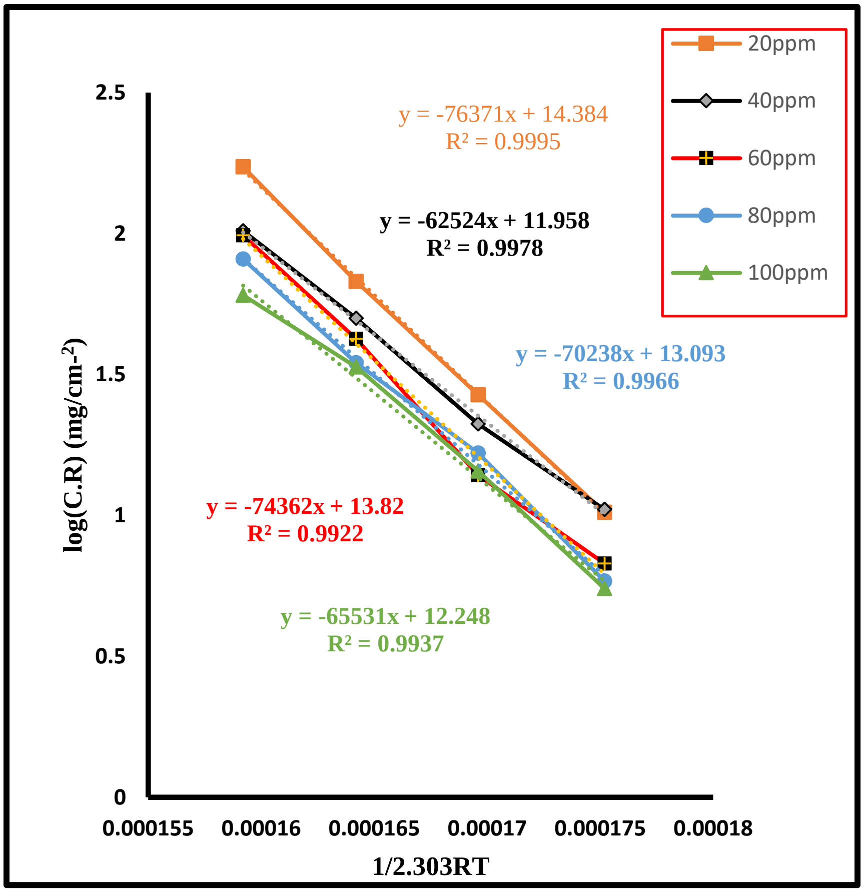


***Figure 13*:**Arrhenius plot wherein x-axis 1/2.303 RT and Y axis consist log (C.R) (mg/cm-^{2})

4.4.2. Thermodynamics of Corrosion and Mode of Inhibition

An alternative form Arrehnius equation help to determine the Enthalpy and Entropy and which shown in below equation (Karki *et al.*, 2020).

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

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.

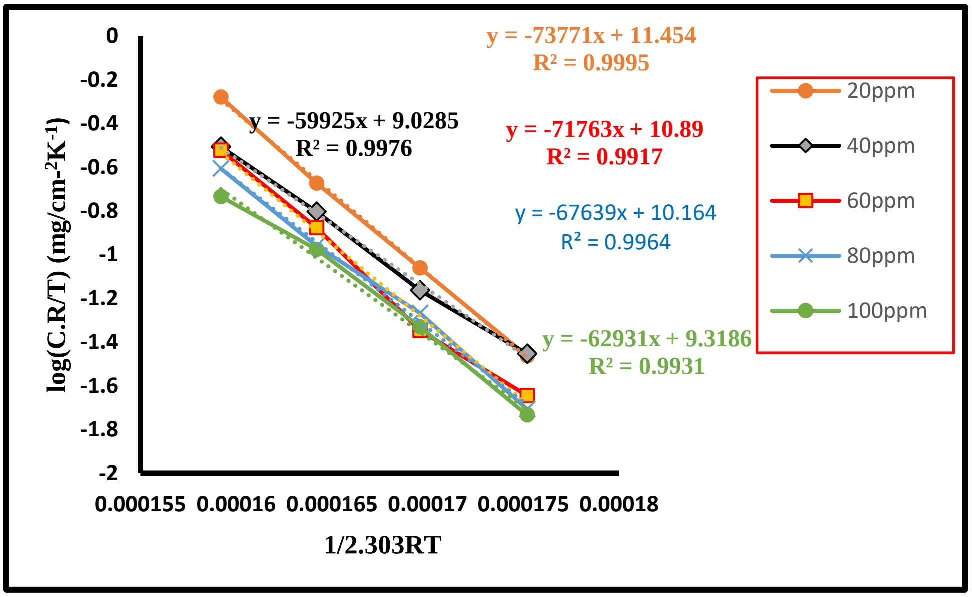


Figure 14: Transition state plot for MS in 1 M HCl where X axis consist 1/2.303RT and Y axis consist log (C.R/T) (mg/cm-^{2}K^{-1})

Table 11: Thermodynamic and activation parameters from Langmuir adsorption isotherms of corrosion of MS in 1.0 M HCl at varying concentration

S.N.	**Concentration**	**Ea (Kj/mol)**	**Log A**	***ΔS***	**Δ*H* (Kj/mol)**	**Ea- Δ*H* = RT = (2.60)**
1.	20 ppm	76.371	14.384	21.735	73.771	2.600
2.	40 ppm	62.524	11.958	-24.706	59.925	2.590
3.	60 ppm	74.362	13.820	10.937	71.763	2.590
4.	80 ppm	70.238	13.093	-2.964	67.639	2.590
5.	100 ppm	65.531	12.248	-19.151	62.931	2.600
6.	Acids	56.565	12.094	-21.987	54.000	2.560

The positive value of Δ***H*** indicat**e** the process is endothermic (Karki, *et al.*, 2021). The comparison value of E_a and Δ***H*** show that E_a value is maximum which suggest participation of gaseous reaction H_2 gas formation at cathodic site and decrease the total reaction volume. The difference between activation energy and the enthalpy (range 2.6-2.56 kJ/mol) was nearly equivalent to value of product of RT (2.602 kJ/mol) that imply that the corrosion process is unimolecular reaction (Lama *et al.*, 2018;Ekanem *et al.*, 2010) .

The negative sign of entropy in Table **11** (at 40,80, and 100 ppm) show that there was formation of activated complex in rate determining step was association rather than dissolution, which means decrease in disordering take place while going from reactant to activation complex. As the more negative value of entropy indicates high order behavior with increase in inhibitor concentration and minimize corrosion rate (Ekanem *et al.*, 2010;Eddy and Odoemelam, 2009). It was also found that the randomness decreases with increase in concentration of inhibitor as value of entropy decrease with increase in concentration because of mixed adsorption, with domination of chemo-adsorption process.

4.5. Electrochemical Measurement

4.5.1. Polarization Measurement

Polarization measurement was carried on electrochemical laboratory, central department of chemistry, Kirtipur, Tribhuvan University, using 3 electrode system with metal as working electrode, Pt as counter and saturated Calomel electrode as reference electrode, in acid in presence and absence of inhibitor. The concentrations of inhibitor used were 20,40,60,80 and 100 ppm. The current density was found to be 13.54×10^{-6} after immersed for half an hour, which was gradually decrease with increase in concentration of inhibitor solution. The shifted value of OCP for different concentration solution from acid was found to be less than 85 mV which indicate the inhibitor is mixed type (Karki *et al.*, 2021). The cathodic and anodic Tafel slope remain in the range of 15.29 mV/dec to 3.15 mV/dec. The current density was nearly 68 time lesser for acid with inhibitor of 100 ppm concentration as compared to that in acid. This decrease in current density is due to resistance developed by blocking the surface of reactive site by bulky molecules which minimized the corrosion rate.

We can see that cathodic and anodic polarization curve show similar pattern that suggest mechanism of corrosion has not been affected by alkaloids molecules (Gupta *et al.,* 2020). Parallel cathodic curves indicate that the hydrogen evolution is activation-controlled, which does not change the reduction mechanism (Benabdellah *et al.,* 2006). In most of the

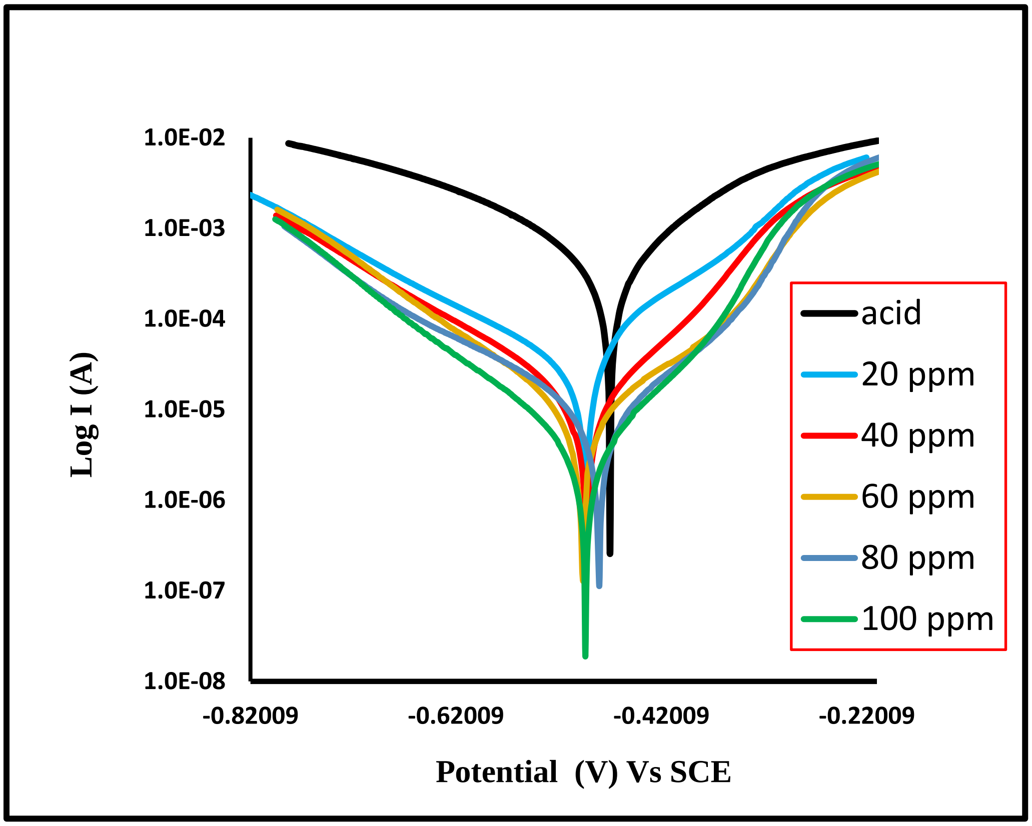


Figure 15: Tafel plots

Table 12: Shows the anodic, cathodic slope and IE immersed for 0.5 hours

S.N	Medium	E_{corr}(mV)	I_{corr}(µA)	Anodic slope (mV/dec)	Cathodic slope -ve (mV/dec)	IE %
1.	Acidic	460	130.000	5.60347	3.15234	-
2.	20 ppm	485	11.300	14.55824	5.84903	91.30%
3.	40 ppm	483	4.900	14.14482	8.9595	96.23%
4.	60 ppm	488	3.096	8.96607	9.31846	97.61%
5.	80 ppm	472	2.720	12.73033	6.14451	97.90%
6.	100 ppm	486	1.910	14.25133	9.00203	98.53%

research paper suppression in either cathodic current density or in very few anodic current density were observed but here both cathodic and anodic current density have been suppressed indicating that both the H_2 liberation on cathodic site and metal dissolution on anodic site are decrease significantly which is due to adsorption of bulky alkaloids groups on MS ,minimize the charge transfer (Karki *et al*., 2021).

4.5.2. Electrochemical Impendence (EIS):

The effect of *C.P* alkaloids was studied at steady-state conditions by impedance measurements of the MS in 1.0 M HCl and *C.P* extract various concentrations (20,40,60,80,100 ppm) at OCP. Below figure represent the Nyquist plot with fitted and unfitted data using Z-view software(3.2b), where, fitted data were represented by solid line whereas unfitted by dotted line having various symbolic representation. The bode phase plots at various concentration of C.P extract and simple equivalent circuit was
shown in below figure 18.

The Nyquist plot shows that there is single charge transferred mechanism and nature of curved was also similar which indicate that the corrosion inhibitor mechanism in mild steel was also in similar pattern called single relaxation process as suggested by various paper (Karki et al., 2021) . At high frequency in Nyquist plot, only one depressed capacitive loop was found that revels to time constant of the electric double layer and charge transfer resistance along with roughness and non-homogeneity seen during adsorption process because of frequency dispersion. As in above Nyquist figure 17 the diameter of the semi-circle curved (capacitive loop) was increase with increased in concentration of an inhibitor which is due to higher resistance developed over the mild steel at higher concentration and minimum the charge transfer was carried out because large number of inhibitor molecules are available and considerable surfaced get covered. Due to minimum charge transferred the corrosion rate of mild steel was very less at higher concentration.

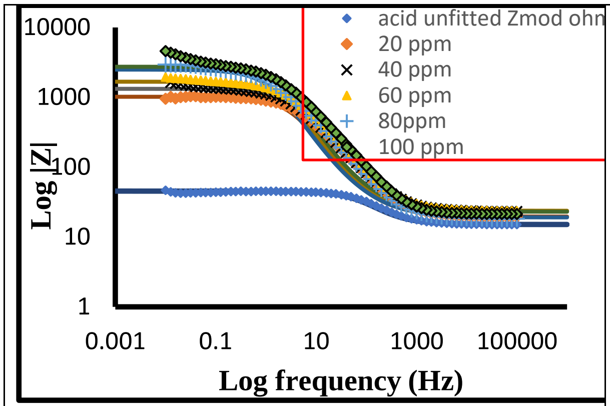


Figure 16: Bode modulus Plot: z modulus vs. frequency for mild steel in 1.0 M HCl without and with CP extract of different concentrations

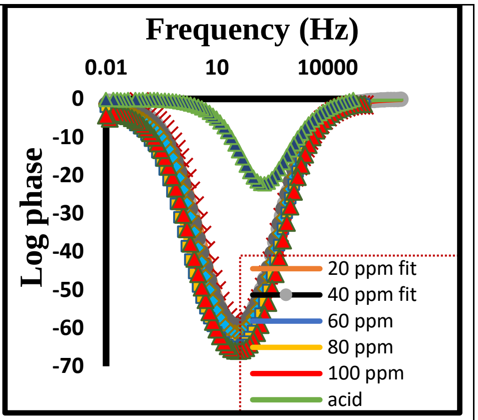


Figure 17: Bode Phase Plot: phase angle vs. frequency for mild steel in 1.0 M HCl without and with CP extract of different concentrations

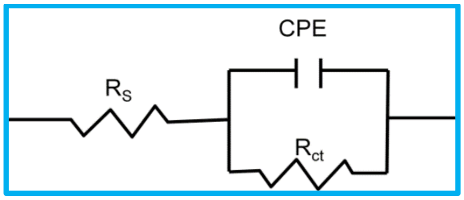


Figure 18: The equivalent circuit used to fit impendence spectra

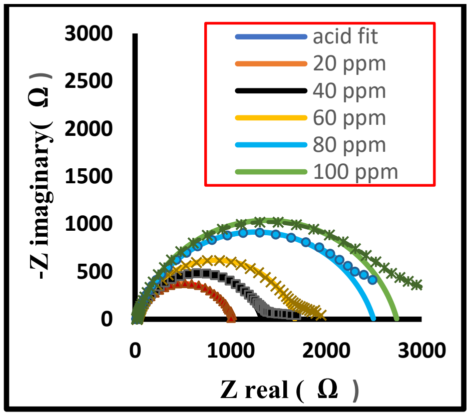


Figure 19: Nyquist plot

In Bode phase diagram increasement in phase angle with inhibitor concentration and in bode modulas diagram higher value of z Modulas at lower frequency provide supporting evidence that inhibition increase with increase in concentration of inhibitor solution. As the single peak was observed which symbolized one time constant for study material mostly related to electric double thus, equivalent circuit as shown in fig was used to analyze the impendence spectra that includes, solution resistance (Rs), charge transfer resistance (Rct), and constant phase element (CPE)

The Constant phase element (CPE) is used in the circuit model to take in account the electrode surface non-homogeneity caused by surface roughness, inhibitors adsorption, grain boundaries, and the creation of a porous layer in corrosion of metal in aggressive acidic media. n is the CPE exponent ($-1 \leq n \leq +1$), whose value is used to evaluate the surface's non-homogeneity or roughness. The CPE depicts a pure resistor when n=0, an inductor when n= -1, and a pure capacitor when n= +1

The impendence function is given by the relation (Karki,*et al.*, 2021).

$$Z\ CPE = \frac{1}{Q(j\omega)^n}$$

Where Q = magnitude of CPE

j = imaginary number

ω = angular frequency (2πf)

n = CPE exponent

***Table 13*:** Impedance parameters for corrosion of mild steel in 1.0 M HCl without and with CP extract.

Medium	$R_s\Omega cm^2$	$R_p\ \Omega cm^2$	CPE ($\mu\Omega^{-1}S^n\ cm^{-2}$	n	IE%
Acid	15.10	30.29	184.01	0.80	-
20 ppm	19.60	998.70	70.31	0..81	96.96
40 ppm	23.26	1300.00	70.87	0.81	97.67
60 ppm	23.74	1650.00	60.40	0.82	98.16
80 ppm	19.60	2478.00	68.06	0.81	98.77
100 ppm	23.02	2717.00	42.95	0.83	98.88

The value of CPE goes on decreasing with increase in concentration of inhibitors (except at 40 ppm and 80 ppm) because of decrease in local dielectric constant. This result can be explained on the basis of formation of double layer increases with increase in concentration because large numbers of molecules are available and get adsorbed by replacing water molecules over MS and form protective layer. Thus, increasing value of CPE also provide proof for supportive statements that, inhibition efficiency rises with rise in concentration of an inhibitors.

4.6. Mechanism of Corrosion inhibition

The mechanism of corrosion inhibition over mild steel in acidic solution can be easily explain on the basis of the size of molecules adsorbed over it by replacing the adsorbed water molecules but the effectiveness of inhibition was explained on the basis of bond it can formed with the mild steel. This nature of bond formed explained on basis of amount of Gibbs free energy and activation energy (Karki, *et al.*, 2021;Thapa Magar *et al.*, 2023)

$$\boxed{Org_{(sol)} + H_2O_{\text{Ads}} \Rightarrow Org_{\text{Ads}} + H_2O_{\text{Sol}}}$$

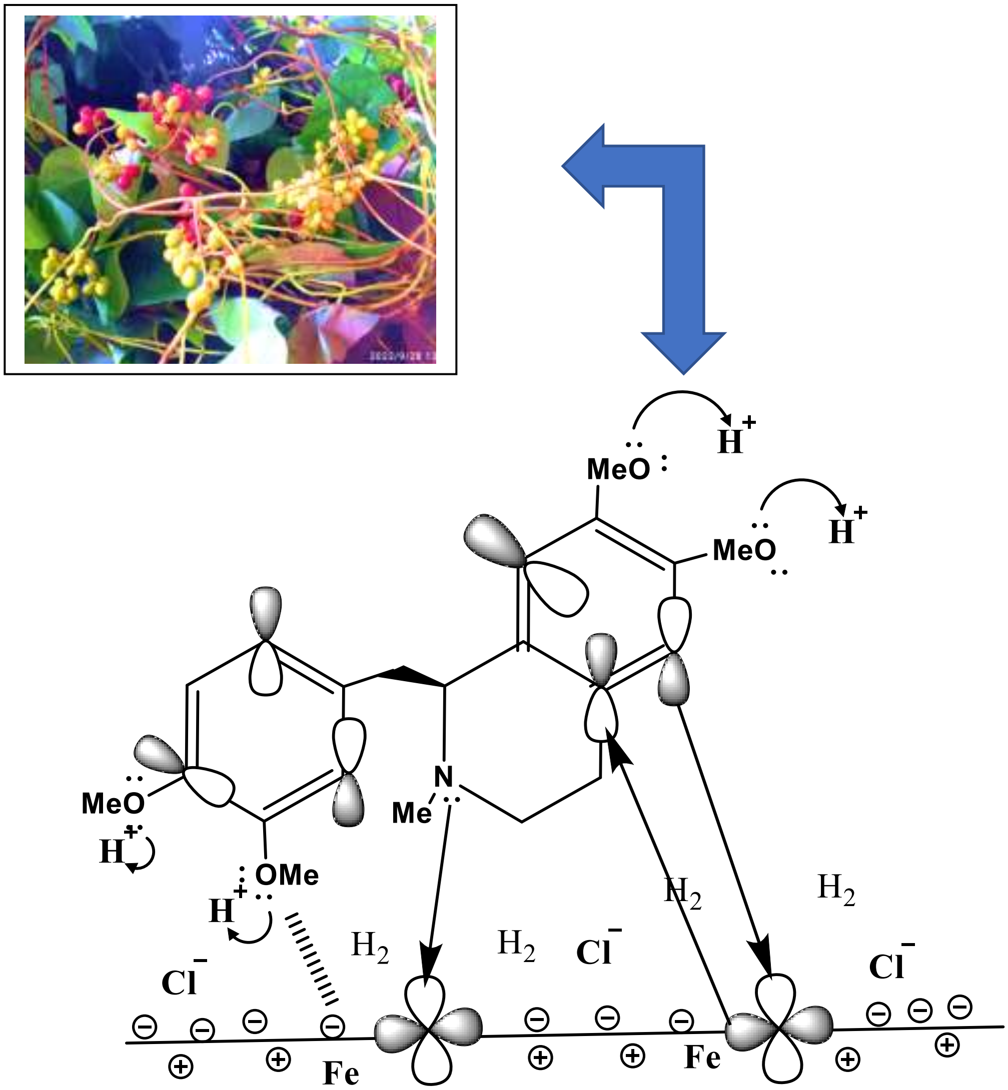


Figure 20: Mechanism of Corrosion inhibition

The Laudanosin alkaloids consist of Methoxy group and single N atom. Thus, in acidic medium, the alkaloids group can be easily protonated and adsorption of Chloride ion take place over positively charge MS surface and finally make it negatively charge surface. Thus, due to development of positive and negatively charge between alkaloids and metal surface there will be chance to interact electrostatically. The alkaloids molecules could be return to neutral state after releasing of H_2 molecules (Karki, e*t al.,* 2021). The alkaloids with nitrogen contain Non-bonding electron with act as HOMO and interact with vacant d orbital of Fe by establishing the new co-ordinate bonds that leads to the over accumulation of negative charge over MS surface. to overcome this charge, negatively charge particle i.e. electrons return to the LUMO with higher electron density i.e. π^* of alkaloids (Dhakal *et al.*,2022;Parajuli *et al*., 2022).

CHAPTER 5

CONCLUSIONS

In my dissertation work, alkaloids which act as green inhibitor had been successfully extracted from the *Cissampelos pareira* for MS dipped in 1 M HCl by using solvent extraction technique. Weight loss method and Electrochemical methods were used to study the corrosion inhibition Efficiency at various condition (concentration effect, immersed time effect and temperature effect). The result obtained from my work can be summarized in following points

- The FT-IR technique provide the proof of presence of alkaloid by showing the absorption peak near of 3198-3484 cm^{-1} further support by chemical test by showing positive result for alkaloids and negative for rest of other.
- The Inhibition Efficiency of alkaloids on MS studied by EIS and Polarization technique show highest inhibition 98.88%, and 98.53% at 100 ppm respectively which is almost 10 unit more compare to weight loss method (87.88%).
- The Inhibition Efficiency increase with increase in concentration when observed either by weight loss method or by electrochemical method but decreases with increase in temperature.
- The Inhibition Efficiency increases with increase in immersed time till 18 hours whereas after that time it moves toward downwards.
- The Langmuir adsorption isotherm support that adsorbed molecules is uni-molecules and endothermic process with mixed type adsorption in nature.

Thus, alkaloids extracted from *Cissampelos pareira* show better inhibition efficiency for MS dipped in 1 M HCl.

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