



Tetrahydrated bis(monoaqua-bis(ethylenediamine))
Copper(II)-diaqua-bis(ethylenediamine)Copper(II)dicitrate

A Dissertation report to the Department of Chemistry

Sonari College, Sonari

Affiliated to Dibrugarh University



SUBMITTED BY

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Session-2020-2023

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This is to certify that the project work entitled "Preparation and spectroscopic characterization of Tetrahydrated copper(II) citrate complex" submitted by Ankita Thapa, a student of 6th semester of the department of chemistry, Sonari College, Sonari. The project meets all the requirements of B.Sc. project

Further, it is all certified that the project has not been submitted for any purpose elsewhere.

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This is to certify that the project work entitled "Preparation and spectroscopic characterization of Tetrahydrated copper(II) citrate complex" submitted by Ankita Thapa, a student of 6th semester to the department of chemistry, Sonari College, Sonari, affiliated to Dibrugarh University is carried out under my supervision and is found suitable to submit for the partial fulfillment of the B.Sc. degree in chemistry. This work in the present form or in part, has not been submitted for any purpose elsewhere.

I wish her great success in future.

Place: Sonari College.



Signature

ACKNOWLEDGEMENT

First and foremost, I would like to express my gratitude to Dibrugarh University for including a project work in the syllabus.

I would like to express my special thanks of gratitude to my chemistry teacher, Mr. Amit Kumar Borpuzari Sir for his valuable guidance and encouragement valuable to complete my project.

I would like to extend my sincere thanks to Mr. Suchitra Narayan Rajkhowa Sir and Dr. Bikash kumar Sharma Sir who helped me in doing a lot of research for this project and I came to know about so many new things.

I would also like to thank my parents and friends who have boosted me up morally with their continuous support.

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CONTENT

CHAPTER-1

- ❖ **ABSTRACT**
- ❖ **INTRODUCTION**

CHAPTER-2

- ❖ **AIMS AND OBJECTIVES**

Chapter -3

- ❖ **EXPERIMENTAL**
- **REQUIREMENTS.**
- I. APPARATUS**
- II. CHEMICAL**
- **METHODOLOGY**
- **DIAGRAM OF CONSECUTIVE STEPS IN THE EXPERIMENT**

Chapter -4

- ❖ **RESULT AND DISCUSSION**
- **APPEARANCE OF THE CRYSTAL**
- **CRYSTAL STRUCTURE**
- **OBSERVATION OF IR**

Chapter -5

- ❖ **CONCLUSION**

Chapter -6

- ❖ **REFERENCE**

CHAPTER -1

❖ ABSTRACT

The Purpose of this project is to present the complex which is found to be interesting because it is quite rare complex among all the other complex compound as it contains three type of molecular ions, monoaqua-bis(ethylenediamine)copper(II) cation, diaqua-bis(ethylenediamine)copper(II) cation and citrate anion, interconnected via a system of hydrogen bonds including four water molecules. The discussed copper(II) complex has been prepared in two steps i.e. the powder of copper(II) citrate precursor was prepared and then dissolved in water solution of ethylendiamine. The colour of the complex is dark violet. The IR spectra was recorded and discussed.

❖ INTRODUCTION

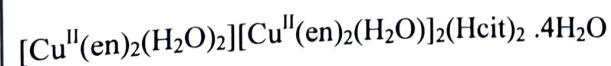
Generally, a complex compound is formed by a complex ion and a simple ion. But the discussed Copper complex is rare in itself as it can be described as a packing of three molecular ions viz. monoqua-bis(ethylenediamine)copper(II)cation, diaqua-bis (ethylenediamine) copper(II) cation and citrate anion interconnected via a system of hydrogen bonds including four water molecules per unit cell trapped in the crystal voids¹.

Ethylenediamine is one of the most prevalent ligands in coordination chemistry and was once termed "God's" gift to coordination chemist². Coordination compounds with ethylenediamine ligands and its derivatives are also used in the pharmaceutical³⁻⁵, agriculture⁶, polymer⁷, bleaching⁸ and industrial sector⁹. The tendency of ethylenediamine to coordinate with metal also make it versatile reagent for the synthesis of inorganic materials through dissolutions of the metals salts or oxides, which can bring out to precipitate in other crystalline forms¹⁰⁻¹².

Copper was one of the first metals used widely because the metal is fairly plentiful (among the 25 most abundant elements in the earth's crust) and can be found in its metallic state¹². In addition, the metal and its alloys have a number of beneficial qualities including ductility, malleability, strength, corrosion resistance, and high thermal and electrical conductivity, combined with an attractive appearance¹². Copper is also an essential trace nutrient for organisms ranging from bacteria to mammals¹². Copper-containing coordination compounds are effective antitumor agents, and are also effective as antimicrobial, antituberculosis, antimalarial, antifungal, and anti-inflammatory drugs.

Citrate anion shows different coordination mode as well as different anionic forms (cit⁴⁻, Hcit³⁻, H₂cit²⁻, etc.) depending on the type of complex. So, a non-coordinated citrate anion (isolated and connected via a system of H-bonding) in a copper(II) complex is a rare example¹².

The rarity of the complex also comes from the fact that there are no reported copper(II) complexes containing simultaneously copper(II) coordinated ethylenediamine species and the citrate anion in any possible coordination mode¹. So, here we present the synthesis and characterization (crystal structure and IR), of a new Tetrahydrated copper(II) citrate ethylenediamine complex.



CHAPTER -2

❖ AIMS AND OBJECTIVES

The main aims of the present work are given below:

1. To prepare Tetrahydrated copper(II) citrate ethylenediamine complex.
2. Spectroscopic analysis of the crystal through IR spectroscopic technique.

CHAPTER -3

❖ EXPERIMENTAL

➤ REQUIREMENTS

I. APPARATUS

- i. Conical flask
- ii. Funnel
- iii. Filter paper
- iv. Glass rod
- v. Digital Balance
- vi. Measuring cylinder
- vii. Dessicator
- viii. Beaker

II. CHEMICALS

- i. Sodium hydrogen carbonate
- ii. Citrate acid monohydrate crystal
- iii. Copper sulphate pentahydrate
- iv. Ethylenediamine
- v. Sodium hydroxide pellets

➤ METHODOLOGY

The discuss copper to complex has been prepared in two stages :-

Stage-1:- Preparation of copper citrate precursor

- i. 12.03 g of sodium hydrogen carbonate dry powder and 10.03 g of citrate acid monohydrate crystals was mixed in a beaker and then 20 ml of distilled water was added into the prepared dry mixture in four installments (until the evolutions of CO₂ gas stop).
- ii. The prepared solution was additionally filtered and evaporated on a water bath at 100°C until the crystalline hydrated trisodium citrate was formed.
- iii. The prepared crystals were dried properly.
- iv. 5.02 g of copper(II) sulphate pentahydrate was dissolved in 20 ml of distilled water and a clear blue solution formed.
- v. 3.93 g of prepared trisodium citrate crystals were dissolve in the CuSO₄ solution. During the citrate dissolution, the reaction mixture changed from clear blue to clear green and no additional precipitate was formed.
- vi. The solution mixture was left on a boiling water bath for 10-15 minutes. After that time, a dense green crystalline precipitate of copper(II) citrate was formed on the bottom and on the walls of the flask.
- vii. The crystallization was finished and the solution become partially colourless with a pale blue colour. The solution was separated and the green copper(II) citrate precursor precipitate was washed with a few portions of distilled water and then dried at 100°C.
- viii. Under drying conditions the green copper(II) citrate precursor slowly turned into a light violet form.

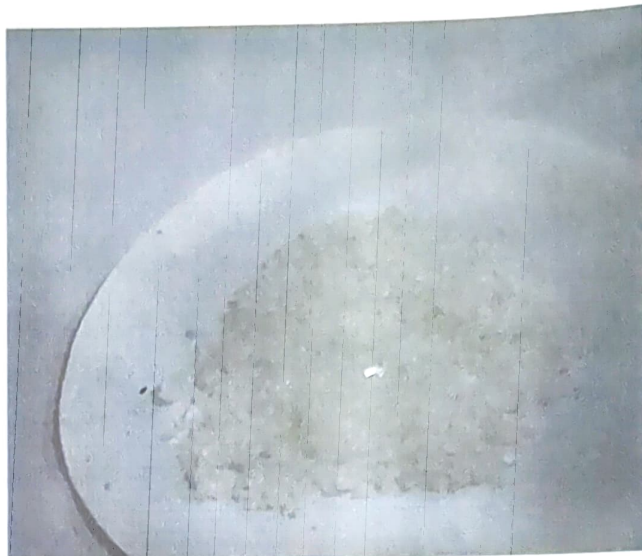
Stage-2:-Target complex

- i. 0.37g of the prepared light violet copper II citrate precursor was covered under 2 ml of distilled water.
- ii. 2 ml of ethylene diamine was added into the layer.
- iii. The colour of the mixture turned the pilot and the temperature of the mixture waste very quickly to 70-80°C.
- iv. After approximately a day in the desiccator over sodium hydroxide pellets, dark violet find crystal of the target complex were formed.

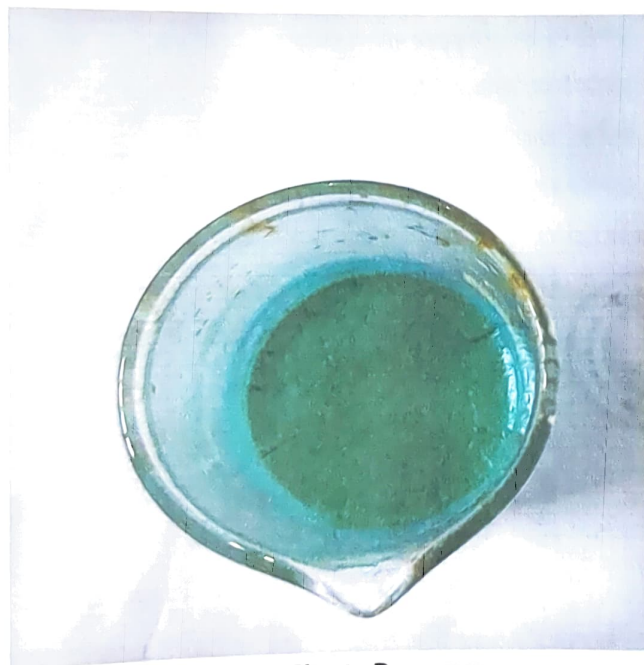
➤ **DIAGRAMS OF CONSECUTIVE STEPS IN THE EXPERIMENT**



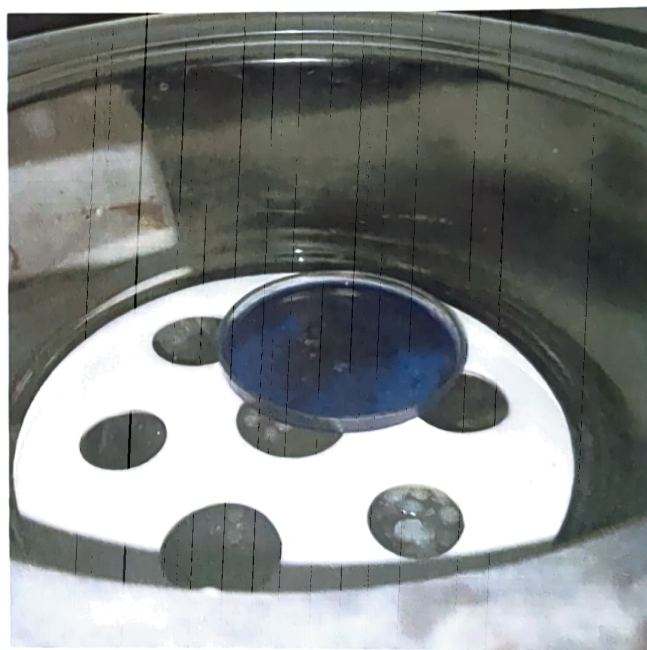
Fig(1) Evolution Of CO₂



Fig(2) Trisodium Citrate Crystals



Fig(3) Copper Citrate Precursor



Fig(4) Final Step Crystallization

CHAPTER -4

❖ RESULT AND DISCUSSION

➤ APPEARANCE OF THE CRYSTAL

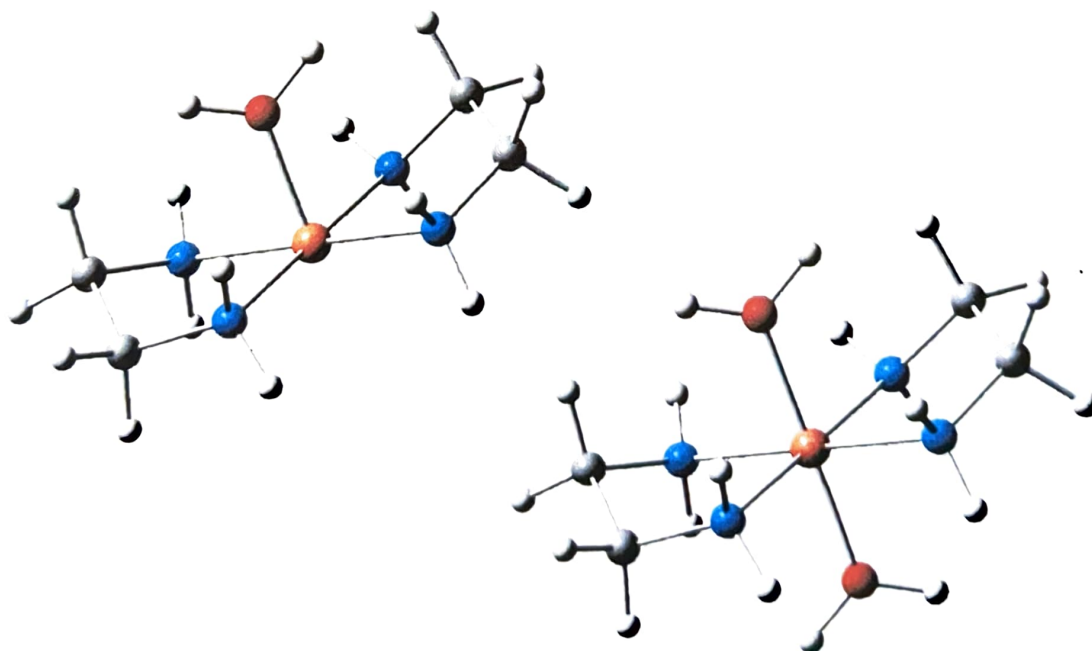
Dark violet coloured irregular crystals.

➤ CRYSTAL STRUCTURE

The crystals of tetrahydrated ethylenediamine copper(II) citrate complex can be described as a packing of three types of molecular ions, mono-aqua-bisethylenediamine copper(II) cation $[\text{Cu}^{\text{II}}(\text{en})_2(\text{H}_2\text{O})]^{2+}$ diaqua-bisethylenediamine copper(II) cation and citrate anion, interconnected through a system of hydrogen bonds including four water molecules per unit cell trapped in the crystal voids. The coordination polyhedron of the copper ion in the $[\text{Cu}^{\text{II}}(\text{en})_2(\text{H}_2\text{O})]^{2+}$ cation possesses an octahedral structure with two apical water molecules and four nitrogen atoms lying on the equatorial plane.

Inturn the copper ion in the $[\text{Cu}^{\text{II}}(\text{en})_2(\text{H}_2\text{O})]^{2+}$ possesses a square pyramidal coordination with a water molecule at the apical position.

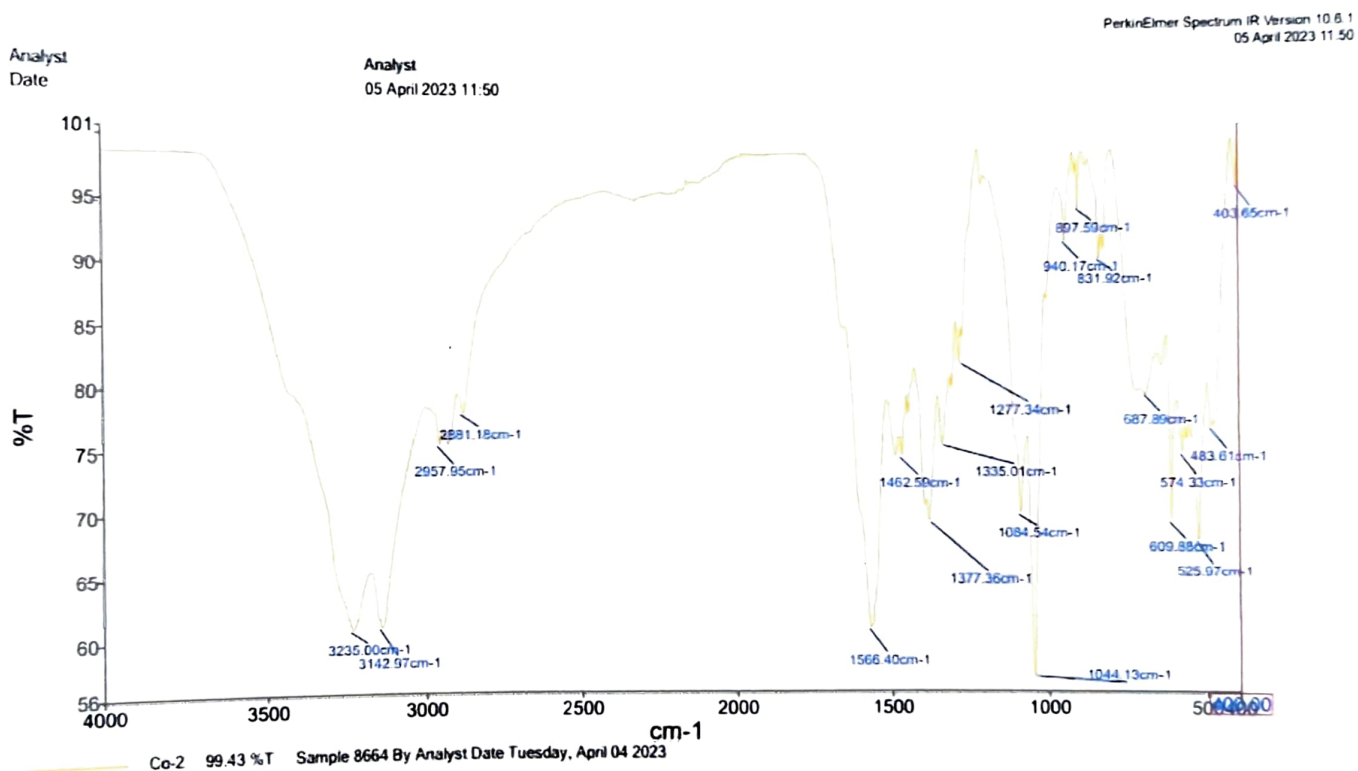
The citric anion is Hcit^{3-} adopts the lowest possible symmetry and possesses OH---O hydrogen bonding between hydrogen atom of the OH group and one of the carbonyl group. The citric anion also form a complicated network of intermolecular hydrogen bonding.



➤ OBSERVATION OF IR

The IR spectrum of the prepared complex recorded in the 400 – 4000 cm^{-1} spectral range.

The IR spectrum showed a peak at 3235.00 cm^{-1} should be because of NH group, at 2957.95 cm^{-1} for CH group, at 3142.97 cm^{-1} may be for OH group and a peak at 1566.40 cm^{-1} for the COO^{-} group.



CHAPTER -5

➤ CONCLUSION

From the literature review, it is concluded that copper complexes are found to be very useful due to the redox activity and biogenicity of copper ions which provide multiple pathways of biological activity. Copper complexes are effective antitumor agents and also effective as antimicrobial, antimalarial, antifungal etc. The synthesis and characterization of tetrahydrated ethylenediamine copper citrate complex have been performed. The characterization is done using IR spectroscopy which shows peaks for the different groups involved in the complex compound and thus confirm the identity of complex.

CHAPTER -6

➤ REFERENCE

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Green chemistry for the preparation of Silver Nanoparticles using Mint leaves extract



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This is to certify that this dissertation report entitled "*Green chemistry for the preparation of silver nanoparticles using mint leaf leaves extract*" submitted by **Bastav Das** bearing **Roll No: 10520011**, in department of Chemistry, Sonari College is found suitable for fulfillment of the requirements for the Degree in Bachelor of science in Chemistry.

This project has not been submitted to any other institution for the award of a diploma.

*Examined
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CERTIFICATE

This is to certify that the project work entitled "*Green Chemistry for the preparation of Silver Nanoparticle using Mint leaves extract*" has carried out by **Bastav Das** bearing **Roll No: 10520011** under my supervision in laboratories of the Department of Chemistry, Sonari College, is found suitable for submission to the partial fulfillment of the requirements for the Degree of Bachelor of Science in Chemistry.

This work in the present form or part has not been submitted anywhere for any other purpose elsewhere.

Supervisor

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Dept. of Chemistry

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Sign: *Bikash Kumar Sarmah*

Date:

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I am also indebted to my friends for their valuable support and encouragement which helped me to complete this project with the given time frame.

Date: 01.06.2023

Place: Sonari college, Sonari.

.....

Bastav Das

ABSTRACT

Nowadays, it has become very necessary to change traditional method for synthesizing nanoparticles, and focus on using safer and more eco-friendly approaches. In the present study, silver nanoparticles (NP) were synthesized by using *Mentha arvensis* (M.A.) (mint) aqueous plant extract. The suspension of plant extract was yellowish- green in color. After addition of AgNO_3 , within 15min, the suspension showed a change in color to dark brown after 5 hours of incubation at room temperature. The formation of *Mentha arvensis*-Ag-NPs was ascertained with UV-visible spectroscopy that gave a surface Plasmon resonance peak at 200-500nm, FTIR analysis is done for detection of various functional group. This work offers a quick, simple and non-toxic method for the synthesis of silver nanoparticles.

CONTENT

- **Introduction**
- **Procedure**
- **Characterization**
- **Result and Discussion**
- **Conclusion**
- **Reference**

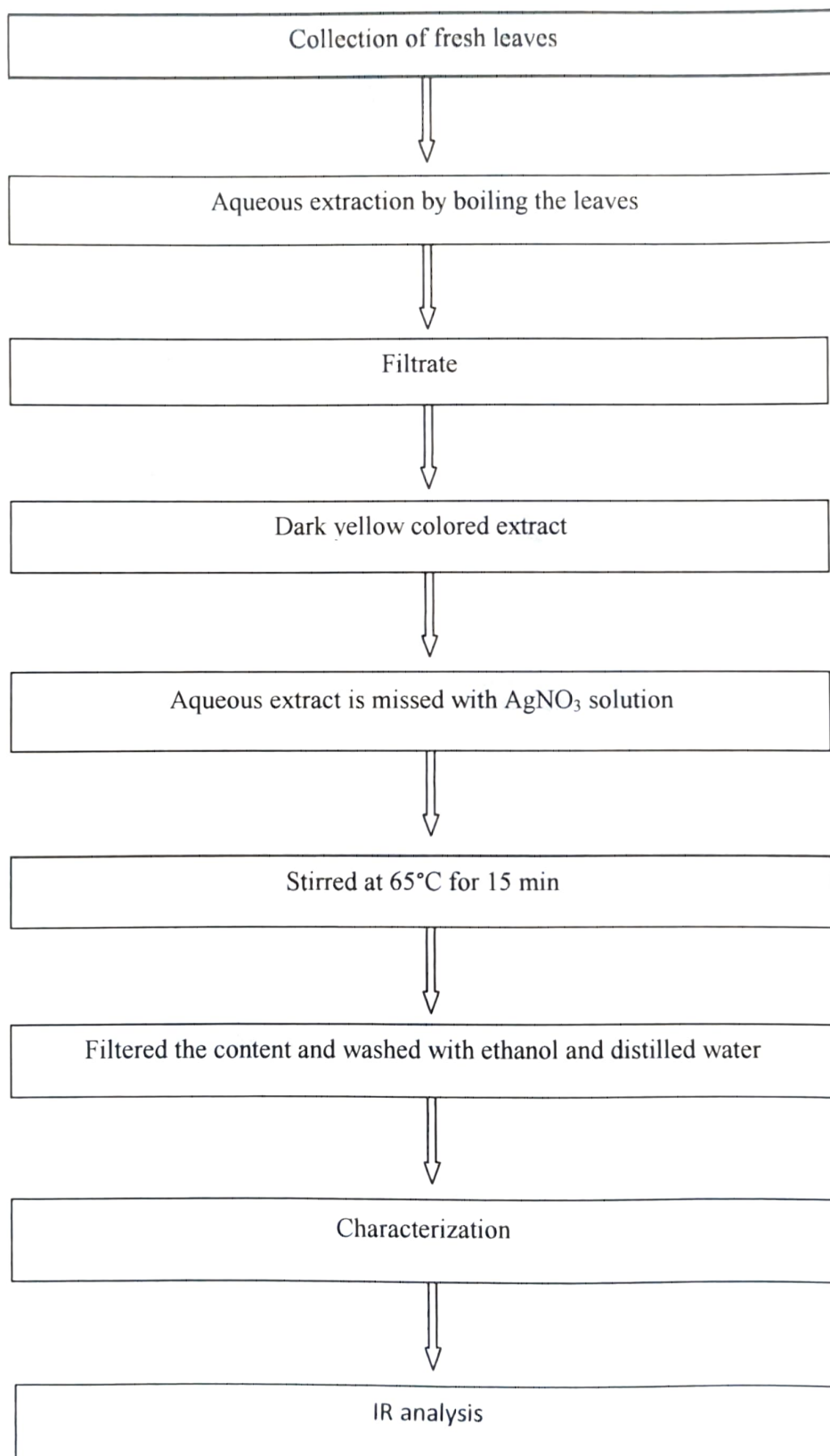
INTRODUCTION

Nanotechnology is an interdisciplinary field of research combining biology, chemistry, physics and material science, which involves working with particles which have diameter less than 100 nm.¹ Nanoparticles present widely in the nature for so many of years because of various photochemical and volcanic activity, created by plants and algae, or as the product of combustion of food energy.² Owing to the morphology, size and distribution characteristics, nanoparticles exhibit different physical and chemical properties.³ The metal nanoparticles (NPs) display extraordinary catalytic properties if compared with larger particles of the same material.⁴

In past decade the synthesis and use of silver nanoparticles (Ag-NPs) has rapidly increased because of their unique catalytic, electronic and magnetic properties.⁵ Silver is long utilized since ancient times as an important agent in biological sciences, particularly in medicine, for various purposes.⁶ It has immense potential as therapeutic agent, as it causes high lethality to bacteria while exerting minimal toxicity to animal cells.^{3,6} Studies have reported that owing to their extreme stability and unique catalytic and biological properties, silver nanoparticles, display considerable antimicrobial activities.⁷ Researchers explored the extracts of different plants for synthesis of Ag-NPs. The conventional methods of NP synthesis are well-explored because they allow control over the shape and size of the particles. However, this method has disadvantages due to high cost, energy consumption, toxicity and unsuitability for biological applications.⁸

In the context of green chemistry, an emerging field necessary for achieving the goal of sustainable development, silver nanoparticles are benign and non-toxic to human and animal cells at low concentrations and causes minimal harm to the environment when compared to other metal nanoparticles.⁹ Due to extensive list of benefits conferred by silver nanoparticles, their synthesis becomes essential, with current practice of ensuring minimum harm to the human and environment.^{8,9} So, the main aim of the study is to explore green chemistry for preparation of Ag-NPs using Mint (*Mentha arvensis*) leaf extract as reducing agent of Silver Nitrate (AgNO₃).

FLOW CHART



PROCEDURE

Requirements:

Apparatus:-

1. Beaker
2. Conical flask
3. Mortar and pestle
4. Magnetic Bead

Chemicals required:-

1. 10 gm of Mint leaf powder
2. 15 ml of 0.1M AgNO₃ solution
3. Distilled Water.
4. Ethanol

Extraction procedure:

Mint plants (*Mentha arvensis Linn.*) were collected from a local garden. Mint leaves were separated from the stem, washed with distilled water and dried in oven at 50°C for 5 hours. Dried leaves of mint are taken and crushed using motor and pastel. 10 gm of this powder are taken in a beaker and 100mL of distilled water is added. The contents were boiled with constant stirring for 15 minutes. After cooling, the mixture was filtered with Whatmann No.1 filter paper (pore size 25µm). Dark yellow-colored extract was obtained, which is used as reducing agent and stabilizer.

Now, 15mL of aqueous 0.1M AgNO₃ solution was prepared. 15mL of Mint extract was taken in a beaker and 10 ml of 0.1M AgNO₃ solution was added to it under stirring condition using a magnetic bead. Then the mixture was heated to 60-70 °C for 15min. To ensure the formation of Ag nanoparticles. A gray precipitate was collected by filtration, washed with ethanol and distilled water several times, finally dried in air at 30°C for 24 hours.

PICTURES TAKEN DURING PROJECT WORK



Figure: 1 Collected Mint leaves



Figure: 2 Crushed dried leaves

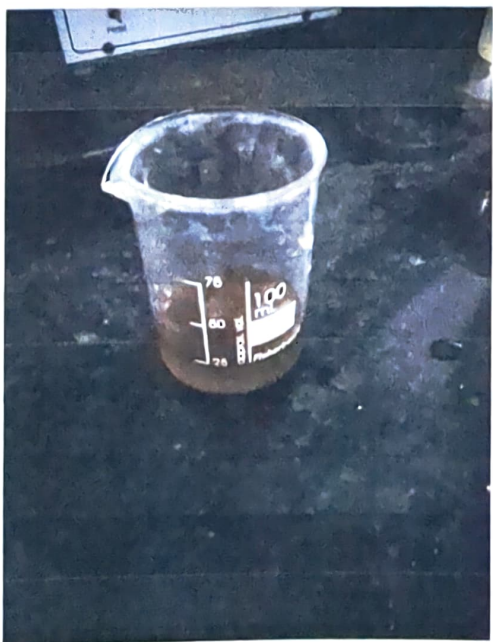


Figure: 3 Aqueous Mint leaves extract



Figure: 4 Stirred in Magnetic bead



Figure: 5 Aqueous sample AgNO₃



Figure: 6 Silver nanoparticles

CHARACTERIZATION

Fourier-transform infrared spectroscopy (Perkin Elmer Spectrum 10.6.1) was adopted for detection of reducing agents from the plant extract and also for determining the size distribution, identification of chemical bonds and functional groups of Ag-NPs.

The IR analysis spectrum of Ag-NPs was recorded in the range of 3000-700 cm^{-1} .

Spectral analysis:

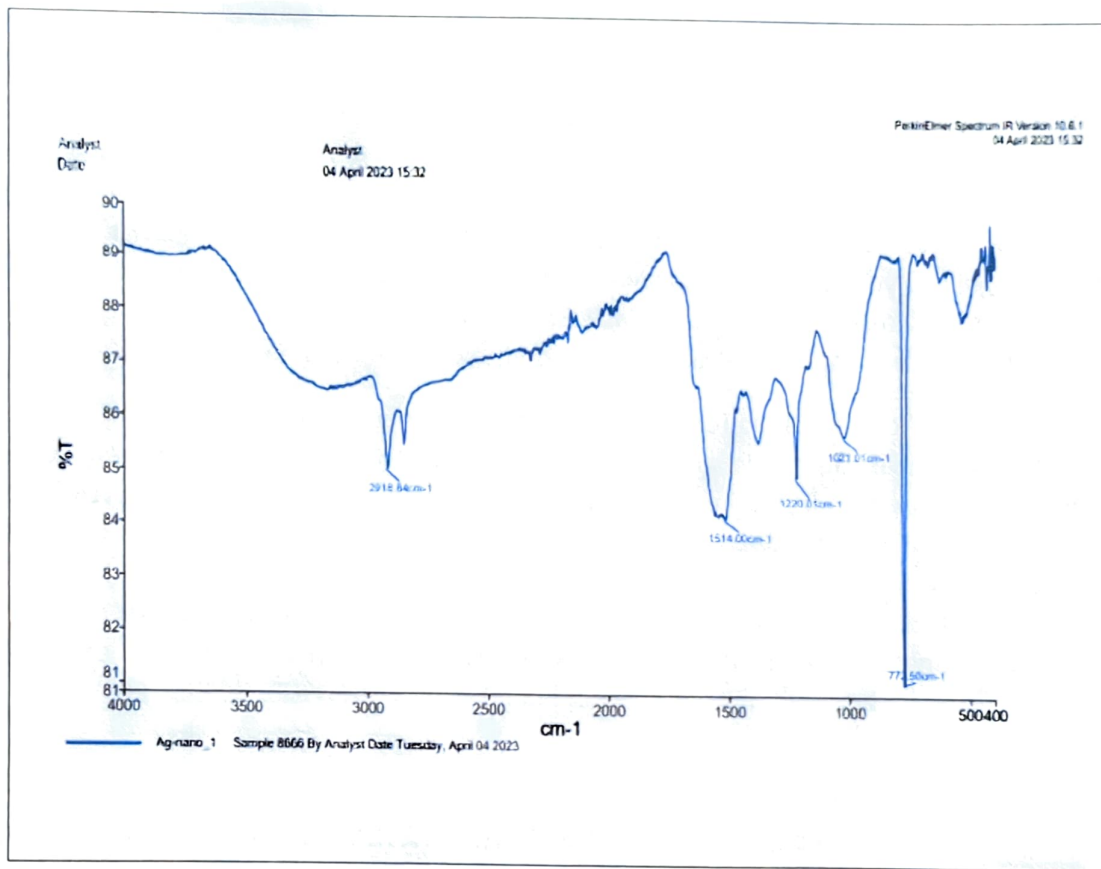


Figure: 7 FTIR spectra of silver nanoparticles

RESULT AND DISCUSSION

In this study, silver nanoparticles are synthesized using an aqueous extract of *Mentha arvensis*. The obtained results showed that the aqueous extract of this plant is a greener alternative for synthesis of Silver nanoparticles. The use of this plant extracts has proved to be a good alternative for synthesis of silver or gold nanoparticles due to the reducing and stabilizing capabilities of the secondary metabolites that can be extracted by boiling in water any kind of plant tissue, from roots to flowers. To date, there are more than 50 plant species and cultivars that have been used for the synthesis of metallic NP, mainly of silver and gold.

The FTIR spectrum analysis revealed that the chemical complexity of Ag-NPs owing to the possession of various functional groups. The IR spectrum of silver nanoparticles showed peaks at 772.50 cm^{-1} to 2918.84 cm^{-1} . The IR spectrum shows absorption bands 2918.84 , 1514 , 1220 , 1021 , 772.50 cm^{-1} . The broad and very intense bands 2918.84 cm^{-1} could be attributed in the presence of C-H stretching. The peak at 1514 cm^{-1} is related to the presence of C=O stretching carbonyl compound, which may be involved in the bio reduction of silver ions.⁹ The peaks at 1220 cm^{-1} arises from C-N stretching mode of aromatic groups.¹⁰ The peak at 1021 cm^{-1} is also related to C-O bond from proteins in the plant extract.⁹

Synthesis of silver nanoparticles: Conventional and green methods:

Nanoparticles are synthesized by three fundamental method, these include chemical, physical and biological process.¹¹ In chemical methods are most common yet have few benefits, such as used of water or organic solvent for synthesis of nanoparticles. Chemical synthesis required reducing agent such as Citrate, ascorbate, borohydride and hydrogen gas and stabilizing agent includes polymers with specific functional group polyethylene glycol, polyvinyl pyrrolidone.¹² The toxicity of this solvent has long lasting effect in human health. To achieve sustainable development goal, large scale of silver nanoparticles synthesis by chemical method is not a practical option. In Physical methods show promise in terms of greenness, in this method evaporation-condensation is done with the help of tube furnace at room temperature and no chemical reagent is used or solvent-less in this process makes it reasonably environmental friendly. Despite giving sizeable yield of nanoparticles, this method has a range of disadvantages such as high temperature is required and consumption of large amount of energy.¹³ A suitable green alternative to chemical and physical methods of synthesis are the biological methods. Biological methods seemed to uphold the principle of green chemistry almost absolutely. Living organism, such as plants, algae, microbes and fungi, even animal play vital rule in nanoparticles synthesis because of some constituent bio-molecules, such as enzymes, that can be extracted from them and help in the reduction of Ag^+ silver ions to form nanoparticles.¹⁴

Bio-molecules from plant extracts are commonly employed for production of silver nanoparticles it is not more economical and can be easily scaled for industrial purposes. An aqueous plant extract is prepared from leaves and aqueous silver nitrate solution is added reduction occurs with the help of bio molecules that present in the plant extract. Thus the overall process resembles the chemical methods closely, the reagent are non-toxic and derived from plant. The reaction is highly efficient and quick. So, this is a green method for synthesis of silver nanoparticles.¹⁵

CONCLUSION

Green synthesis of silver NPs were synthesized by *Mentha arvensis* plant leaves extract at room temperature was reported in this work. Silver NPs were successfully biosynthesized by this simple, fast and cost effective, eco-friendly, and efficient method, which excludes external stabilizers or reducing agent. In addition they also act as stabilizing agents of NPs. The properties of the final silver nanoparticles including average diameter, dispersion and optical properties can be easily controlled by changing the main parameter in this process, such as silver nitrate concentration. Thus, the green synthesis of silver nanoparticles from Mint plant extract can be used as curative agent for targeted drug delivery cure diseases. This is due to mode of action of silver ions against bacteria. This silver ion, and bind with DNA bases destroy cell membrane, condense its ability to replication.

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Analysys of Physico- Chemical Parameter of water of Sonari Pukhuri, Rajapukhuri and Borpukhuri of Sonari

A Dissertation report to the Department of Chemistry

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CERTIFICATE

This is to certify that the project work entitled "To analysis the physico-chemical parameter of three ancient ponds of Sonari" submitted by Nabajyoti Paul, a student of 6th sem of the Department of Chemistry, Sonari affiliated to Dibrugarh university is carried out under my supervision and is found suitable to submitted the partial fulfillment of the B.Sc. Chemistry. This work in the present form in part, has not been submitted for any purpose elsewhere.

I wish her great success in future.

Place: Sonari College

S. N. Rajkhowa
Signature of Guide

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Date: 02/05/23

Associate Professor, Dept. of Chemistry(HOD)

CERTIFICATE

This is to certify that the dissertation report entitled, '*Analysis of Physico-Chemical Parameter of water of Sonari Pukhuri, Rajapukhuri and Borpukhuri of Sonari*' submitted to the Department of Chemistry, Sonari College is a record of project work carried out by **Nabajyoti Paul**. The project meets all the requirements of B.Sc. project. Further, it is also certified that the project has not been submitted for any purposes elsewhere.

Examined
Project 01/6/23
Entered & examined

Place: Sonari College

(Amrit Kumar Borpuzari)

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Date: 1.06.2023

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CONTENTS

Certificate	i
Acknowledgement.....	ii
Abstract	iii

CHAPTER 1

1. INTRODUCTION	01.
1.1 STRUCTURE OF WATER	
1.2 SOURCE OF WATER	
1.3 STATE OF WATER	
1.4 WATER CYCLE	
1.5 FORMATION OF WATER	
1.6 CHARACTERISTICS OF WATER	
1.7 IMPORTANCE OF WATER.....	
1.8 USES OF WATER	
1.9 CAUSES OF WATER POLLUTION.....	
1.10 EFFECT OF WATER POLLUTION	
1.11 WATER POLLUTION CONTROL	
2. AIM AND OBJECT	
WHY WE SELECT THE PROJECT?	

CHAPTER 2

METHODOLOGY	
2.1 STUDY AREA	
2.2 SAMPLE COLLECTION	
2.3 MATERIALS	
2.4 PROCEDURE	
2.4.1 DETERMINATION OF TEMPERATURE	
2.4.2 DETERMINATION OF PH	
2.4.3 TEST FOR CHLORINE IN WATER SAMPLE	
2.4.4 TEST FOR ARSENIC IN WATER SAMPLE	
2.4.5 TEST FOR HARDNESS IN WATER SAMPLE	
2.4.6 TEST FOR DISSOLVED OXYGEN IN WATER SAMPLE	
2.4.7 ESTIMANATION OF TDS	
2.5 PICTURE COLLECTED DURNING PROJECT	
2.6 CALCULATIONS.....	

CHAPTER 3

3.1 RESULT.....	
3.2 DISCUSSION.....	

CHAPTER 4

CONCLUSION.....	
-----------------	--

REFERENCE.....	
----------------	--

Abstract :-

The aim of this project is to evaluate the quality of water of three ancient ponds of Sonari, Assam located in North-East India. The Physico Chemical parameter of Sonari pukhuri Borpukhuri and Raja Pukhuri were studied. The physico- chemical parameter such as source temperature, P^H , dissolve oxygen, chlorine, arsenic, hardness, total dissolve solid were determined using standard method. The result shows that the water quality variations are mostly affected by dissolved minerals salts along with antropogenic activities in the area contiguous to the ponds.

Chapter 1 :-

To analysis of Physico – Chemical parameter of Sonari pukhuri, Bor pukhuri, and Raja Pukhuri, Sonari, Charaideo, Assam

Introduction:-

Water is the major constituent of all living being. Water is necessary to sustain all types of lives.

1.1 Structure of Water

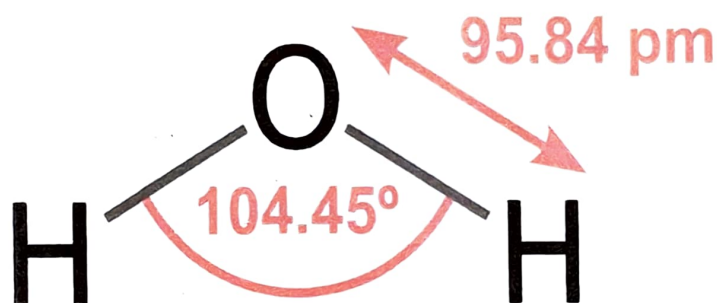


Fig 1. structure of water

A water molecule consists of two hydrogen atom bonded to an oxygen atom, and its overall structure is V-shaped. This is because of sp^3 hybridization of oxygen atom, in forming bond with H-atom, also carries two pair of unshared electron. All of the electron pairs- shared and unshared repel each other.

The bond distance between oxygen and hydrogen atom is approximately 95.84 pm. The angle between two hydrogen atom is $104.45^{[1]}$

1.2 Source of Water -

The flowsheet diagram of sources of water is given below²

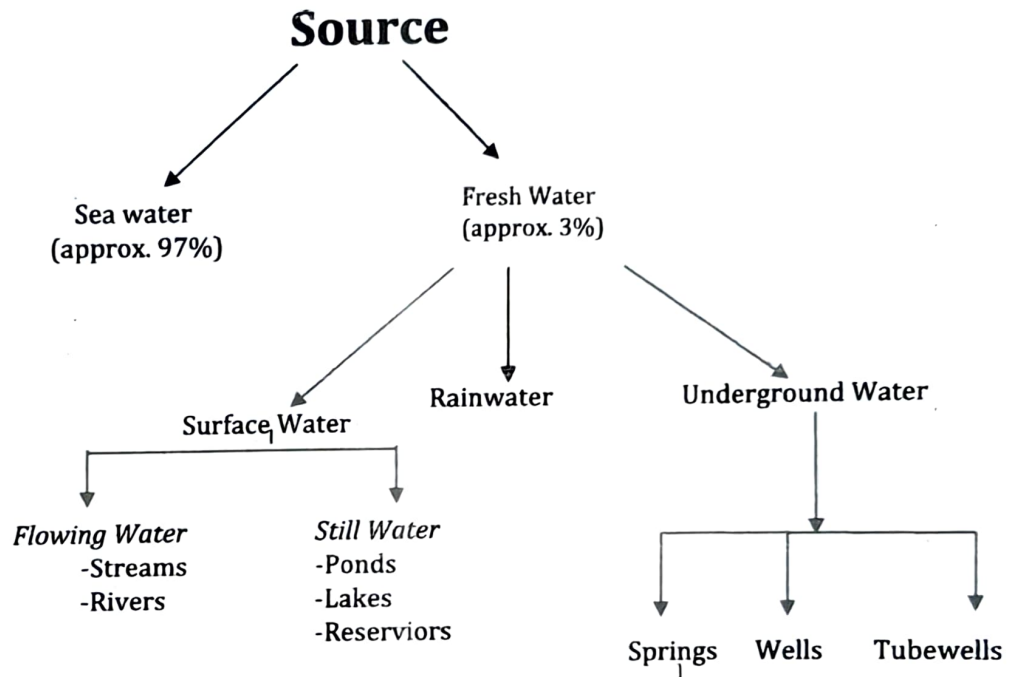


Fig. : Diagram of Source of Water

1.3 State of Water-

- Solid
- Liquid
- Gas

1.4 Water Cycle [3]

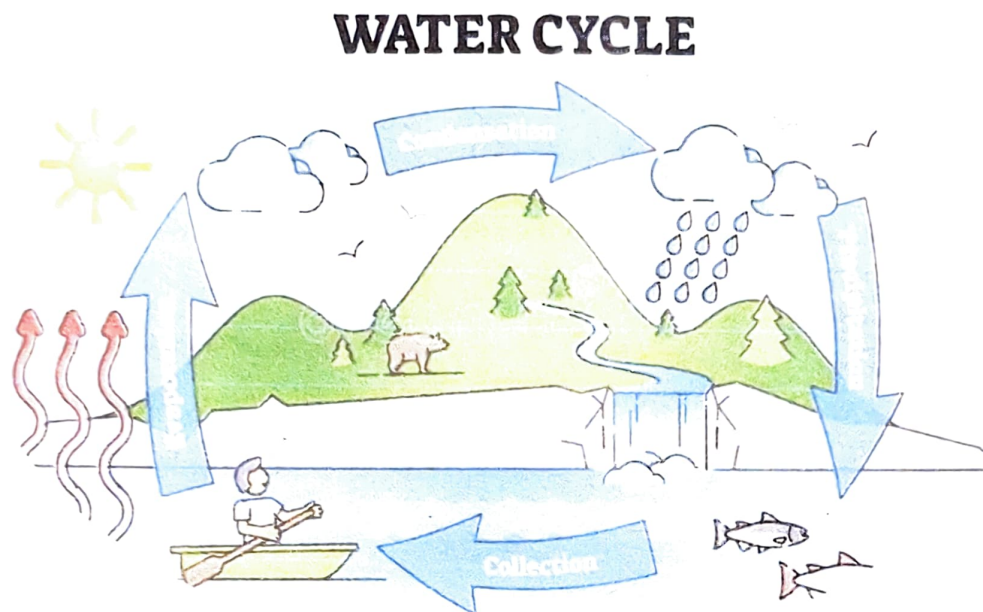
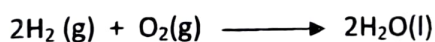


Fig 2. Water Cycle

1.5 Formation of Water :-

Formation of water from hydrogen and oxygen is a combination reaction because hydrogen and oxygen are combining to form single product.



The continuous influx of people and rapid development that is fast taking place in this city has called for concern and one cannot be ponder on what the implication could be on the pressure of aquatic environment.^[4]

The widespread problem of water pollution is jeopardizing our health. Unsafe water kills more. People each year than war and all other forms of violence combined. Meanwhile, our drinkable water sources are finite 1% of the earth's freshwater is actually accessible to us.

1.6 Characteristics of water Water (H_2O) is a polar inorganic compound that is at room temperature, a tasteless and odourless liquid which is nearly colourless apart from an inherent hint of blue. It is described as "Universal Solvent" or "Solvent of life"

1.7 Importance of Water

Without water, there would be no life on earth as it is an essential fluid to every human, plant, and living being on this planet.

Most of the water here is saline water, and it occupies three-fourths of the earth's surface. Freshwater supply is minimal, so we need to stress water conservation.

Fishing in oceans and freshwater provides a source of food and livelihood to man by worldwide.

We have to avoid the wastage and contamination of freshwater and spread awareness on the same.

Water is a colourless inorganic liquid and it is also non-renewable source.

1.8 Uses of Water:-

The water is used for drinking purpose by human. Water is natural wonder and is the most common important useful thing for surviving of all the living thing. Without food, living beings can survive for some day but without water nobody can survive. 70% of our body contains water, Which regulates life processes such as digestion of food, transportation of nutrients and excretion of body wastes. It regulates the body temperature by the process of sweating and evaporation

1.9 Causes of water pollution

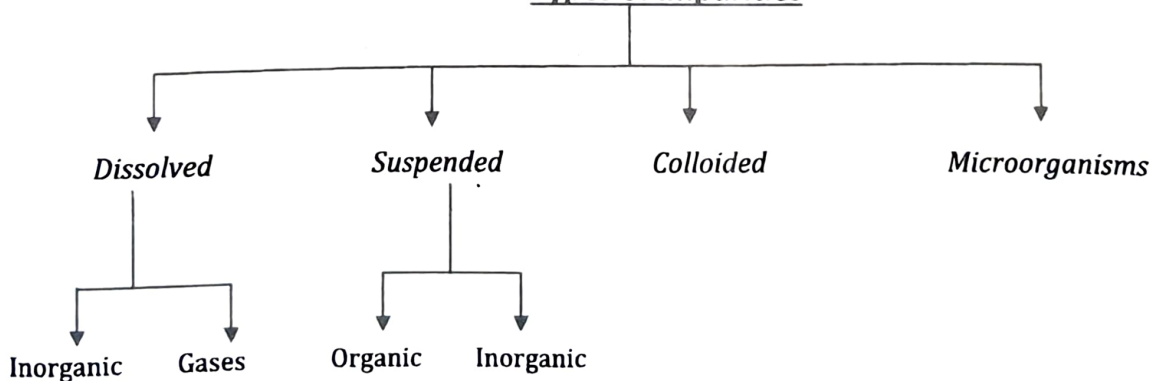
- Global warming
- Deforestation
- Industrial, agriculture and livestock farming
- Rubbish and faecal water dumping
- Maritime traffic
- Fuel spillage

Sewage and waste water-

Sewage, garbage and liquid waste of household, agriculture land and factories are discharge into lake and rivers these wastes contain harmful chemical and toxins which make the water poisonous for aquatic animals and plants.

Dumping- Dumping of solid wastes and litters in water bodies cause huge problems.

Types of impurities



Human settlement, industrial development, agriculture, deforestation, that is taken place around the city may have significantly causes great impact on the physical and ecological feature of the pond. It is known that variation in physico-chemical factor has a profound effect on the distribution and population density of both fauna and flora^{5,6,7}. Water contamination due to pharmaceutical industrial wastes may harm aquatic organisms (phy to

Planktons and Zooplankton). It has been shown that pesticides causes damage to marine algae and cyanobacteria., harmfully fish and phytotoxicity. It has been reported that wastewater contains considerable level of heavy metals^{8,9,10}.

Invertebrates release nutrients into the water through drilling, feeding activity and secretions bacteria, fungi, algae and aquatic plants feed on these nutrients and help to accelerates their growth.

Hard water does not give much lather with soap, as most of the soap is consumed for removing calcium and magnesium salts present in water.

1.10 Effect of water pollution –

The effect of water pollution are varied and depended on what chemicals are dumped and in which locations.

a. Death of aquatic (water) animals:-

The main problem caused by water pollution is that it kills organisms that depend on these water bodies. Dead fish, crabs, birds and sea gulls, dolphin and many other animal often wind up on beaches, killed by pollutants in their habitat.

b. Disruption of food chain:-

Water pollution may lead to reduction in the number of primary consumers which lead to the reduction in the number of secondary and tertiary consumer. Water pollution lead to the poisoning of animals at all tropic levels. Pollutants such as lead or cadmium are eaten by tiny animals, there animals are consumed by fishes, shellfish, and the food chain continues to be disruption at all higher level³.

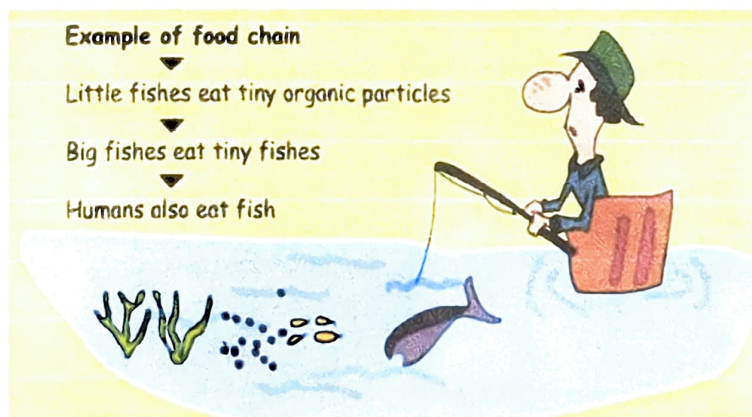


Fig 4 : aquatic food chain

c. Diseases :-

Humans and other living things are eventually effect by water pollution. Diseases such as cholera, diarrhea, dysentery, hepatitis A, typhoid and polio are occured due to pollute water.

d. Disruption of food chain-

Water pollution lead to many problems such as the degradation of aquatic ecosystem or spreading water borne diseases. Water pollution causes land pollution and render the soil pollution.^[11]

1.11 Water pollution control-

The aim of water pollution control is to protect the water from any potential harm caused by discharges of pollutants. Rather than releasing sewage waste into water bodies, it is better to treat them before discharge. A very special plant, the water hyacinth can absorb dissolved toxic chemicals such as cadmium and other such elements. Establishing these in region prone to such kind of pollutants will reduce the adverse effect to a large extent. Some chemicals method that help in the control of water pollution are precipitation, the ion exchange method.

The pesticides used in the tea garden nearer to Sonari - pukhuri should be good in quality, which pollutes pond water very little that does not bring any bad impact on to aquatic animal and human health. The construction should be done very carefully nearer to the pond a good quality of fuel should be used on vehicle which impact less toxic substance, human will reduce activity in the pond.

(II) Aims & Object

Why we select the project:-

Water sampling purpose :-

The primary goal of water sampling is to observe and measure how water is normal to drink within a specific ecological region. We have selected nearby three historical ponds situated at Sonari respectively Sonari pukhuri, Borpukhuri and Rajapukhuri.

We have found good opportunity within our local area to collect the sample with very low expenses. So, we decide to complete our project within nearest Ponds.

Chapter 2 -

(I) Materials and methodology:-

2.1 Study area-

Sonari is a district headquarter, mid - size town and municipal board in newly announced Charaideo district in the Indian State of Assam. The site of Charaideo was the capital of the Ahom kingdom established by the first Ahom king Chao Lung Siu-Ka-Pha in 1253. The total population of Sonari is 19,810 out of which 10,381 are males and 9,429 are females.

The three biggest pond of Charaideo are

1. Rajapukhuri
2. Borpukhuri
3. Sonari pukhuri

The Borpukhuri is located in middle of the Sonari town and along the road side. The water supply on middle town homes is from this Bor pukhuri. There is also a lot of water animals such as fishes, tortoise living in this ponds.

The two other ponds (Sonari pukhuri and Raja pukhuri) are located far away from main town and it is also along the road sides. The two ponds also contain a lot of water animals like many different quality of fishes, tortoise etc. The water of all these is supplied into many houses near by the ponds through pipes.

Sonari - pukhuri is surrounded by Sonari tea garden. The two other ponds are surrounded by rural-area. Rajapukhuri pukhuri is located 3Km far away from Sonari town. Sonari pukhuri pond is located 2 km from Sonari town.

2.2 Sample Collection -

The water samples were collected from the three ponds on 3rd march 2023. The water samples were collected in a plastic water bottle.

2.3 Materials

The requirement for the experimental study of the sample water are as follows -

- a. Sample water : The water is collected from three different historical ponds- : Sonaripukhuri, Borpukhuri and Raja Pukhuri.
- b. Mercury Thermometer
- c. pH meter
- d. AgNO₃ solution
- e. (KI+ I₂) solution
- f. EDTA solution
- g. Buffer solution
- h. Eriochrome black T

- i. Saturated solution of NaHCO_3
- j. Starch indicator
- k. Phenolphthalein indicator

2.4 Procedure:-

2.4.1. Temperature

The water temperature was measured with mercury in glass thermometer. The temperature obtained is different for different ponds.

2.4.2 pH. Level - The water pH was measured with pH meter electrode of pH meter.

2.4.3 Chlorine

1. 20 drops of collected water were taken in a dry test tube with with a burette.
2. Adjusting the PH of the solution 7-10 by adding one drop of NaOH solution.
3. 2 drops of potassiumchromate indicator were added to the above solution.
4. After this, Ag NO_3 solution were added dropwise, stirred the solution till the colour turn permanent reddish.
5. The number of drops of Ag NO_3 solution were counted-

Chemical reaction -



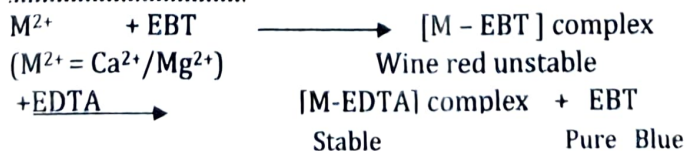
2.4.4 Arsenic

1. 20 drops of saturated solution of sodium bicarbonate was prepared with the sample.
2. 2.5 ml of above solution were taken in a test tube.
3. 1 drop of starch indicator was added and stirred.
4. $\text{KI} + \text{I}_2$ solution is added dropwise no blue colour of the solution appear, which indicator, the level of arsenic is very very low in the water sample.

2.4.5 Hardness -

1. 20 drops of water sample were taken in a dry test tube.
2. 2 drops of buffer solution were added.
3. Small amount of eriochromic black T (EBT) indicator was added.
4. EDTA is added dropwise till the colour changes to blue.
5. The drops of EDTA were counted.

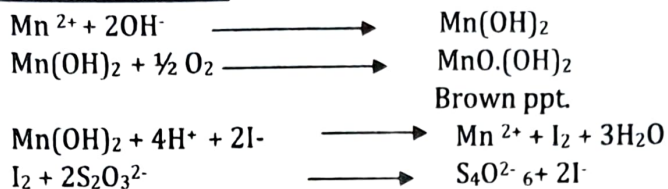
Chemical reaction



2.4.6. Dissolved Oxygen -

1. 20 drops of water sample were taken in a dry test tube.
2. 3 drops of $MnSO_4$ and 2 drops of alkali iodine solution were added to it.
3. 2 drops of dil. H_2SO_4 were added till the ppt dissolved.
4. One drop of starch indicator was added to the above solution, a blue colour appeared.
5. To the above solution $Na_2S_2O_7 \cdot 2H_2O$ were added dropwise till the clear solution were obtained.
6. The number of $Na_2S_2O_7$ were counted.

Chemical reaction.



2.4.7. Total Dissolved solid (TDS)

1. Weight the 25 ml of beaker.
2. Pour 25 ml of sample water into the beaker and weight them.
3. Drying the water, till the total water was evaporated.
4. Cooled the beaker and again weight the beaker after drying.

2.5 Picture of Consecutive steps in the experiment



a.Sonari pukhuri

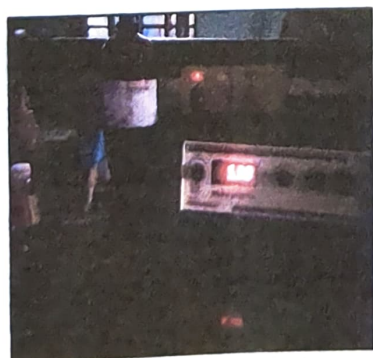


b.Borpukhuri

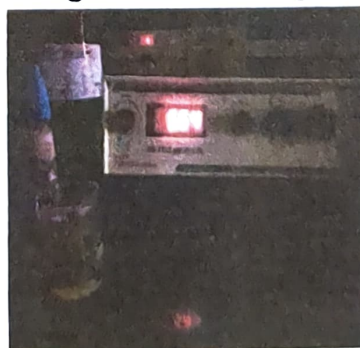


c. Rajapukhuri

Fig.4 Source of sample



a.Sonari pukhuri



b.Rajapukhuri



c.Borpukhuri

Fig.5 pH of Sample



a.



b.

Fig.6 Test for chlorine



a



b

Fig.7 Test for Hardness

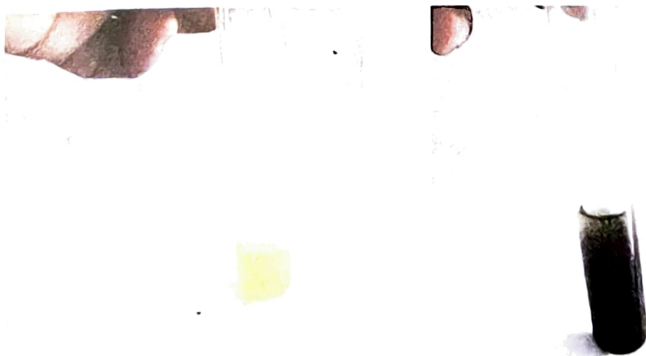


Fig.8 Test for Dissolved Oxygen

2.6 Calculation --

2.6.1 The temperature of Sonari Pukhuri is the highest among all the three i.e 25 ° C . The temperature of Rajapukhuri is the lowest among all the three i.e 22 ° C . The temperature of Borpukhuri is 24 ° C .

2.6.2 The pH of Rajapukhuri is highest i.e 5.91. The pH of Sonari pukhuri is low i.e, 5.30 and pH of Borpukhuri is 5.64.

2.6.3 Test for Chlorine

For Sonari pukhuri :-

$$\begin{aligned}\text{Volume of AgNO}_3 \text{ solution, } V_1 &= \text{nos. of drops} \times \text{Drops value of Pipette} \\ &= 60 \times 0.05 \text{ ml} \\ &= 3 \text{ ml} \\ \text{Normality of AgNO}_3, N_1 &= 0.0001 \text{ N} \\ \text{Volume of water used, } V_2 &= 1 \text{ ml} \\ \text{Amount of chlorine in 1 L} &= \frac{V_1 N_1 \times 35.5 \times 1000}{1} \\ &= 106.5 \text{ mg/L}\end{aligned}$$

For Borpukhuri -

$$\begin{aligned}\text{Volume of AgNO}_3 \text{ solution, } V_3 &= \text{nos. of drops} \times \text{Drops value of Pipette} \\ &= 65 \times 0.05 \text{ ml} \\ &= 3.25 \text{ ml} \\ \text{Volume of water, } V_4 &= 1 \text{ ml} \\ \text{Amount of chlorine in 1 L} &= \frac{V_3 N_1 \times 35.5 \times 1000}{1} \\ &= 113.6 \text{ mg/L}\end{aligned}$$

For Rajapukhuri -

$$\begin{aligned}\text{Volume of AgNO}_3 \text{ solution, } V_5 &= \text{nos. of drops} \times \text{Drops value of Pipette} \\ &= 70 \times 0.05 \text{ ml} \\ &= 3.5 \text{ ml} \\ \text{Volume of water, } V_6 &= 1 \text{ ml} \\ \text{Amount of chlorine in 1 L} &= \frac{V_5 N_1 \times 35.5 \times 1000}{1} \\ &= \frac{3.5 \times 0.001 \times 35.5 \times 1000}{1} \\ &= 124 \text{ mg/L}\end{aligned}$$

2.6.4. Test for Arsenic -

The level of arsenic is very very low in these three ponds. So, the trace amount of arsenic is not determined in our experiment.

2.6.5 Test for hardness -

For Sonari pukhuri-

$$\begin{aligned}\text{Volume of EDTA, } V_1 &= \text{nos. of drop} \times \text{drop value of pipette} \\ &= 6 \times 0.05 \\ &= 0.3 \text{ ml}\end{aligned}$$

$$\text{Molarity of EDTA} = N_1 = 0.001 \text{ M}$$

$$\begin{aligned}\text{Volume of water, } V_2 &= \text{nos. of drop} \times \text{drop value of pipette} \\ &= 20 \times 0.05 \\ &= 1 \text{ ml}\end{aligned}$$

$$\begin{aligned}\text{Hardness of water (mg/L)} &= V_1 M_1 10^5 / V_2 \\ &= 0.3 \times 0.001 \times 10^5 \\ &= 30 \text{ mg/L}\end{aligned}$$

For Borpukhuri

$$\begin{aligned}\text{Volume of EDTA, } V_3 &= 7 \times 0.05 \\ &= 0.35 \text{ ml}\end{aligned}$$

$$\text{Volume of water, } V_4 = 1 \text{ ml}$$

$$\begin{aligned}\text{Amount of hardness (mg/L)} &= V_3 N_1 \times 10^5 / V_4 \\ &= 35 \text{ mg/L}\end{aligned}$$

For Rajapukhuri

$$\begin{aligned}\text{Volume of EDTA, } V_5 &= 6 \times 0.05 \\ &= 0.30 \text{ ml}\end{aligned}$$

$$\text{Volume of Water, } V_6 = 1 \text{ ml}$$

$$\begin{aligned}\text{Amount of hardness (mg/L)} &= V_5 N_1 \times 10^5 / V_6 \\ &= 30 \text{ mg/L}\end{aligned}$$

2.6.6 Determination of Dissolved Oxygen

For Sonari Pukhuri

$$\text{Volume of Na}_2\text{S}_2\text{O}_3, V_1 = 8 \times 0.05 = 0.4 \text{ ml}$$

$$\text{Volume of water } V_2 = 1 \text{ ml}$$

$$\begin{aligned}\text{Amount of dissolved oxygen (mg/L)} &= 2 \times V_1 / V_2 \\ &= 2 \times \frac{0.4}{1} \\ &= 0.8 \text{ mg/L}\end{aligned}$$

For Borphukhuri :-

$$\text{Volume of Na}_2\text{S}_2\text{O}_3, V_3 = 7 \times 0.05 = 0.35$$

$$\text{Volume of water, } V_4 = 1 \text{ ml}$$

$$\text{Amount of Dissolved Oxygen} = 2 \times \frac{V_3}{V_4} = 2 \times \frac{0.35}{1} = 0.70 \text{ mg/L}$$

For Rajapukhuri -

$$\text{Volume of Na}_2\text{S}_2\text{O}_3, V_5 = 9 \times 0.05 = 0.45 \text{ ml}$$

$$\text{Volume of water, } V_6 = 1 \text{ ml}$$

$$\begin{aligned}\text{Amount of dissolved Oxygen} &= 2 \times V_5 / V_6 \\ &= 2 \times 0.45 / 1 \\ &= 0.90 \text{ mg/L}\end{aligned}$$

2.6.7 Determination of TDS :-

For Sonari Pukhuri -

Weight the empty glass beaker, $W_1 = 18.264$ g

Beaker with water sample, $W_2 = 18.266$ g

$$\begin{aligned}\text{Total dissolved solid (ppm)} &= (W_2 - W_1) \times 2 \times 10^4 \\ &= 40 \text{ ppm.}\end{aligned}$$

For Borpukhuri -

Weight the empty glass beaker, $W_3 = 18.289$ g

Weight of Beaker with water sample, $W_4 = 18.292$ g

$$\begin{aligned}\text{Total dissolved solid (TDS)} &= (W_4 - W_3) \times 2 \times 10^4 \\ &= (18.266 - 18.289) \times 2 \times 10^4 \\ &= 60 \text{ ppm.}\end{aligned}$$

For Rajapukhuri -

Weight of empty glass beaker, $W_5 = 17.819$ g

Beaker with water sample, $W_6 = 17.822$ g

$$\begin{aligned}\text{TDS} &= (W_6 - W_5) \times 2 \times 10^4 \\ &= (17.822 - 17.819) \times 2 \times 10^4 \\ &= 60 \text{ ppm.}\end{aligned}$$

Chapter 3:

3.1 Result:

Table 1: The Physico-Chemical study of three different water station.

Sl no.	PARAMETER	SONARI PUKHURI	BORPUKHURI	RAJAPUKHURI
1.	Temperature	25 °C	24 °C	22 °C
2.	pH	5.30	5.64	5.91
3.	Dissolved Chlorine	106 mg/L	113.6 mg/L	124 mg/L
4.	Arsenic	Not traced	Not traced	Not traced
5.	Hardness	30 mg/L	35 mg/L	30 mg/L
6.	Dissolved O ₂	0.80 mg/L	0.70mg/L	0.90 mg/L
7.	TDS	40 ppm	60 ppm	60 ppm

The datas obtained for different ponds are different, which indicates that the quality of water in different ponds are different.

3.2 Discussion:

3.2.1 Temperature

The temperature of the Sonari pukhuri is 25 °C is slightly higher than the other two. The lowest temperature among these ponds is 22 °C for Rajapukhuri. And the temperature of Borpukhuri is in between two other ponds i.e, 24 °C.

3.2.2 pH

The pH of the Rajapukhuri pond is higher than the other two ponds i.e., 5.91. The lowest pH among the three is Sonari pukhuri which is 5.30 Borpukhuri contain 5.64 pH.

The pH value of all the ponds indicates that the pond is slightly acidic in nature.

3.2.3 Dissolved Chlorine

Chlorine levels up to 250mg/L are considered safe in drinking water. At this level, harmful health effects are unlikely to occur.

But the level of Chlorine obtained on the three ponds are very low. Rajapukhuri contain more Chlorine i.e, 124 mg/L.

3.2.4 Arsenic

The levels of arsenic is very low in all the three ponds, Which could be not be traced/ determined by normal method.

3.2.5 Hardness

We know that 0 to 60 mg/L as CaCO_3 is classified as soft water. Our result also obtained in this range which indicates that the hardness of all the three water is very low which is good for aquatic animals and domestic purpose. Borpukhuri consists more hardness i.e 35 mg/L than the others two.

3.2.6 Dissolved Oxygen

The solubility range of oxygen for a good pond is 5-14 mg/L.

The dissolved oxygen is higher in Rajapukhuri i.e. 0.70 mg/L. Due to the low amount of dissolved oxygen, the aquatic life is put under stress.

3.2.7 TDS

The TDS levels 150-250 is good for aquatic animal and human beings. But our results shows that the TDS levels is low which indicates that the dissolved solid in water are low.

Among these, Borpukhuri and Rajapukhuri have some TDS value i.e, 60 ppm but Sonari pukhuri has 30 ppm TDS value.

CHAPTER 4 :-

Conclusion

After the analysis of ponds water, it has been found that-

1. The temperature for all the three ponds are good which does not cause and harmful effect on aquatic animals and human beings.
2. All the three ponds water are slightly acidic in nature.
3. Dissolved Chlorine is very low which is good for drinking purpose and arsenic is absent.
4. These pond water are not much hard which is used in domestic purpose.
5. The dissolved oxygen is very low which is insufficient for aquatic living organism.
6. TDS is low which indicate that the calcium and magnesium ions are present in very low amount.

The experiment done during the project was proceeded just under limited equipment inside the laboratory. The project is just a small attempt in finding the water qualities of the three historical ponds, Sonari Charaideo Assam. This project has definitely future aspects for further advance research.

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Green Synthesis of Schiff Base Using Natural Acid Catalyst

A Dissertation report to the Department of Chemistry
Sonari College, Sonari
Affiliated to Dibrugarh University



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Session -: 2023



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This is to certify that the dissertation report entitled, *Green Synthesis of Schiff base using natural acid catalyst* submitted to the Department of Chemistry, Sonari College is a record of project work carried out by Nazia Sultana Choudhury.
The project meets all the requirements of B.Sc. project.

Further, it is also certified that the project has not been submitted for any purposes elsewhere.

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CERTIFICATE

This is to certify that the project work entitled '*Green Synthesis of Schiff base using natural acid catalyst*' submitted by Nazia Sultana Choudhury, a student of 6th semester to the Department of Chemistry, Sonari College, Sonari, affiliated to Dibrugarh University is carried out under my supervision and is found suitable to submit for the partial fulfillment of the B.Sc. Degree in Chemistry. This work, in the present form or in part, has not been submitted for any purpose elsewhere.

I wish her great success in future.

Place: Sonari College

Bikash Kumar Sarmah

Signature of Guide

(Dr. Bikash K. Sarmah)

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At the very outset, I would like to offer my sincere thanks and gratitude to Dibrugarh University for including a project work in the BSc. Semester syllabus.

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CONTENTS

ABSTRACT	6
CHAPTER :1	7
1.1 INTRODUCTION.....	7
1.2 STRUCTURE.....	9
1.3 Present Work.....	9
CHAPTER : 2	9
2.1 AIMS AND OBJECTIVES.....	9
CHAPTER : 3	9
3. EXPERIMENTAL.....	9
3.1 REQUIREMENTS.....	9
3.1.1 APPARATUS.....	9
3.1.2 CHEMICALS.....	10
3.2 METHODOLOGY.....	10
3.3 DIAGRAMS OF CONSECUTIVE STEPS IN THE EXPERIMENT.....	11
CHAPTER : 4	12
4. RESULT AND DISCUSSION.....	12
4.1 YIELD CALCULATION.....	13
4.2 CHARACTERISATION.....	14
4.2.1 APPEARANCE.....	14
4.2.2 MELTING POINT.....	14
4.2.3 THIN LAYER CHROMATOGRAPHY.....	14
4.2.4 OBSERVATION OF NMR	15
4.2.5 OBSERVATION OF IR	16
4.2.6 MECHANISM OF THE REACTION	17
CHAPTER : 5	17
5.1 CONCLUSION.....	17
CHAPTER : 6	18
6.1 REFERENCE.....	18,19

Abstract:-

The utilization of green chemistry techniques dramatically reduces chemical wastes and reaction time as recently have been proven in several organic syntheses. The objective of present research work is also to use green methodologies for synthesis of schiff bases in solvent free condition. The reaction is carried out by the condensation of primary amine and aldehyde by using natural acid catalyst such as fruit juices of tamarind (*Tamarindusindica*), grapes (*Vitis virifera*) and lemon (*Citrus limon*). Compared to traditional methods, this reaction is very simple and has many benefits including cheap and easily available catalyst, good yield of product, simple experimental condition and without generation of pollution in shorter reaction time. The synthesized product was identified by its physical properties, melting point, TLC and characterized by IR and NMR spectroscopy.

CHAPTER 1

1.1 Introduction:

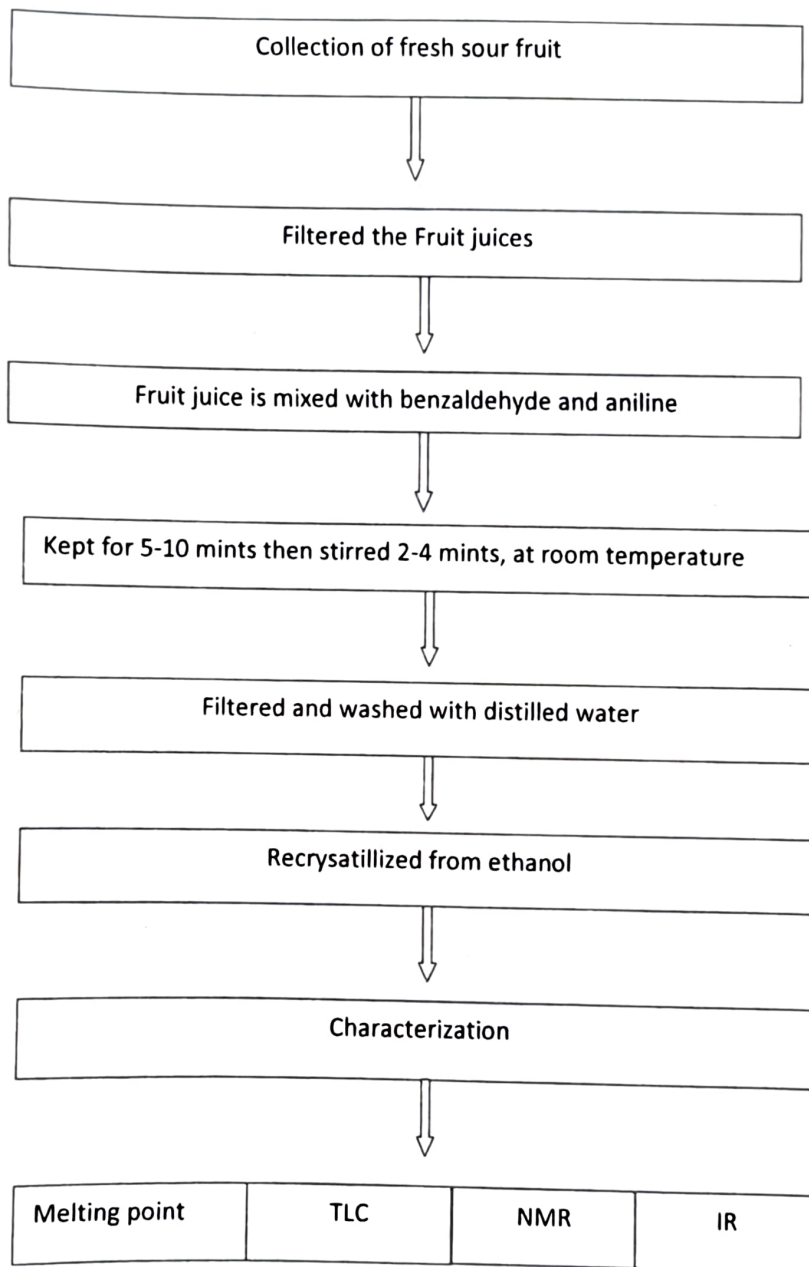
Green chemistry is the design of chemical products and processes that reduce or eliminate the use or generation of hazardous substances [1]. It is helpful to chemists & chemical engineers in research, development and production of more ecofriendly and efficient products which may also have significant financial benefits[2]. Conventionally, synthesis of schiff base is carried out with or without acid catalyst and sometimes by the mixture of aldehyde (or ketone) and amine in organic medium under azeotropic distillation with the simultaneous removal of water[3]. The growing concern for the environment demands the development of eco-friendly and economic process without generating undesired hazardous by-products. Keeping this in mind, a green methodology is used for Schiff base preparation where the reaction of primary aromatic amine with aryl aldehyde is catalyzed by fruit juice as natural acid under solvent free condition.

Schiff bases are condensation products of primary amines with carbonyl compounds and they were first reported by Hugo Schiff in 1864[4]. The common structural feature of these compounds is an azomethine group $-(HC=N)-$ with a general formula $RHC=NR_1$, where R and R_1 are alkyl, aryl, cycloalkyl or heterocyclic groups[5]. These compounds are also known as anils, imine or azomethines. Several studies showed that the presence of a lone pair of electrons in a sp^2 hybridized orbital of nitrogen atom of the azomethine group is of considerable chemical and biological importance[6]. Interest in these compounds is largely due to their structural similarities with natural biological substances and relatively simple procedures of synthesis as well as synthetic flexibility.

Schiff bases are some of the most widely used organic compounds. They are used as pigments and dyes, catalyst, intermediates in organic synthesis, and as polymer stabilizers[7]. They are fundamental materials for the synthesis of various ligands which can be used as chiral auxiliaries in asymmetric synthesis. Metal complexes of Schiff bases have also been used in oxidation reactions[8]. They are well known intermediates for the preparation of azetidinone, thiazolidinone, formazone, arylacetamide, metal complexes and many other derivatives [9]. Schiff base derivatives are employed in different aspects including magnetochemistry, non-linear optics, photo physical studies, catalysis, materials chemistry, chemical analysis, absorption and transport of oxygen.[10]

The biological application of schiff bases such as antitubercular, anticancer, anti bacterial, anti inflammatory, antifungal, antitumor, diuretic, insecticidal, herbicidal, anthelmintic, anti HIV, antiproliferative, anticonvulsant, antihypertensive and antiparasitic activities.[11]

FLOW CHART

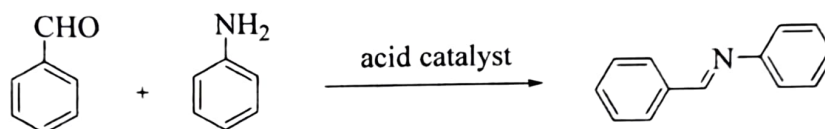


1.3-Structure of *N*-Benzylideneaniline:

The molecular formula of *N*-benzylideneaniline is $C_{13}H_{11}N$ and a molecular weight is 181.23g/mol. The percentage composition of *N*-benzylideneaniline is C(86.16%), H(16.12%), N(7.73%).[12]

1.4 Present Work:

Here, I have prepared *N*-benzylideneaniline by the reaction of benzaldehyde with aniline in the presence of natural acid catalyst viz. tamarind, grapes and lemon juice.



Chapter 2

2.1 Aim and Objective:

1. Green Synthesis of *N*-benzylideneaniline by using natural acid catalyst viz. grapes, lemon, tamarind.
2. Determination of melting point of the prepared compound.
3. Determination of the purity by TLC method.
4. Spectroscopic analysis of the product through ¹H NMR and IR spectroscopic techniques.

Chapter 3

3. Experimental details

3.1 Requirements

3.1.1 Apparatus:

- I. Beakers
- II. Filter paper
- III. Glass rod
- IV. Measuring cylinder
- V. Watch glass
- VI. Capillary tube
- VII. TLC plate

3.1.2 Chemicals

- I. Benzaldehyde
- II. Aniline
- III. Acetone
- IV. Chloroform
- V. Methanol

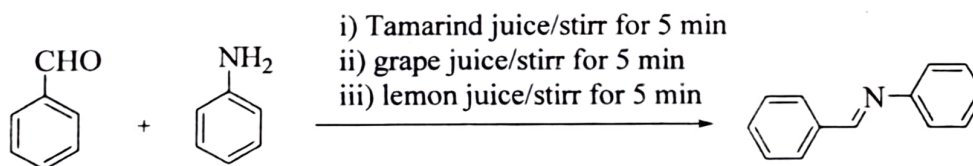
3.2 Methodology:

a. Preparation of a catalyst: Fresh tamarind, grapes and lemon were procured locally. Then grapes were pressed into fruit juicer and filtered with cotton to get liquid juice.

Fresh lemon was cut by using knife and pressed by hand to extract juice. The juice was filtered through filter paper to remove solid residues to obtain a clear juice which was used as catalyst.

Tamarind was soaked in water and kept overnight. Then it was filtered through filter paper to obtain a clear filtrate which was used as a catalyst.

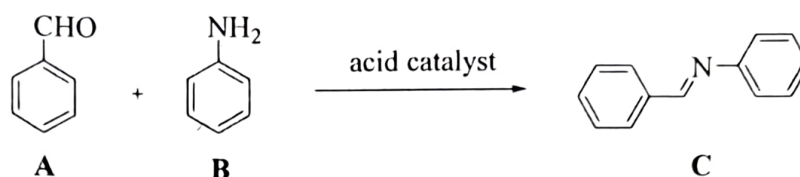
b. Synthesis of Schiff bases with grapes juice, lemon and tamarind under solvent free condition by stirring method:



Scheme 1: Reaction for Schiff base Synthesis in presence of acid Catalyst

4. RESULT AND DISCUSSION:

Reaction Optimisation:-



Equimolar amounts of benzaldehyde **A** (0.1 mol) and aniline **B** (0.1 mol) were mixed in a beaker. Then to it, prepared acid catalyst *i.e.*, tamarind juice was added in variable amounts (1.5 ml, 1 ml, 0.5 ml) and then kept for 5-10 min. Further each reaction mixture was stirred for 2-5 minutes at room temperature. After completion of reaction pale yellow solid crude product appeared which was washed with distilled water and purified by recrystallisation with minimum amount of ethanol. Same procedure is repeated with grapes and lemon juice. Melting point of the products were measured by open capillary method. Purity of the product was checked with the help of TLC method and confirmed by using IR and NMR spectra.

Table 1:- Tamarind juice catalyst optimization.

A	B	Catalyst amount	Product Yield (g)	Percentage yield (%)
0.1 mol	0.1 mol	1.5 mL	14.9	82
0.1 mol	0.1 mol	1 mL	15.9	88
0.1 mol	0.1 mol	0.5 mL	16.9	93

In the above table, we see that by taking 1.5 ml of catalyst, percentage yield of 82% is obtained. When the reaction is done by decreasing the amount of catalyst to 1 ml and 0.5 ml, the percentage yield is 88% and 93% is obtained respectively.

From above data we found that by taking minimum amount of catalyst, maximum amount of product is obtained. On increasing the amount of acid catalyst product yields were decreasing because of acid concentration cannot be too high due to the basicity of amines. If the amine is protonated and becomes non-nucleophilic, equilibrium is shifted to the left and carbinolamine formation cannot occur. Therefore, many Schiff base syntheses are best carried out at mild acidic conditions. Then we decided to check the efficiency of some other natural acid catalysts for this transformation (table 2).

Equimolar amounts of benzaldehyde **A** (0.1 mol) and aniline **B** (0.1 mol) were mixed in a beaker. Then to it, prepared acid catalyst was added (0.5 mL) and then kept for 5-10 min. Further each reaction mixture was stirred for 2-5 minutes at room temperature. After completion of reaction pale yellow solid crude product appeared which was washed with distilled water and purified by recrystallisation with minimum amount of ethanol.

3.3 Diagrams of Consecutive steps in the experiment:



Fig. 1 – Raw Sour Fruits



Fig. 2 – Filtered the juices



Fig.3 – Mixture of Benzaldehyde and aniline with juices



Fig.4– Mixture of Benzaldehyde and aniline after stirring.

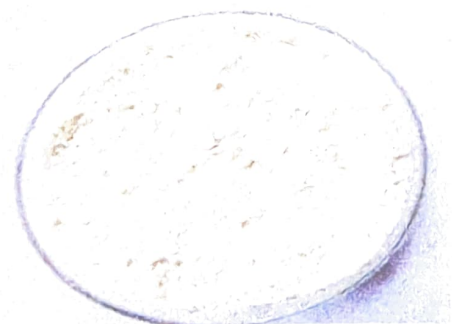


Fig.5- Formation of N-Benzilideneaniline after recrystallisation.

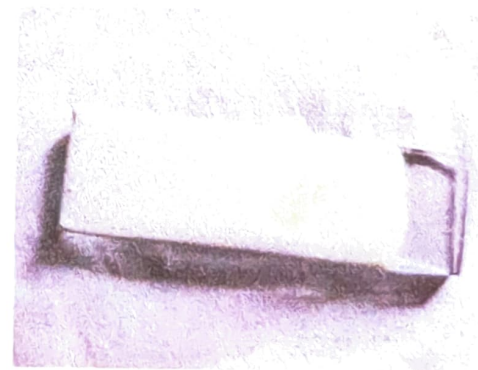


Fig. 6 – TLC

$$\begin{aligned} \text{\% of yield} &= \frac{\text{mmol of product obtained}}{\text{mmol of reactant A}} \times 100\% \\ &= \frac{93.1}{100} \times 100\% \\ &= 93.1\% \end{aligned}$$

4.2 Characterisation:

4.2.1 Appearance:-Yellowish crystal, stable in room temperature and non-hygroscopic. It decomposes at high temperature, insoluble in water but soluble in ethanol.

4.2.2 Determination of melting point:-A small amount of the prepared sample is introduced in a capillary tube and its melting point is determined. The compound found to be melted at 48°C which resembles the melting point of *N*-Benzylideneaniline (49-51°C)[13]. This indicates that the prepared sample is *N*-benzylideneaniline.

4.2.3 - Thin layer Chromatography

TLC plate was prepared by using a uniform coating of silica gelG over the glass plate which was dried in hot air oven. The sample was dissolved in acetone and the spot was applied on the TLC plate with the help of capillary tube. Then the TLC plate was run by using chloroform and methanol(3:7) as eluent in a closed container. When the solvent front has nearly reached the top of the stationary phase, the plate was removed from the container and the solvent front was marked with a pencil. The spot was visualized by inserting the TLC plate in an iodine chamber. After removing the plate, the distance travelled by the compound was marked. From this, the retention factor or R_f value can be calculated[14].

$$\begin{aligned} R_f &= \frac{\text{distance travelled by the solute}}{\text{distance travelled by the solvent}} \\ &= \frac{5.0 \text{ cm}}{6.7 \text{ cm}} \\ &= 0.7 \end{aligned}$$

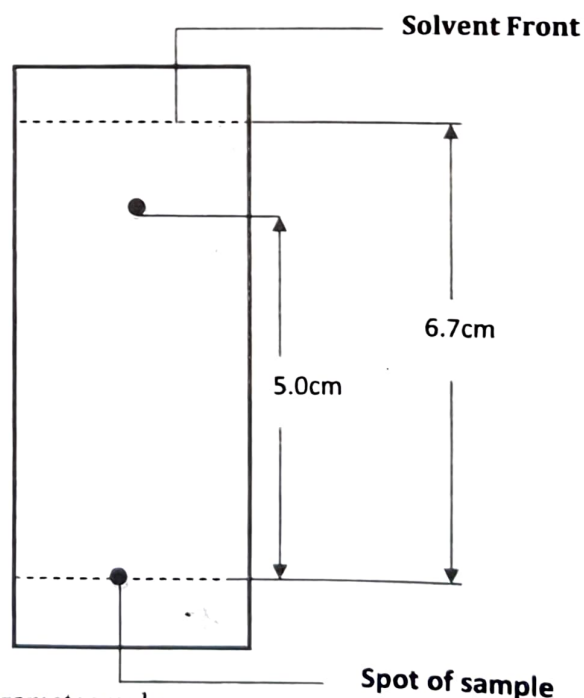


Fig. 7: Thin layer chromatography

Table 3: Physical Characteristics of Product

Products name and chemicals formula	Product colour	Product smell	Physical state	Solubility	Melting Point	R _f Value
N-benzylidene aniline(C ₁₃ H ₁₁ N)	Pale yellow	Disagreeable	Crystalline solid	Insoluble in distilled water but soluble in ethanol	48° C	0.7

4.2.5 Observation of NMR

The ¹H NMR spectrum of the prepared *N*-benzylidene aniline is recorded using CDCl₃ as a solvent at room temperature. It is presented below.

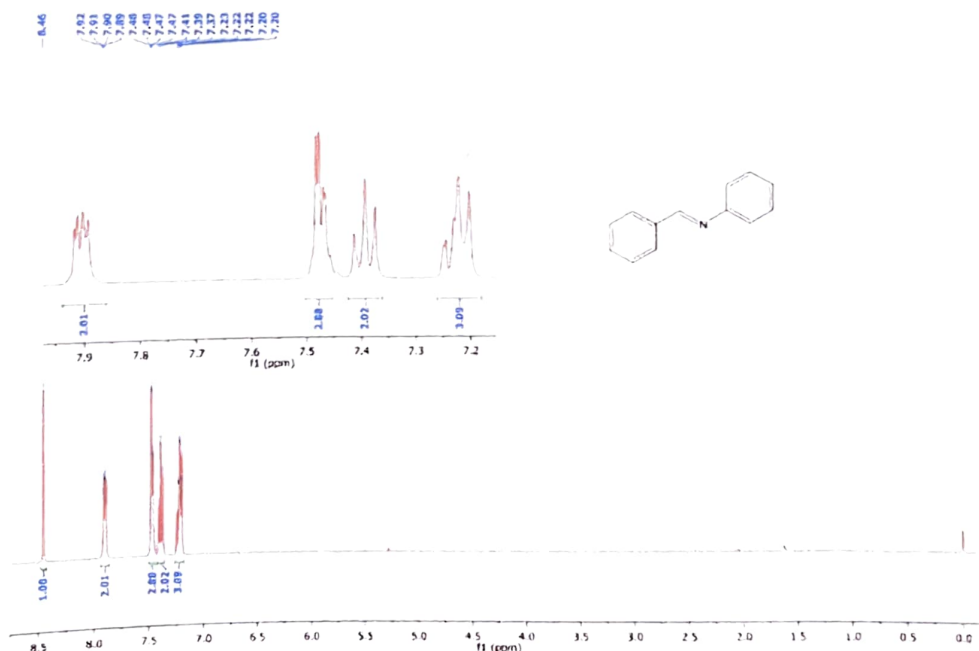


Fig 8: ¹H NMR spectrum of *N*-benzylideneaniline (CDCl₃, 400 MHz, 298 K)

¹H NMR(400 MHz, CDCl₃) δ8.46(s,1H, -HC=N), 7.92-7.89(dd,2H, aromatic *o*-CH),7.48-7.47(m, 3H, aromatic *m*, *p*-CH),7.39-7.37(m, 2H, aromatic *m*-CH), 7.23-7.20(m, 3H, aromatic *o*, *p*-CH).

The NMR spectrum showed a highly de-shielded signal at 8.46 ppm which can be attributed to the azomethine proton (HC=N-) due to -I effect of nitrogen atom. The aromatic ring which

is bonded with C center of imine group has the *ortho* Hs at the range 7.92-7.89 ppm as a doublet of doublet and *para*-Hs at the range of 7.48-7.47 as a multiplet. The second aromatic ring which is attached with N-atom of imine is comparatively shielded due to the +R effect of imine N. In this group *meta* Hs come at the range of 7.39-7.37 as a multiplet. But *ortho* and *para* Hs are comparably shielded from *meta* Hs and show peaks at the range of 7.32-7.20 ppm as a multiplet[15].

4.2.6 Observation of IR:

The IR spectrum of the prepared *N*-benzylidene aniline sample showed stretching and out of plane bending vibration for ($sp^2 = C-H$) at 3060.06cm^{-1} and 690.73cm^{-1} respectively. This indicates the presence of aromatic ring in the prepared compound. Due to $C=C$ stretching in aromatic ring the peak obtained in 1624.87cm^{-1} . *N*-benzylidene aniline also shows stretching vibration of the azomethine group ($C=N$) at 1576.88cm^{-1} in the spectrum[15].

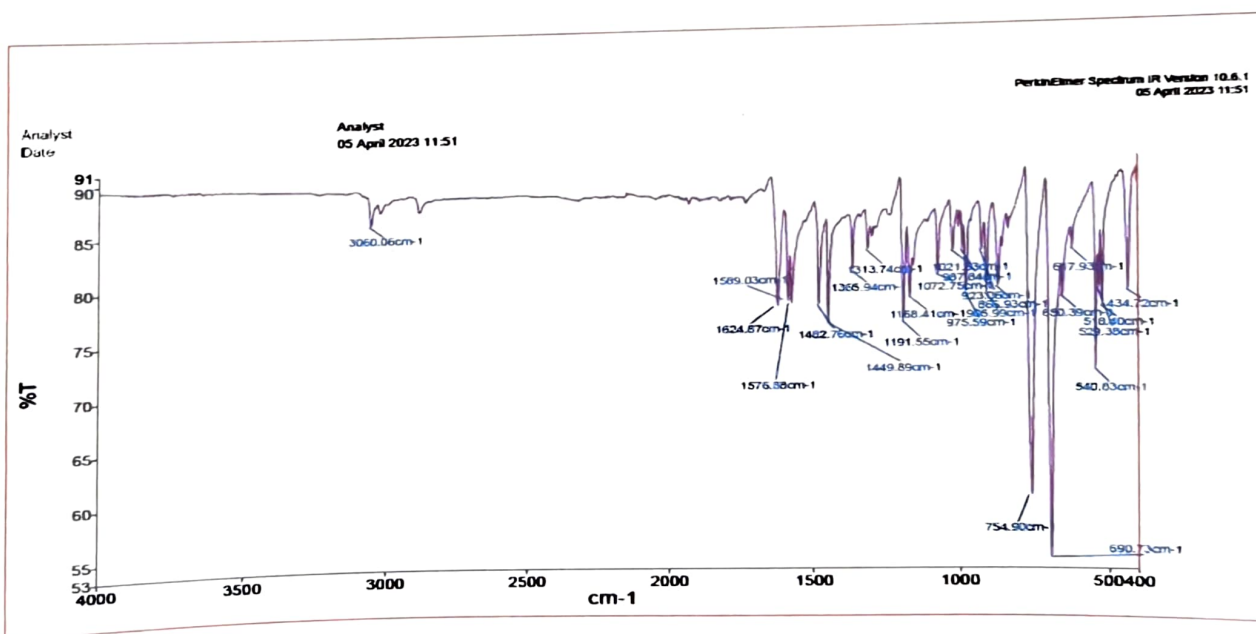
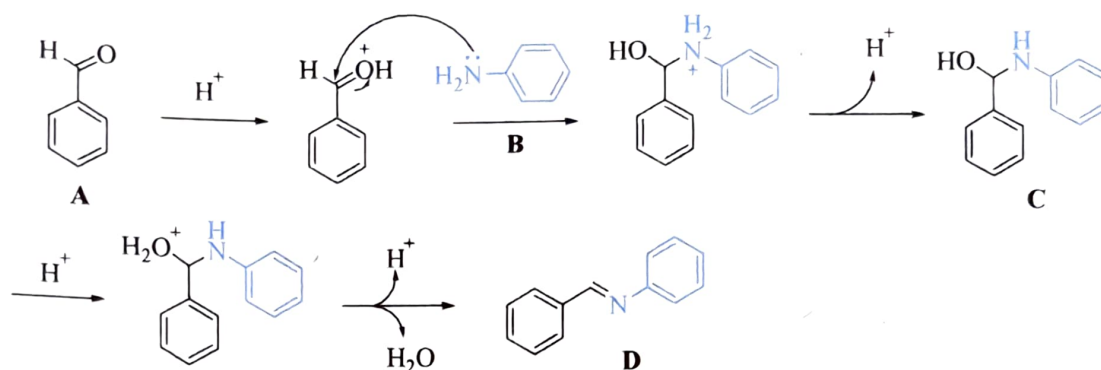


Fig. 9: IR Spectrum of *N*-benzylideneaniline

4.2.6 Mechanism –



Scheme2: Mechanism for formation of *N*-benzylideneaniline

The carbonyl group in benzaldehyde is electrophilic in nature whereas amino group in aniline is nucleophilic.

At first benzaldehyde **A** gets protonated by acid catalyst. Then nucleophilic attack by the lone pair of N atom of aniline **B**, followed by deprotonation forms unstable addition compound called carbinolamine **C**. Then carbinolamine forms the stable product **D** by removal of water in the presence of catalyst.

Chapter 5 :-

Conclusion : The present work focuses the importance of normal acids of fruits in chemical reaction as a biocatalyst in organic transformation. The growing interest of fruit juice in organic synthesis is mainly due to their acidic properties, enzymatic activity, environmental-friendly characteristic, low cost and easy availability. The catalytic activity including the application of fruit juices in formation of Schiff base has been studied. The Schiff base product yield is good about in different natural acid catalyst. The reaction shows a higher percentage yield of schiff base derivative in presence of tamarind juice compared to grape and lemon juices where the reaction time is only about 15 min. The prepared Schiff base was characterized using different spectroscopic techniques such as ¹H NMR and IR Spectroscopy and confirmed it by comparing with literature report.

Chapter 6:-

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**Synthesis and Spectroscopic Characterization of the
Metabolic Byproduct Hippuric Acid**

(A Dissertation report to the Department of Chemistry)

Sonari College, Sonari

Affiliated to Dibrugarh University



SUBMITTED BY:

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Session: 2022-23

Mr. Amrit Kumar Borpuzari

Date: 23/05/2023

Associate Professor (HoD), Dept. of Chemistry

CERTIFICATE

This is to certify that the dissertation report entitled, **“Synthesis and Spectroscopic characterization of the Metabolic Byproduct Hippuric Acid”** submitted to the Department of Chemistry, Sonari College is a record of project work carried out by **Pinak Chakraborty**.

Further, it is also certified that the project has not been submitted for any purpose elsewhere.

Examined
Raj 21/6/23
Internal Examiner

Place: Sonari college



(Mr. Amrit K. Borpuzari)

Dr. Bikash Kumar Sharma

Date: 23/05/2023

Asst. Professor (Dept. of Chemistry)

CERTIFICATE

This is to certify that the project work entitled "**Synthesis and Spectroscopic characterization of Hippuric Acid**" submitted by **Pinak Chakraborty**, a student of 6th semester to the Department of Chemistry, Sonari College, Sonari, affiliated to Dibrugarh University is carried out under my supervision and is found suitable to submit for the partial fulfillment of the B.Sc. Degree in chemistry. This work, in the present form or in part, has not been submitted for any purpose elsewhere.

I wish his great success in future.

Place: Sonari College

Bikash Kumar Sarmah
Signature of Guide

(Dr. Bikash K. Sarmah)

ACKNOWLEDGEMENT

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CONTENTS

CHAPTER-1

ABSTRACT

INTRODUCTION

STRUCTURE AND CHEMICAL BONDING OF HIPPURIC ACID

PRESENT WORK

CHAPTER-2

2.1 AIMS AND OBJECTIVES

CHAPTER-3

3 EXPERIMENTAL

REQUIREMENTS

APPRATUS

CHEMICALS

METHODOLOGY

RECRYSTALLISATION

CHAPTER-4

4 RESULTS AND DISSCUSSION

APPEARANCE OF THE PRODUCT

DETERMINATION OF THE MELTING POINT

YIELD CALCULATION

OBSERVATION OF NMR

OBSERVATION OF IR

OBSERVATION OF TLC

CHAPTER-5

5.1 CONCLUSION

CHAPTER-6

6.1 REFERENCE

CHAPTER-1

ABSTRACT

The aim of this project is to carry out the synthesis and spectroscopic characterization of a metabolic byproduct i.e. hippuric acid which is present in urine. Hippuric acid molecules are held together in three dimensional spaces by one O-H-O and one N-H-O hydrogen bond to the peptide oxygen atom. Herein, we have prepared this acid by the reaction of benzoyl chloride with glycine in presence of NaOH as base. The color of the complex is white. The IR, NMR spectra and TLC were recorded and discussed.

INTRODUCTION

Hippuric acid is a major amino acid produced in the liver and it is important for body metabolism, it acts as a metabolite for urinary excretion of benzoic acid.¹ There are different reports for the synthesis of substituted Hippuric acid, for example: 4 amino Hippuric acid², P-amino hippuric acid³ and many more were synthesized and utilized for many applications. Ringertz had demonstrated or set up the structure of hippuric acid by the study of x-ray diffraction. It is found that hippuric acid is formed in the order of orthorhombic structure⁴.

Hippuric acid has a tendency to form complex with metals due to the presence of two donor sites, viz, amine and carboxylate groups containing oxygen and nitrogen respectively. Hippuric acids can react with various metal ions like Cd(II), Hg(II), Zn(II), Mn(II), Co(II), Ni(II), Ag(II) to form coordinate bonds from its carboxylic oxygen atom.⁵ The coordination bond is formed in hippuric acid with other metal ions through the carboxylic oxygen atom, which acts as monodentate ligand and the other formed through nitrogen atom as bidentate ligand.⁶

Hippuric acid is present in the urine of animals and a little amount in humans. Subramanian et. al. has extracted hippuric acid from buffalo urine and assessed its antioxidant activities.⁷

Hippuric acid has many applications in various fields, it acts as an intermediate and starting materials for various organic and inorganic synthesis like production of 4-arelydin-2 phenyl

5(4n) oxadone derivatives (A'z lactone).⁸ It has also found many application in pharmaceuticals such as it is used in study of cell biology ,chemical biology, bioactive small molecule, amino acid derivatives ,peptide synthesis and used in manufacturing of medicines. .

In 1829, Justus ven Liebig outlined that hippuric acid is different compound than benzoic acid .⁹ Later French scientist Victor Dessaiyanes synthesized hippuric acid by reaction between Zinc salt of glycine and benzoyl chloride .¹⁰ Now a days, synthesis of hippuric acid is done by acylation reaction of benzoyl chloride with glycine .¹¹

From above discussion on hippuric acid,we may conclude that it is an important by product of urine which has good environment effect as well as more in medicinal industry .Hippuric acid is not only acts as a body metabolite but it is also acts as an indicator in many reaction¹². So the synthesis of hippuric acid is still a valuable process which encouraged us to carry out this study.

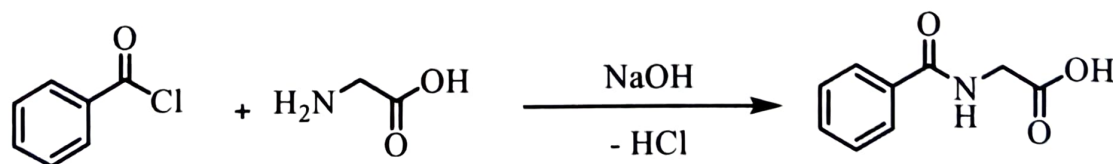
STRUCTURE AND CHEMICAL BONDING OF HIPPURIC ACID

Hippuric acid crystallization with four molecules in an orthorhombic unit cell with a=8.874,b=10.577 and c=9.1171. The benzene ring, the peptide part and the carboxylic group are planner and twisted with each other.

The hippuric acid molecule contains a total number of 22(twenty two) bonds .There are 13(thirteen) non H-bonds, eight multiple bonds, three rotatable bonds, six aromatic bonds, six member ring ,one carboxylic acid ,one secondary amides and one hydroxyl groups.

PRESENT WORK

In this work, I have prepared hippuric acid by the reaction between glycine and benzoyl chloride in the presence of NaOH as base.



2.1 AIMS AND OBJECTIVES

The main aims of the present work are given below:

1. To study N-Benzoylation of glycine towards synthesis of hippuric acid.
2. Determination of melting point of the prepared compound.
3. Spectroscopic analysis of the product through ^1H NMR and IR spectroscopic techniques.

CHAPTER 3

3. EXPERIMENTAL

REQUIREMENTS

APPARATUS

- i. Conical flask
- ii. Filter paper
- iii. Separating Funnel
- iv. Cork
- v. Glass rod
- vi. Digital balance
- vii. Volumetric Cylinder
- viii. Beaker.

CHEMICALS

- i. Glycine

ii. 0% NaOH

iii. Benzoyl chloride

METHODOLOGY

1. First 2.5 g of glycine is weighed. A 10% NaOH is prepared separately dissolving 10 g of NaOH in 100 ml of water.

2. Now, 2.5 g of glycine is dissolved in 2.5 ml of 10% NaOH solution until a clear solution is obtained.

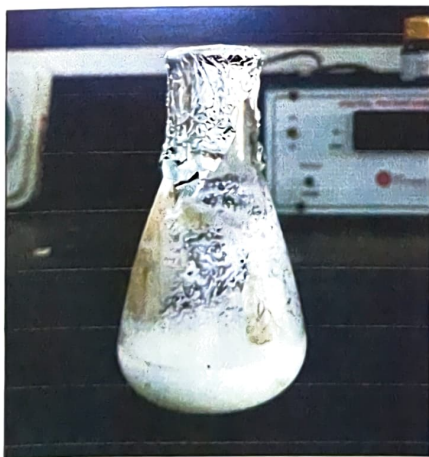
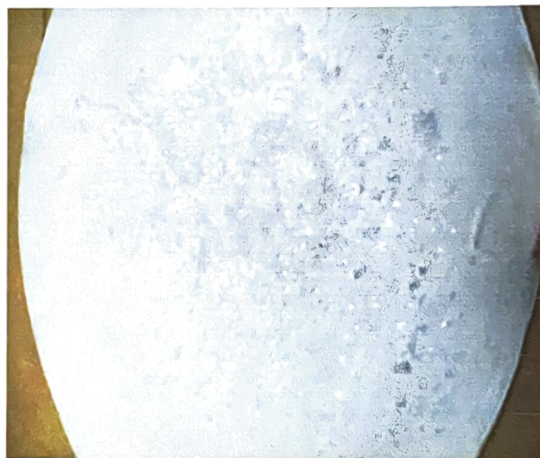
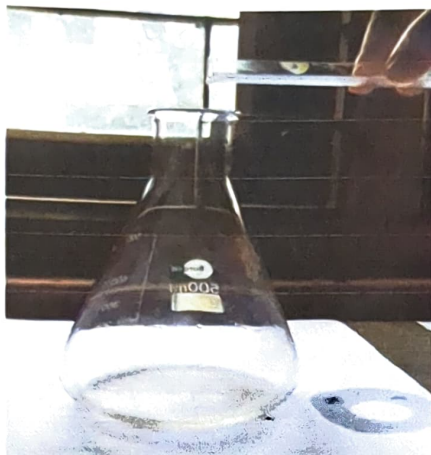
3. Using measuring flask, 4.5ml of benzoyl chloride is measured. Then it is added to the previous solution in two portions. The conical flask is closed using the cork and the solution is stirred using magnetic stirrer after addition for 3 minutes.

4. Now, the solution is transferred into a beaker containing cold water. The precipitate is filtered.

RECRYSTALLISATION:

After the formation of the product, it is dissolved in boiling water. The mixture is again filtered to remove the impurities. The filtrate is left for 10 minutes. When white crystals are formed which are again filtered and dried. Now the crystal is weighed with the help of a digital balance.

DIAGRAMS OF CONSECUTIVE STEPS IN THE EXPERIMENTS



CHAPTER-4

4. Result and Discussion

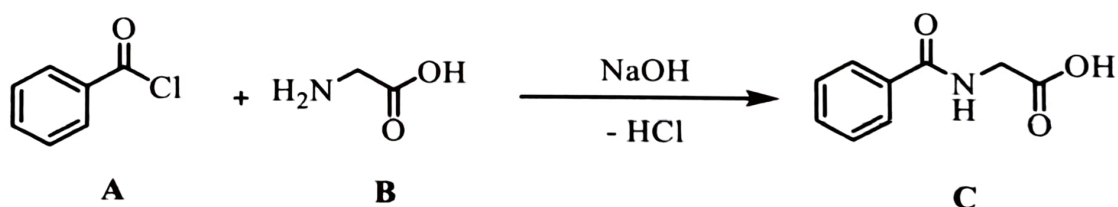
Appearance of the product

Hippuric acid is obtained as sharp shining crystal.

Determination of melting point

A small amount of prepared product is carefully placed in a capillary tube and tests its melting point on a thermometer. The compound is found to be melt at 183°C which resembles the melting point of hippuric acid (187°C). This indicates that the prepared sample is hippuric acid.

Yield calculation



	A	B	NaOH
Molecular weight	140.6g/mol	75.0g/mol	39.99g/mol
Weight taken	5.4g	2.5g	10 g
mmol	38.40	33.4	250
Equivalent	1.1	1	7.57

Here, mmol = weight taken in mg /molecular weight

Since benzoyl chloride is liquid, its density =1.21g/ml

Weight taken=4.5ml x 1.21g/ml

$$\Rightarrow 5.4 \text{ gm} = 5.4 \times 1000\text{mg}$$

$$= 5400\text{mg}$$

mmol for benzoyl chloride (A)

$$= 5400\text{mg} / 140.6\text{g/mol}$$

$$= 38.40\text{mmol}$$

For glycine(B),

$$= 2.5\text{g} \times 1000 / 75(\text{g/mol})$$

$$= 2500\text{mg} / (75\text{g/mol})$$

$$= 33.4\text{mmol}$$

For NaOH = 250 mmol

Molecular weight of hippuric acid = 179.2 g/mol

Yield after crystallization = 1.59 g

Therefore, amount of hippuric acid in mmol = $(1.59 \times 1000) / 179.2$

$$= 8 \text{ mmol}$$

Percentage of yield = $(\text{wt. mmol of product}) / (\text{wt. in mmol of glycine}) \times 100\%$

$$= (8.0 / 33.4) \times 100\%$$

$$= (.2395) \times 100\%$$

$$= 23.95\%$$

OBSERVATION OF NMR

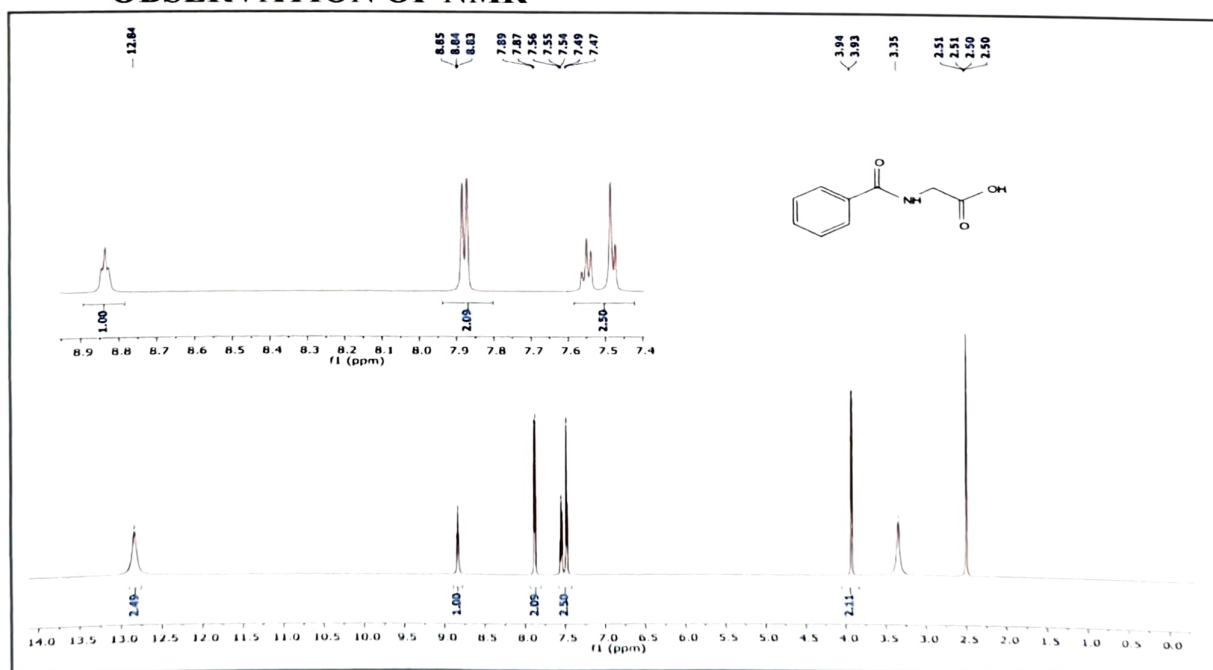


Fig: ^1H NMR spectrum of Hippuric Acid (DMSO-d_6 , 500MHz, 298K)

The above NMR figure of our prepared hippuric acid is analyzed in the basis of ^1H NMR. The ^1H NMR spectrum provides information about no of proton types of the compound and the environmental properties of each type of hydrogen proton.

The different peaks in the diagram represent the absorption of different groups of the compound. In the spectrum the the peak at 3.93 ppm represents $-\text{CH}_2-$ group at alpha position to both $-\text{COOH}$ and $-\text{NH}-$ groups and due to the prtseence of these elecycyon withdrawing groups,it comes at downfiled region. The H of carboxylic acid group shows peak at 12.84 ppm.The two o-H's of benzene ring come at 7.8-7.9ppm whereas the m- and p-H's comes at 7.4-7.6 ppm.The peak of NH proton shows peak at 8.84ppm.

OBSERVATION OF IR

The IR analysis spectrum of our prepared hippuric acid compound showed a sharp peak at 1681.79 cm^{-1} of COOH group and the peak at 2827 cm^{-1} of CH_2 and 1600.17 cm^{-1} of NH group.

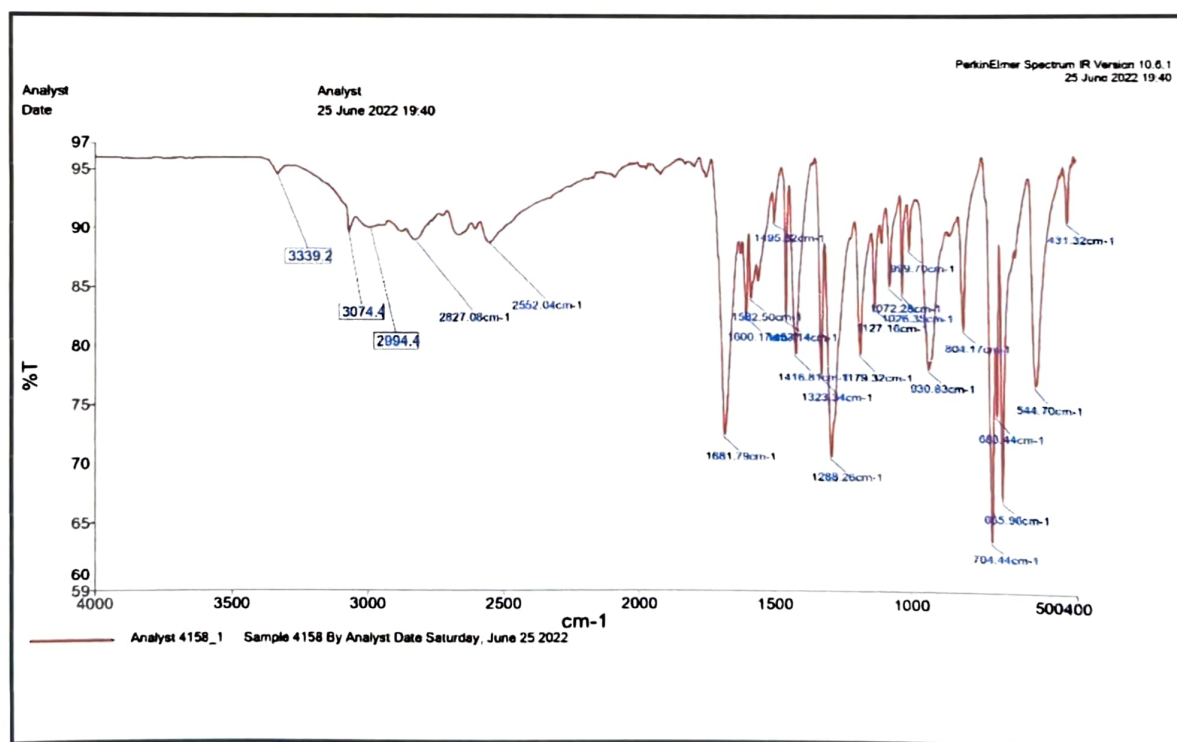


Fig: IR spectrum of Hippuric Acid

OBSERVATION OF TLC

Thin layer chromatography (TLC) plate was prepared by uniformly applying a coating the slurry of silica gel over the glass plate and drying it in an oven for one hour.. The prepared sample is dissolved in acetone and spot was applied on the TLC plate with the help of capillary tube. Then the TLC plate was run by using solvents n-hexane and methyl acetate (5:5) as eluent a container Iodine was used as the indicator. Placed the TLC plate within the iodine chamber until the plate gives light brown color on it. Removed the plate carefully and record the distance travelled by product or compound. From this we can calculate the R_f value.

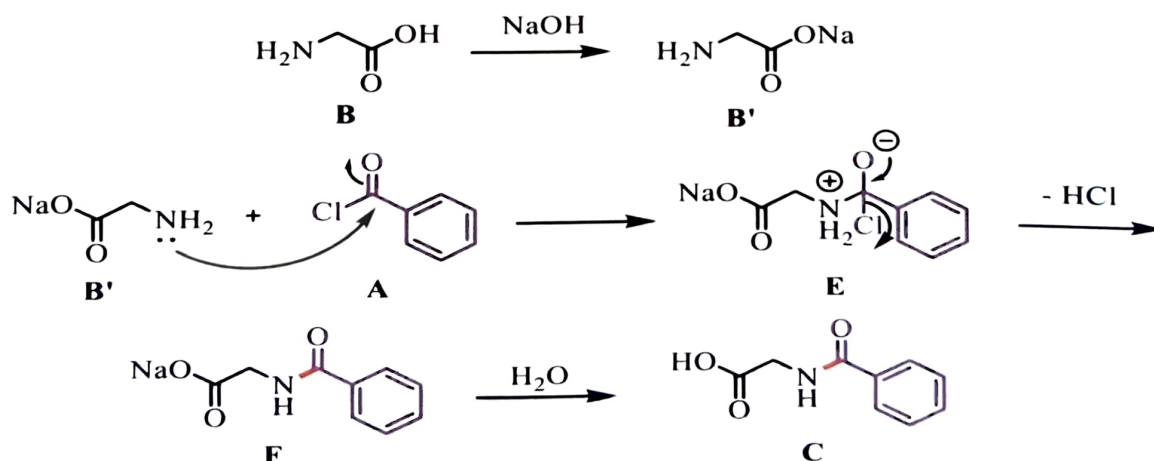
$$R_f = \text{Distance travelled by the solute} / \text{Distance travelled by the solvent}$$

$$= 4.8\text{cm} / 6.3\text{cm}$$

$$= 0.76$$

4.6 MECHANISM OF THE REACTION

First glycine **B** will be deprotonated by the base to give **B'**. Then **B'** will attack on the electrophilic carbonyl carbon centre of benzoyl chloride **A** to give **E**. **E** upon elimination of HCl and subsequent treatment with water, will give hippuric acid **C**.



CHAPTER-5

5. CONCLUSION

From the literature review, it is concluded that Hippuric acid has environmental importance. It is also serves as ligand to form coordination complexes with various metals. So the synthesis of hippuric acid has been of interest from a very long time. Using a literature report procedure, We have prepared hippuric acid and characterized using different techniques such as ^1H NMR and IR spectroscopy and confirm it by comparing with literature report. Further, the main point is also determined to confirm the identity of the compound.

CHAPTER-6

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Department of Chemistry

Sonari College, Sonari

Charaideo-785690, Assam

CERTIFICATE

This is to certify that the project work entitled "*Green Chemistry for the preparation of Silver Nanoparticle using Mint leaves extract*" has carried out by **Bidyut Bikash Gogoi** bearing **Roll No: 10520013** under my supervision in laboratories of the Department of Chemistry, Sonari College, is found suitable for submission to the partial fulfillment of the requirements for the Degree of Bachelor of Science in Chemistry.

This work in the present form or part has not been submitted anywhere for any other purpose elsewhere.

Supervisor

Dr. Bikash Kr. Sarmah

Dept. of Chemistry

Sonari College

Sign: *Bikash Kr. Sarmah*

Date: *25.2023*



Department of Chemistry

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Charaideo-785690, Assam

CERTIFICATE

This is to certify that the project work entitled "*Green Chemistry for the preparation of Silver Nanoparticle using Mint leaves extract*" has carried out by **Anupam Deb** bearing Roll No: **10520009** under my supervision in laboratories of the Department of Chemistry, Sonari College, is found suitable for submission to the partial fulfillment of the requirements for the Degree of Bachelor of Science in Chemistry.

This work in the present form or part has not been submitted anywhere for any other purpose elsewhere.

Supervisor

Dr. Bikash Kr. Sarmah

Dept. of Chemistry

Sonari College

Sign: *Bikash Kumar Sarmah*

Date: *2/5/23*



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Suchitra Narayan Rajkhowa

Date: 20.05.2022

Associate Professor (Dept. of Chemistry)

CERTIFICATE

This is to certify that the project work entitled "To analysis the physico-chemical parameter of three ancient ponds of Sonari" submitted by Jyoti Kumari Sah, a student of 6th sem of the Department of Chemistry, Sonari affiliated to Dibrugarh university is carried out under my supervision and is found suitable to submitted the partial fulfillment of the B.Sc. Chemistry. This work in the present form in part, has not been submitted for any purpose elsewhere.

I wish her great success in future.

Place: Sonari College

S. N. Rajkhowa
Signature of Guide

Suchitra Narayan Rajkhowa



Department of Chemistry

Sonari College ,Sonari

Charaideo – 785690, Assam, India

Suchitra Narayan Rajkhowa

Date: 2/5/23

Associate Professor(Dept. of Chemistry)

CERTIFICATE

This is to certify that the project work entitled “To analysis the physico-chemical parameter of three ancient ponds of Sonari” submitted by Pratiksha Baruah, a student of 6th sem of the Department of Chemistry, Sonari affiliated to Dibrugarh university is carried out under my supervision and is found suitable to submitted the partial fulfillment of the B.Sc. Chemistry. This work in the present form in part, has not been submitted for any purpose elsewhere.

I wish her great success in future.

Place: Sonari College

S. N. Rajkhowa
Signature of Guide

Suchitra NarayanRajkhowa



Head Department of Chemistry
Sonari College, Sonari
Charaideo-785690, Assam, India

Mr Amrit Kumar Borpuzari
Assistant Professor, Dept. of Chemistry(HOD)

Date: 2/05/23

CERTIFICATE

This is to certify that the dissertation report entitled, *Green Synthesis of Schiff base using natural acid catalyst* submitted to the Department of Chemistry, Sonari College is a record of project work carried out by **Isha Paul**

The project meets all the requirements of B.Sc. project.

Further, it is also certified that the project has not been submitted for any purposes elsewhere.

Examined
Prj 01/6/23
Internal Examiner

Place: Sonari College

(Amrit Kumar Borpuzari)



Head Department of Chemistry
Sonari College, Sonari
Charaideo-785690, Assam, India

Mr Amrit Kumar Borpuzari
Assistant Professor, Dept. of Chemistry(HOD)

Date: 02.05.2023

CERTIFICATE

This is to certify that the dissertation report entitled, *Green Synthesis of Schiff base using natural acid catalyst* submitted to the Department of Chemistry, Sonari College is a record of project work carried out by **Parthajit Thakur**.
The project meets all the requirements of B.Sc. project.

Further, it is also certified that the project has not been submitted for any purposes elsewhere.

Examined
01/6/23
Internal Examiner

Place: Sonari College

(Amrit Kumar Borpuzari)

Mr. Amrit Kumar Borpuzari

Date: 23/05/2023

Associate Professor (HoD), Dept. of Chemistry

CERTIFICATE

This is to certify that the dissertation report entitled, "**Synthesis and Spectroscopic characterization of the Metabolic Byproduct Hippuric Acid**" submitted to the Department of Chemistry, Sonari College is a record of project work carried out by **Raj Gogoi**.

Further, it is also certified that the project has not been submitted for any purpose elsewhere.

*Examined
Raj 01/26/23
Internal Examiner*

Place: Sonari college



(Mr. Amrit K. Borpuzari)

Dr. Bikash Kumar Sharma

Date: 02/05/23

Asst. Professor (Dept. of Chemistry)

CERTIFICATE

This is to certify that the project work entitled "**Synthesis and Spectroscopic characterization of Hippuric Acid**" submitted by **Tushar Deep Tamang**, a student of 6th semester to the Department of Chemistry, Sonari College, Sonari, affiliated to Dibrugarh University is carried out under my supervision and is found suitable to submit for the partial fulfillment of the B.Sc. Degree in chemistry. This work, in the present form or in part, has not been submitted for any purpose elsewhere.

I wish his great success in future.

Place: Sonari College

Bikash Kumar Sarmah

Signature of Guide

(Dr. Bikash K. Sarmah)

Mr. Amrit Kumar Borpuzari

Date: 1-06-23

Asst. Professor (Dept. Of Chemistry)


CERTIFICATE

This is to certify that the project work entitled "Preparation and spectroscopic characterization of Tetrahydrated copper(II) citrate complex" submitted by Ruma Turi, a student of 6th semester of the department of chemistry, Sonari College, Sonari. The project meets all the requirements of B.Sc. project

Further, it is all certified that the project has not been submitted for any purpose elsewhere.

*Examined
Project 1/6/23
Internal Examiner*

Place: Sonari College.


(Amrit Kumar Borpuzari)