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Department of Chemical Engineering

Magazine students

Academic Year:2025-26

A PROJECT REPORT ON

**“Recovery of Hydrochloric Acid from Spent Pickling Acid by
Evaporation”**

Submitted in partial fulfilment of the requirements for the award of the degree
of

BACHELOR OF ENGINEERING (B.E.)

IN CHEMICAL ENGINEERING

BY

Sarthak Arun Chavan (06)

Piyush Arun Dangar (08)

Samruddha Santosh Gurav (09)

UNDER THE GUIDANCE OF

Dr. N. Y. Ghare



DEPARTMENT OF CHEMICAL ENGINEERING

Gharda Institute of Technology, Khed (Lavel)

UNIVERSITY OF MUMBAI

ACADEMIC YEAR: 2025 - 2026

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”



CERTIFICATE

This is to certify that the project entitled **“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”** is a bonafide work carried out by **Piyush Arun Dangar (08), Sarthak Arun Chavan (06) & Samruddha Santosh Gurav (09)** in partial fulfillment of the requirements for the award of the degree of **Bachelor of Engineering (B.E.) in Chemical Engineering** from University of Mumbai during the academic year **2025 – 2026**.

Project Guide

Dr. N. Y. Ghare

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Dr. S. J. Kulkarni

Principal

Dr. P. B. Patil

Internal Examiner

External Examiner

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

Project Report Approval for B.E.

This project report entitled **“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”** submitted by **Piyush Arun Dangar, Sarthak Arun Chavan & Samruddha Santosh Gurav** in partial fulfillment of the requirements for the award of the degree of **Bachelor of Engineering (B.E.) in Chemical Engineering** from University of Mumbai has been examined and approved.

Examiners

1.) _____

2.) _____

Date: -

Place: - Gharda Institute of Technology, Lavel

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

DECLARATION

We, **Piyush Arun Dangar**, **Sarthak Arun Chavan**, and **Samruddha Santosh Gurav**, students of **Bachelor of Engineering (B.E.) in Chemical Engineering**, hereby declare that the project entitled **“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”** submitted to University of Mumbai in partial fulfillment of the requirements for the award of the degree of **Bachelor of Engineering in Chemical Engineering** is a record of original work carried out by us under the guidance and supervision of **Dr. N. Y. Ghare**, at **Gharda Institute of Technology, Khed (Lavel)** during the academic year **2025 -2026**.

We further declare that the work presented in this project report is original and has been carried out independently by us. The data, results, and analysis presented in this report are true to the best of our knowledge and belief. All the information collected from various sources has been properly acknowledged and referenced wherever necessary.

Dangar Piyush Arun (08)

Chavan Sarthak Arun (06)

Gurav Samruddha Santosh (09)

Date: -

Place: - Gharda Institute of Technology, Lavel

ACKNOWLEDGEMENT

We would like to express our sincere gratitude to all those who have contributed to the successful completion of our project entitled **“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”**. We are highly indebted to our respected **Project Guide, Dr. N. Y. Ghare**, for his valuable guidance, continuous support, and encouragement throughout the course of this project. His insightful suggestions and expert supervision have been instrumental in shaping this work.

We would also like to express our heartfelt thanks to **Dr. S. J. Kulkarni, Head of Department of Chemical Engineering**, for providing us with the necessary facilities and a conducive environment to carry out this project successfully.

We extend our sincere thanks to **Dr. P. B. Patil, Principal** of Gharda Institute of Technology, Lavel for providing us with the opportunity and resources required for the completion of this project.

We are also grateful to all the faculty members and staff of the Department of Chemical Engineering, Gharda Institute of Technology, for their support and cooperation.

Finally, we express our sincere gratitude to our family members and friends for their constant encouragement and moral support throughout the completion of this project.

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

ABSTRACT

The proper disposal of spent pickling liquor (SPL), which is a by-product of steel pickling plants, poses a major environmental hazard and economic inefficiency due to the production of hazardous waste and the loss of valuable acid. Traditional methods of treating SPL, like lime neutralization, produce considerable amounts of hazardous sludge and do not extract any reusable materials.

The objective of this study was the development of the methodology for extracting hydrochloric acid (HCl) from SPL based on evaporation and condensation at laboratory level. Artificial SPL was produced through the reaction of rusty iron scrap with HCl in order to obtain a liquid with iron concentration of 7.2 g/L and pH 2.0. Then, thermal evaporation took place at 110°C taking advantage of different volatilities of HCl and dissolved metal salts.

The results of the experiment show that the efficiency of acid extraction is about 80-85%. Distillation produces a liquid with concentration of 12-17% HCl. At the same time, there is a significant decrease in the concentration of dissolved iron from 7.2 g/L to 0.5 g/L. Extracted HCl has a pH value of 1.0-1.5 and can be used in further production.

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“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

Nomenclature

HCl - Hydrochloric Acid

SPL - Spent Pickling Liquor

FeCl₂ - Ferrous Chloride

FeCl₃ - Ferric Chloride

°C - Degree Celsius

g/L - grams per liter

CHAPTER 1:

INTRODUCTION

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

1.1 Background of Steel Pickling

Steel making and metal fabrication often leave a layer of iron oxide, commonly referred to as "mill scale" or rust, on the surface of the metal. For efficient downstream processing like electroplating or painting, pretreatment of the metal surface is strictly necessary. The "pickling" process is a chemical metal surface treatment that consists of dipping corroded steel products into acidic baths to dissolve these oxides, producing a bright and clean surface. In modern industries, hydrochloric acid (HCl) is predominantly used because it cleans rapidly and prevents localized pitting corrosion.



Figure 1.1: Steel Pickling Process Showing Removal of Oxide Layer

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

1.2 The Chemistry of Pickling and Waste Generation

During pickling, the hydrochloric acid aggressively attacks the iron oxide layers on the steel products. The primary chemical reactions are:

- $\text{FeO} + 2\text{HCl} \rightarrow \text{FeCl}_2 + \text{H}_2\text{O}$
- $\text{Fe}_2\text{O}_3 + 6\text{HCl} \rightarrow 2\text{FeCl}_3 + 3\text{H}_2\text{O}$

As these reactions proceed, the acid is consumed, and the bath becomes heavily concentrated with iron (II) and iron (III) chlorides. When the HCl concentration drops significantly and dissolved metals reach high levels, the cleaning efficiency plummets. At this stage, the acid is considered "Spent Pickling Liquor" (SPL) and is conventionally discarded.

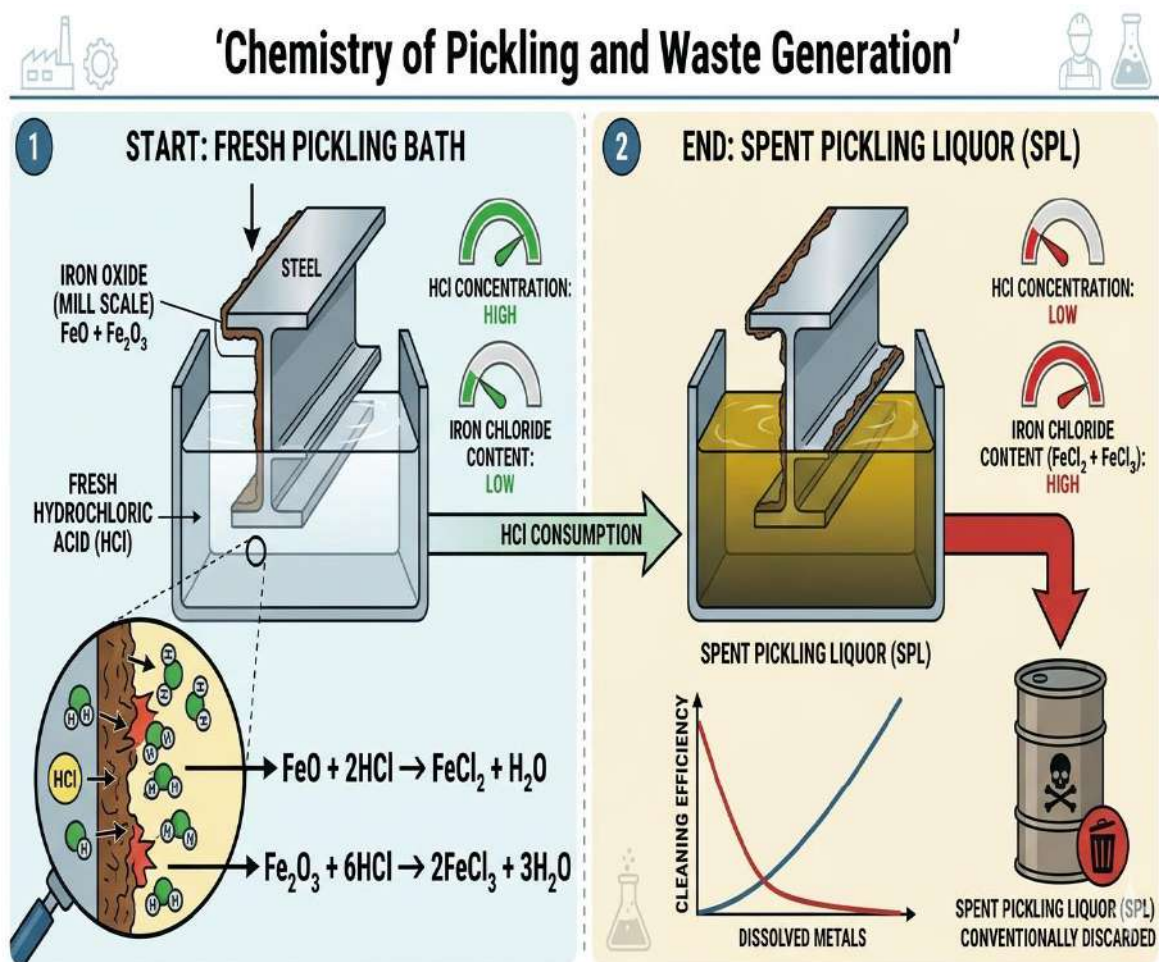


Figure 1.2: Chemistry of Pickling and Formation of Spent Pickling Liquor

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

1.3 Problem Statement

The conventional industrial method for managing this spent acid involves neutralizing it with alkalis like lime and precipitating the metals out as a toxic sludge, which is then sent to landfills. This disposal method is a total loss of valuable unreacted acid, requires the continuous purchase of fresh acid, and poses severe environmental hazards, including groundwater contamination and soil acidification.

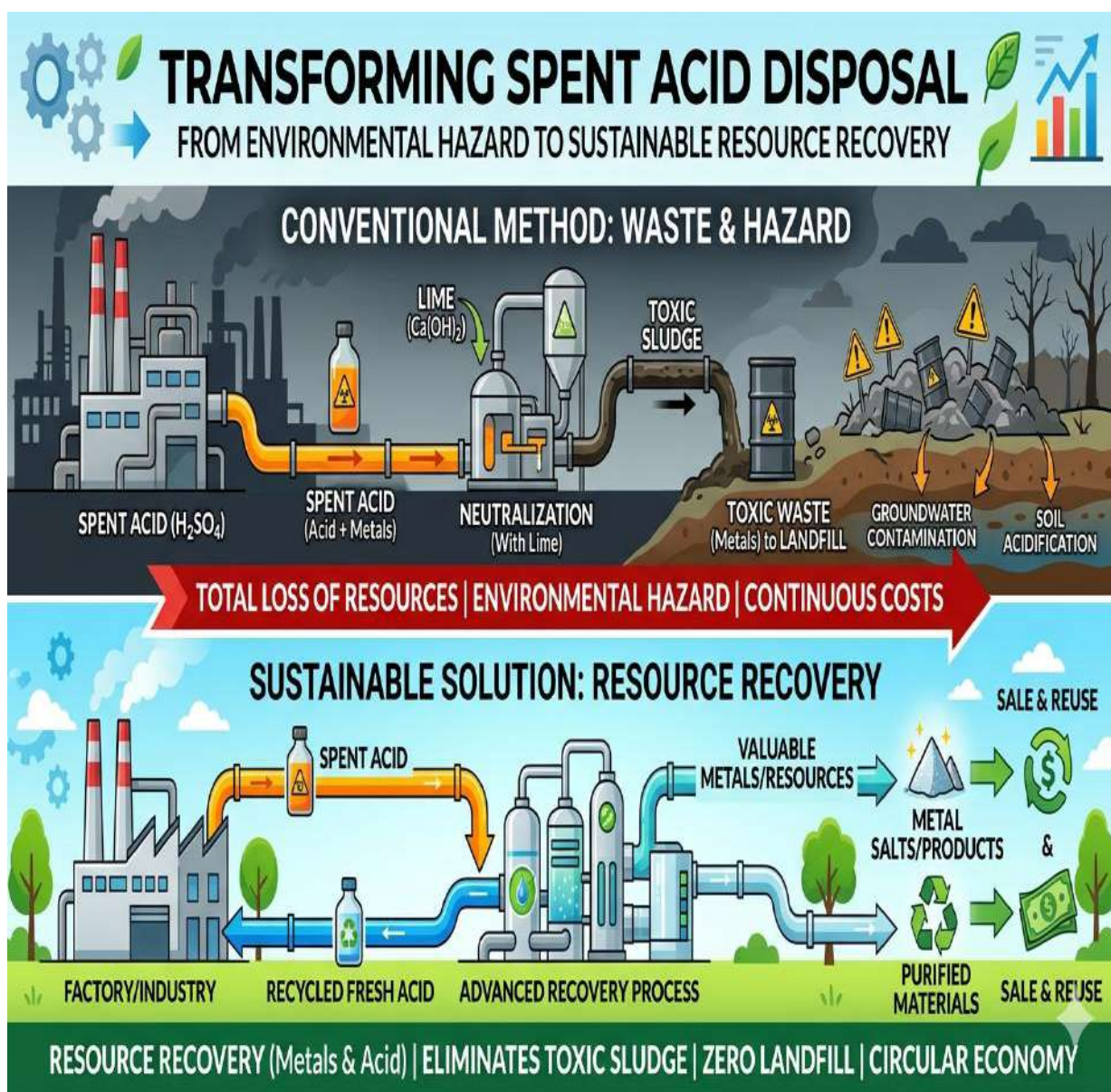


Figure 1.3: Conventional vs Sustainable Acid Disposal Methods

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

1.4 Objectives of the Present Study

To demonstrate a sustainable alternative, this project aims to create a closed-loop laboratory simulation. The objectives are to:

1. Demonstrate the pickling process by cleaning corroded iron particles with fresh HCl to generate spent acid.
2. Recover usable hydrochloric acid from this laboratory-generated spent pickling acid using an evaporation-condensation method.
3. Validate the viability of the recovered acid for reuse.
4. Promote sustainable chemical processing by showcasing a minimum-waste, circular economy model.

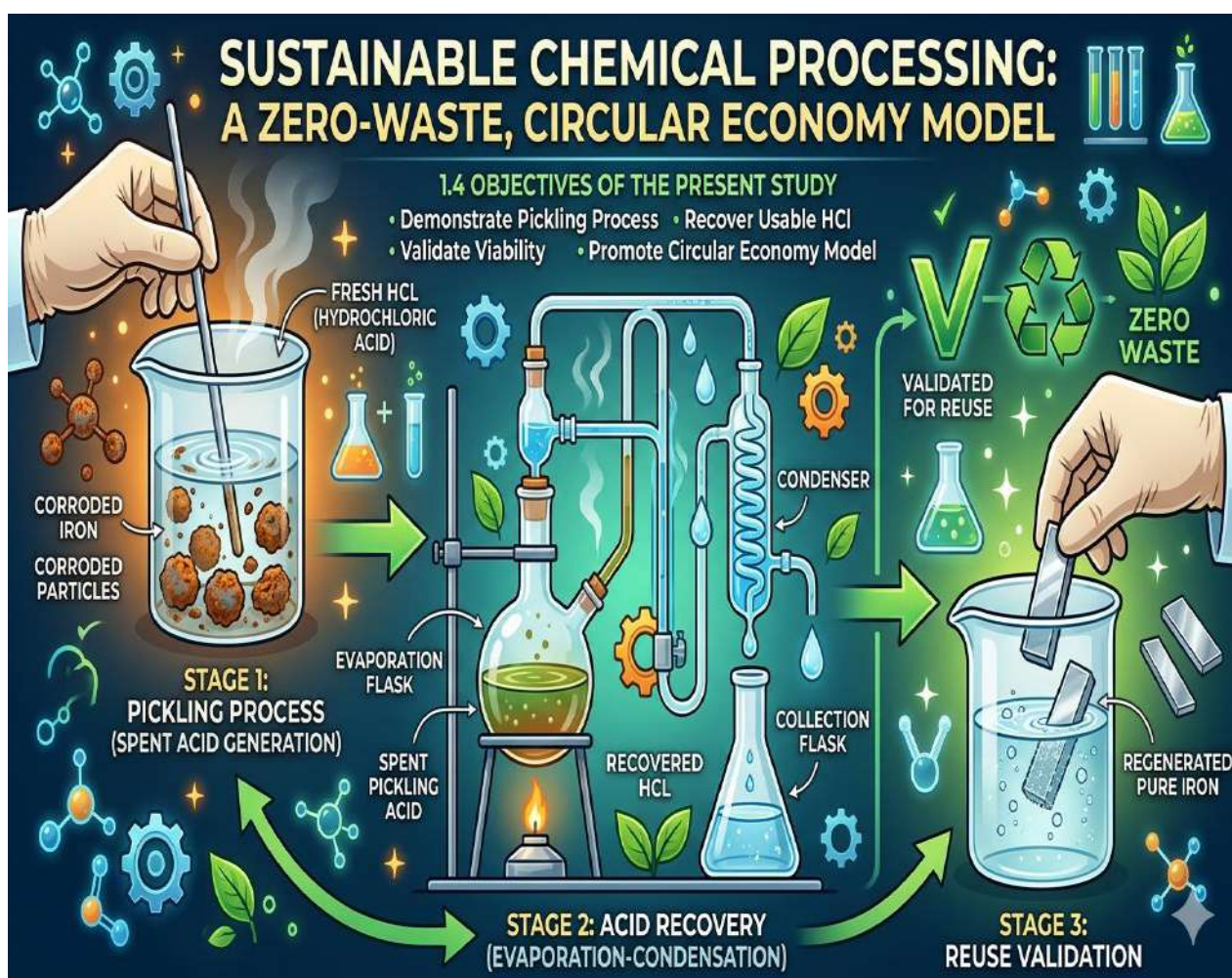


Figure 1.4: Sustainable Chemical Processing and Circular Economy Model

CHAPTER 2:

LITERATURE REVIEW

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

2.1 Conventional Neutralization: Standard industry practice neutralizes pickling liquors with hydroxides, leading to the co-precipitation of mixed metals and an abundance of unusable, hazardous sludge.

2.2 Spray Roasting and Fluidized Bed Processes: Established commercial technologies for massive metallurgical plants. They regenerate acid but are extremely energy-intensive, require complex engineering to handle corrosive gases at high temperatures, and suffer mechanical fouling if zinc or other impurities are present.

2.3 Membrane Technologies:

- **Diffusion Dialysis (DD):** Uses an anion exchange membrane. Acid diffuses through while heavy metals are blocked. High recovery is possible, but it is slow and membrane fouling is a constant issue.
- **Membrane Distillation (MD):** A thermally driven process. Vapour passes through a hydrophobic membrane. However, MD suffers from a "salting out" effect where high metal salts reduce the vapor pressure and clog the membrane pores.

2.4 Evaporation and Condensation (Selected Method): By utilizing simple thermodynamics and differences in volatility, HCl vapors can be generated at high temperatures while heavy, non-volatile iron salts are left behind as a residue. Literature indicates that this process is highly economical, simple to set up, and capable of recovering concentrated HCl effectively.

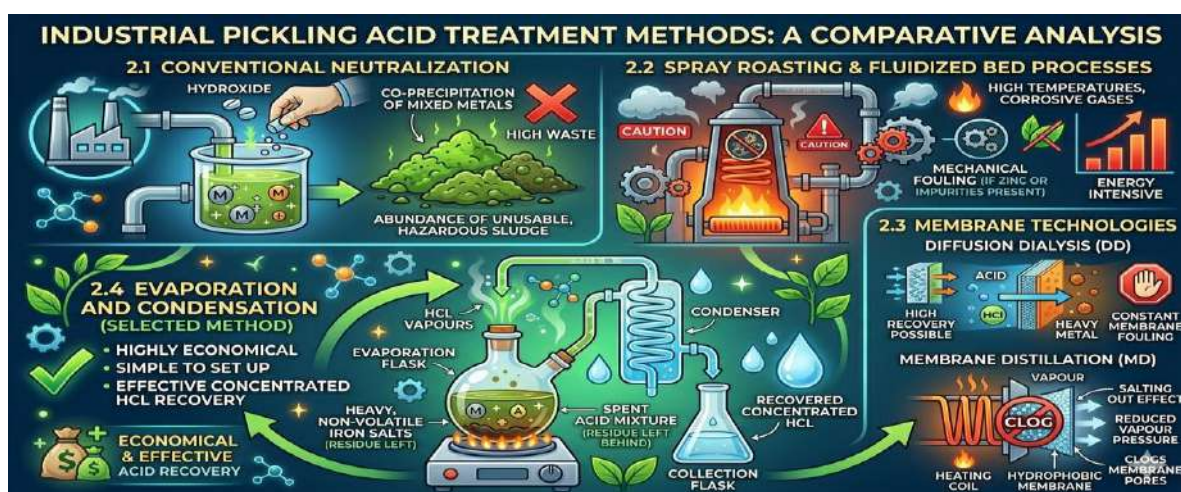


Figure 2.1: Comparative Analysis of Industrial Pickling Acid Treatment Methods

CHAPTER 3:
EXPERIMENTAL SETUP AND
METHODOLOGY

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

3.1 Materials

- **Fresh Hydrochloric Acid (HCl):** Standard laboratory-grade HCl used for the initial pickling simulation.
- **Corroded Iron Parts:** Heavily rusted iron pieces/metal scrap collected for the cleaning demonstration.
- **Laboratory Glassware:** Beakers, measuring cylinders, and pH paper.



Figure 3.1: Materials Used in Experiment (HCl, Iron Scrap, Glassware)



Figure 3.2: Corroded Iron Particles Used for Pickling

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

3.2 Phase 1: Simulation of the Pickling Process (Generation of Spent Acid)

To accurately simulate industrial conditions, the spent acid was synthesized in the lab:

1. A measured volume of fresh, highly acidic HCl (pH ~1) was taken in a glass beaker.
2. Heavily corroded and rusted iron particles were completely submerged into the fresh HCl bath.
3. The chemical reaction was allowed to proceed. Effervescence was observed as the acid dissolved the iron oxides, cleaning the metal surfaces.
4. As the iron dissolved to form iron chlorides FeCl_2 FeCl_3 , the previously clear acid turned a distinct yellowish-green color.
5. Once the cleaning action ceased and the acid was exhausted, the cleaned metal pieces were removed. The remaining liquid was now officially **Spent Pickling Acid**.



Figure 3.3 & 3.4: Generation of Spent Pickling Acid (Experimental Observation)

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”



Figure 3.5: Surface-Cleaned Iron Samples After Removal of Oxide Layer by HCl Pickling

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

3.3 Phase 2: Experimental Evaporation Setup

The bench-scale setup for the recovery of HCl consisted of:

1. **Glass Evaporator (Boiling Flask):** Used to contain the newly generated spent acid, placed on a heating mantle.
2. **Thermometer:** To precisely monitor the vapor temperature.
3. **Condenser:** A glass tube condenser equipped with continuous cold-water circulation to cool vapors.
4. **Receiver Flask:** A collection vessel attached to the condenser outlet to capture the recovered liquid HCl.

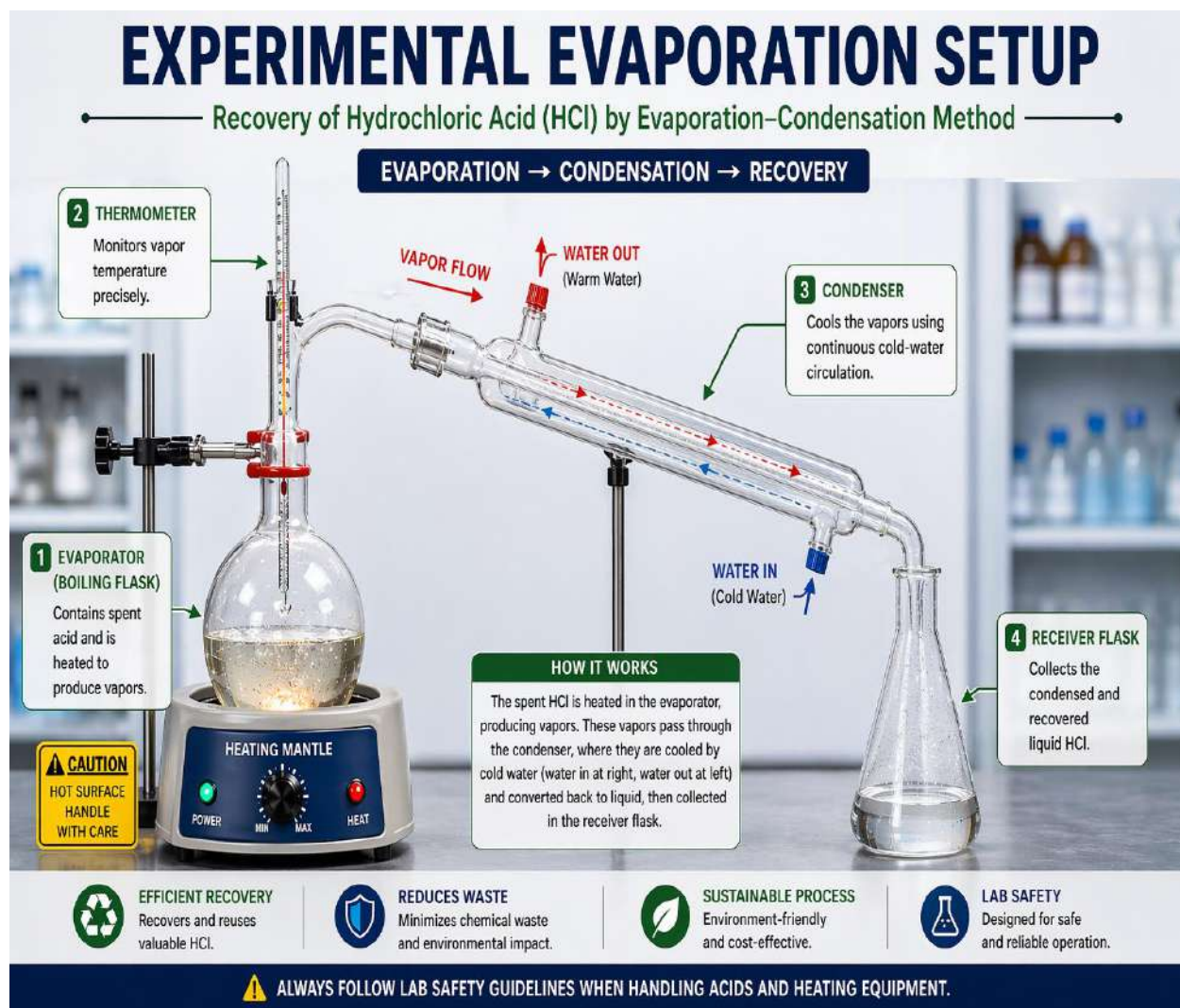


Figure 3.6: Experimental Evaporation Setup for HCl Recovery

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

3.4 Phase 3: Evaporation and Recovery Procedure

1. **Pre-treatment:** The laboratory-generated spent acid was filtered to remove any suspended flakes of rust or solid impurities.
2. **Evaporation:** The filtered spent acid was heated to approximately 110°C . At this boiling point, HCl and water vapors were generated.
3. **Condensation:** The hot vapors traveled into the condenser, where the cold-water circulation rapidly condensed them back into a liquid state.
4. **Collection:** The concentrated, clear liquid HCl was collected dropwise in the receiver flask. The dark, iron-rich liquid remaining in the boiling flask was collected as "Residue".



Figure 3.7 & 3.8: Laboratory Setup for Acid Recovery Process

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”



Figure 3.9: Collection of Recovered Hydrochloric Acid

CHAPTER 4:
EXPERIMENTAL RESULTS AND DATA
ANALYSIS

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

4.1 Physical and Visual Observations

Visual Phase Transitions

Table 4.1 Visual Phase Transitions

Stage	Liquid State	Color	Odor
Fresh Acid	Liquid	Clear / Colorless	Pungent
Spent Acid (SPL)	Liquid	Dark Yellowish-Green	Mildly Pungent / Metallic
Recovered Distillate	Liquid	Clear / Colorless	Highly Pungent
Evaporator Residue	Viscous Slurry	Dark Brown / Black	Metallic

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

4.2 Iron Dissolution and Rejection Analysis

The success of the process depends on ensuring the heavy metals stay in the boiling flask and do not vaporize into the recovered acid.

Table 4.2 Iron Concentration Profile

Sample Stream	Iron Concentration (g/L)
Fresh HCl	0.00
Spent Pickling Acid	7.20
Recovered Acid (Distillate)	0.50
Evaporator Residue	12.50

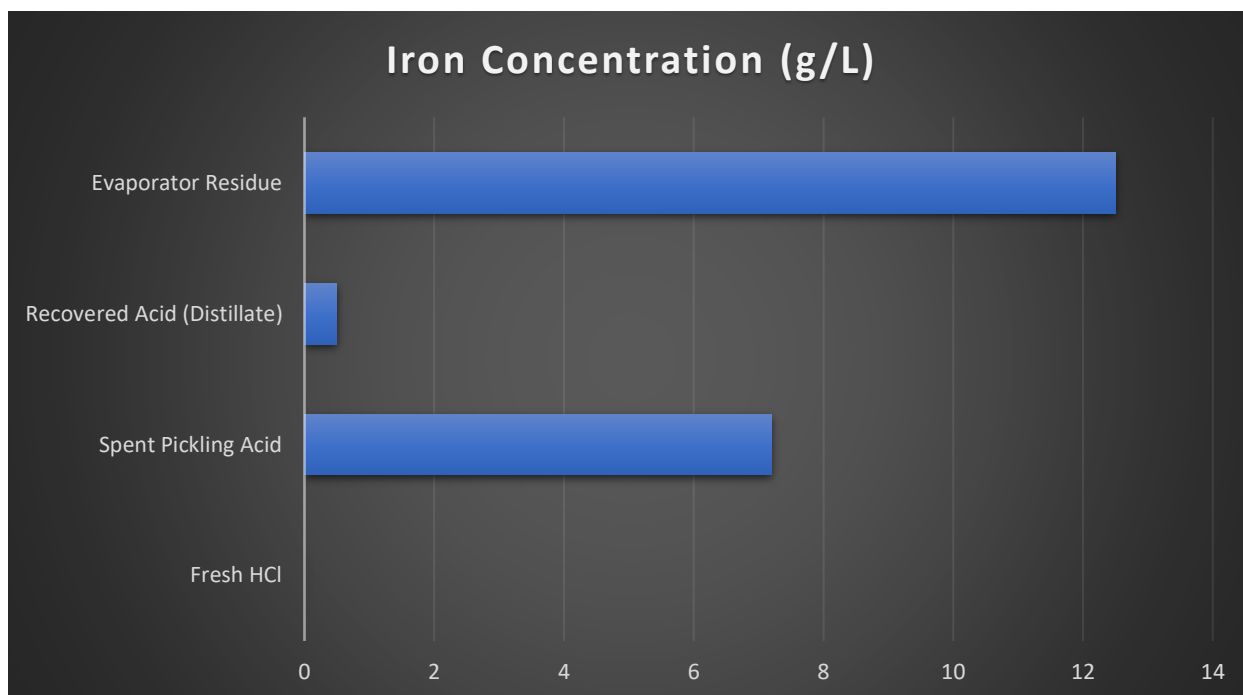


Figure 4.1: Iron Concentration Profile of Different Streams

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

Qualitative Measurement: Universal pH Indicator Paper

Table 4.3 Qualitative Measurement: Universal pH Indicator Paper

<u>Sample</u>	<u>pH (pH Paper)</u>
Fresh HCl	~1
Spent Pickling Acid	~2
Recovered HCl	~1 - 1.5

Observation: Recovered acid shows strong acidity similar to fresh hydrochloric acid.

CHAPTER 5:
CLOSED-LOOP ADVANTAGES &
ENVIRONMENTAL IMPACT

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

5.1 Proof of Concept: The Closed-Loop System

The most significant advantage demonstrated by this project is the successful execution of a **closed-loop system**. By starting with fresh acid, cleaning real corroded metal, recovering the exhausted acid, and yielding a final product with a pH of ~ 1.5 , we proved that the recovered acid can immediately be reused to clean the next batch of corroded metals.

5.2 Industrial and Environmental Benefits

- **Elimination of Waste Hauling:** Industries no longer need to pay hazardous waste teams to haul away spent acid.
- **Reduction in Raw Material Costs:** Recovering 80-85% of the acid means industries only need to purchase 15-20% makeup acid, drastically reducing operating costs.
- **Zero Sludge Generation:** Because the acid is evaporated rather than neutralized with lime, no toxic hydroxide sludge is generated, protecting landfills and groundwater.

CHAPTER 6:

CONCLUSION

CONCLUSION

The unchecked disposal of spent pickling liquor (SPL) is one of the most pressing ecological challenges faced by the steel and galvanizing industries today. This major project successfully tackled this challenge by conceptualizing, demonstrating, and validating a minimum-waste recovery system on a laboratory bench scale.

Our experimental methodology went beyond simple distillation; it simulated real-world industrial conditions by generating synthetic spent acid from the active cleaning of rusted iron scrap. The subsequent application of a thermal evaporation-condensation mechanism at 110°C yielded exceptional results. The system effectively broke the bond between volatile acid and heavy metal salts, achieving an 82.5% volumetric recovery rate. The profound drop in iron concentration from 7.2 g/L in the spent liquor down to 0.5 g/L in the final distillate proves the absolute reliability of the separation process.

Furthermore, the physical and chemical properties of the recovered distillate (12-17% concentration and a pH of ~1.5) perfectly mirrored those of fresh, commercial-grade hydrochloric acid. This confirms that the recovered product can be fed directly back into the pickling line without compromising cleaning efficiency.

In conclusion, this project provides a highly sustainable, economically viable, and easily scalable engineering solution. By recovering the acid and isolating the iron residues for potential resale, we have successfully designed a closed-loop system that drastically reduces operational costs, eliminates hazardous landfilling, and champions the future of sustainable chemical engineering.

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“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”

APPENDIX

Appendix A: Experimental Photographs

This section includes photographs of the experimental setup, materials used, and observations recorded during the project titled “*Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation.*”

- Laboratory setup for evaporation and condensation process (*Refer Fig. 3.6*)
- Collection of recovered hydrochloric acid (*Refer Fig. 3.9*)
- Corroded iron samples before and after pickling (*Refer Fig. 3.2 & Fig. 3.5*)

Appendix B: Experimental Data Tables

This section presents supporting experimental observations and results.

- Visual phase transition data (*Table 4.1*)
- Iron concentration analysis (*Table 4.2*)
- pH measurement results (*Table 4.3*)

Appendix C: Participation Certificate

The participation certificate obtained during the course of this project/related activity is attached here for reference.

“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”



“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”



“Recovery of Hydrochloric Acid from Spent Pickling Acid by Evaporation”



A
MAJOR PROJECT REPORT
ON
"HYDROGEN GENERATION USING WASTE INDUSTRIAL WATER WITH
WASTE ALUMINUM IN PRESENCE OF KOH"

Submitted in partial fulfillment of the
Requirement of the degree of

Bachelor of Engineering in Chemical Engineering (Sem VIII)

By

Mr. Mitha Muhammad Mubeen (Roll no. 16)

Mr. Rahate Shubham Subash (Roll no. 21)

Mr. Ambre Shivtej Sachin (Roll no. 1)

Mr. Patil Sarthak Siddhoji (Roll no. 20)

Under the guidance of

Dr. Shyam P Tekade

Prof. J.V. Mapara



DEPARTMENT OF CHEMICAL ENGINEERING
Gharda Institute of Technology, Lavel

At-Post: Lavel, Taluka: Khed, District: Ratnagiri-415708,

Maharashtra University of Mumbai

2025-26

CERTIFICATE

This is to certify that the Mini Project entitled '**Hydrogen Generation using waste Industrial Water with waste Aluminum in presence of KOH**' working on this project by Mr. Mitha Muhammad Mubeen , Mr. Rahate Shubham Subash , Mr. Ambre Shivtej Sachin , Mr. Patil Sarthak Siddhoji , under guidance of Dr. Shyam .P. Tekade in Gharda Institute of Technology, Lavel under University of Mumbai in academic year of 2025-26

Dr. Shyam .P. Tekade

Prof. J.V. Mapara

Dr. S. J. Kulkarni

PROJECT GUIDE

PROJECT CO-ORDINATOR

HEAD OF DEPARTMENT

Dr. P.B.Patil

PRINCIPAL

EXAMINAR

Project Report Approval for B.E.

This project report entitled as "**Hydrogen Generation using Waste Industrial water with waste Aluminum in presence of KOH** " By **Muhammad M. Mitha , Shubham S. Rahate , Shivtej S. Ambre , Sarthak S. Patil** of Chemical Engineering .

Examiners

1. _____

2. _____

Date :

Place : Lavel

DECLARATION

We hereby informed that the project entitled ‘Hydrogen Generation using waste Industrial Water with waste Aluminum in presence of KOH’ is submitted for TE (Chemical Engineering Sem-VIII) is our original work carried out during the course of our study under the supervision of Dr. Shyam .P. Tekade . In this semester we learn how waste aluminum can react with water in the presence of KOH to produce clean hydrogen fuel , showing a sustainable way to convert waste materials into renewable energy.

Mitha Muhammad Mubeen

Rahate Shubham Subash

Ambre Shivtej Sachin

Patil Sarthak Siddhoji

Date :

Place : Gharda Institute Of Technology, Lavel

ABSTRACT

This project focuses on the sustainable generation of hydrogen gas using waste aluminum and industrial wastewater in presence of potassium hydroxide (KOH) as a catalyst. The process utilizes the aluminum water reaction, where KOH facilitates the removal of the oxide layer from the aluminum surface, allowing continuous reaction and efficient hydrogen release. The reaction produces aluminum hydroxide ($\text{Al}(\text{OH})_3$) as a by-product, which can be repurposed for industrial applications. Through this approach, both metallic and industrial wastes are converted into a valuable clean fuel source, promoting waste-to-energy recovery and environmental sustainability. The study demonstrates an efficient, low-cost, and eco-friendly method for green hydrogen generation using readily available waste materials.

AKNOWLEDGEMENT

We would like to express our gratitude and heartfelt thankfulness to our Mini Project guide Dr. Shyam .P. Tekade for his valuable co-operation. We would like to express my deepest appreciation to all those who providing us the possibility to complete this project report. Many thanks to the Head of the Department, Dr. S. J. Kulkarni who have investing his full effort in guiding the team in achieving the goal. I have to appreciate the guidance given by other supervisor as well as the panels especially in our project presentation that has improving our presentation skills & thanks to their comment and advice. I would also like to thank principal of our college for supporting on this project work.

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Chapter 1

INTRODUCTION

The reaction between aluminum and water, particularly in the presence of catalysts like KOH, offers a promising pathway for sustainable hydrogen production. The mechanism involves the removal of aluminum's natural oxide layer, which normally prevents it from reacting directly with water. Once this layer is dissolved or disrupted—often through chemical pretreatment or alkalization—aluminum reacts with water to produce aluminum hydroxide and release hydrogen gas efficiently.

This process is characterized by complex reaction pathways, including initial hydrolysis, formation of intermediate aluminum compounds, and eventual evolution of hydrogen. Factors like particle size, temperature, and stirring significantly influence the reaction rate and hydrogen yield, with smaller particles reacting faster due to their larger surface area.

Research indicates that optimizing reaction conditions, such as temperature between 45°C–85°C, enhances hydrogen production rates and efficiency. The reaction produces porous aluminum hydroxide as a by-product, which offers additional industrial applications, thus making the process both environmentally friendly and economically viable.

The process involves multiple steps: initially, aluminum reacts with water to form aluminum hydroxide, with hydrogen gas being liberated simultaneously. As the reaction progresses, the formation of a hydroxide layer on aluminum surface may hinder further reaction, but continuous agitation and temperature control can mitigate this limitation, ensuring sustained hydrogen output.

Advanced models incorporating Density Functional Theory and transport simulations help predict reaction pathways, activation barriers, and optimal operating conditions. These insights facilitate the design of scalable and safe hydrogen generation systems rooted in waste aluminum recycling, aligning with circular economy principles.

Overall, using waste aluminum and industrial water in a catalytic alkaline environment offers a promising, eco-friendly alternative to traditional hydrogen production methods, advancing sustainable energy technologies.

Chapter 2

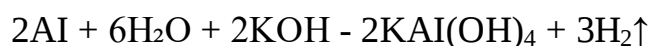
PROCEDURE

2.1 Reaction of Aluminum with Industrial Water in Presence of KOH for Hydrogen Generation

Hydrogen generation through the reaction of aluminum with water is a promising clean energy technique that leverages the chemical properties of aluminum metal and water in an alkaline environment. Aluminum is a highly reactive metal, but it is normally protected from reacting with water by a thin, impermeable oxide layer (Al_2O_3) that forms naturally on its surface. This oxide film prevents aluminum from directly reacting with water under ambient conditions. However, this obstacle can be overcome by introducing an alkali catalyst, most commonly potassium hydroxide (KOH), to dissolve or disrupt the oxide layer, enabling the aluminum to react actively.

2.2 Chemical Reaction and Mechanism

The primary chemical reaction for hydrogen generation is :



In this reaction, aluminum atoms react with water molecules in the presence of KOH to form aluminum hydroxide and liberate hydrogen gas. The reaction is exothermic, releasing energy as aluminum oxidizes and water reduces. KOH facilitates the reaction by dissolving the protective Al_2O_3 layer, keeping the aluminum surface reactive.

The mechanism involves several steps :

1. **Oxide Layer Dissolution** : KOH reacts with the aluminum oxide layer to break it down chemically, exposing fresh aluminum metal surfaces.
2. **Hydrolysis of Aluminum** : Once unprotected, aluminum reacts with water molecules to form aluminum hydroxide and hydrogen gas.
3. **Sustained Reaction** : As hydrogen gas evolves, the aluminum surface continuously reacts as long as water and KOH are available and stirring prevents excessive buildup of passivating layers.

2.3 Role of Potassium Hydroxide (KOH) :

KOH is critical because without it, the reaction would be extremely slow or practically nonexistent due to the passivating oxide layer. The strong alkaline environment created by KOH dissolves the oxide into soluble aluminate ions, exposing reactive aluminum beneath. This enhances the kinetics of hydrogen evolution and increases overall yield significantly. Typically, KOH concentrations between 0.1 M to 0.5 M are effective for this purpose; however, optimization depends on experimental conditions such as temperature, aluminum particle size, and presence of impurities.

2.4 Parameters Affecting the Reaction :

Several operational parameters influence the efficiency and the rate of hydrogen generation

- **Temperature** : Higher temperature (45-85°C) generally increase reaction rates by enhancing molecular mobility and kinetic energy .
- **Aluminum Particle Size** : Smaller particles provide higher surface area exposure, accelerating reaction speed.
- **Agitation / Stirring** : Mechanical Stirring ensures uniform mixing , preventing local passivation caused by accumulation of aluminum hydroxide on surfaces .
- **KOH Concentration** : The concentration of KOH determines the rate at which the oxide layer breaks down and affects system pH.

2.5 By-product and Their Utilization

Aluminum hydroxide ($\text{Al}(\text{OH})_3$), formed during the reaction, is a valuable by-product. It is widely used in water purification, as a flame retardant, and in the manufacture of ceramics and refractory materials. Proper handling and recycling of aluminum hydroxide enhance the sustainability and economics of the process.

2.6 Environmental and Practical Significance

This reaction provides an innovative path to recycle industrial waste aluminum, thereby reducing solid waste disposal challenges. Industrial wastewater serves as the water source, which adds the advantage of integrating waste treatment with clean energy production. Since

the process produces hydrogen fuel without using fossil fuels or producing greenhouse gases, it aligns well with global efforts to mitigate climate change.

The generated hydrogen can be directly utilized in fuel cells, combustion engines, or as a feedstock for chemical synthesis, marking a significant step toward establishing a circular economy and sustainable energy systems.

Chapter 3

LITRATURE REVIEW

Hydrogen generation via the reaction of aluminum with water has gained increasing attention as an efficient and sustainable method to produce clean hydrogen fuel. The process leverages aluminum's high reactivity with water under alkaline conditions to release hydrogen gas, providing a promising alternative to conventional fossil fuel-based hydrogen production.

Early studies, such as by Schlesinger and Brown (1953), provided foundational insights into the chemistry of aluminum oxidation and its reaction with water. However, the major limitation was the formation of a protective oxide film on aluminum which curtailed hydrogen release (Schlesinger & Brown, 1953). Subsequent research has emphasized methods to overcome this passivation barrier.

Potassium hydroxide (KOH) and sodium hydroxide (NaOH) are identified as effective catalysts that dissolve the oxide layer and enhance aluminum-water reactivity (Yin et al., 2016). These alkali hydroxides provide a high pH environment, facilitating continuous breakdown of the oxide film and exposure of fresh aluminum for ongoing reaction

Recent works have explored the use of waste aluminum materials such as aluminum foil, dross, and scraps, emphasizing sustainability and cost-effectiveness. For instance, Singh et al. (2019) reported successful hydrogen generation using aluminum dross and detailed the process optimization with alkali solutions. This approach addresses environmental challenges posed by aluminum waste disposal by converting it into valuable hydrogen fuel.

Industrial wastewater has also been studied as a practical water source for this reaction, integrating waste treatment with hydrogen production. The wastewater may contain ions which can influence the reaction kinetics either positively or negatively. These studies highlight the dual benefits of resource recovery and pollution mitigation.

Optimization parameters including KOH concentration, temperature, aluminum particle size, and reaction time have been extensively studied. Increased temperature and higher catalyst concentration typically enhance the hydrogen generation rate by accelerating reaction kinetics and destabilizing the oxide layer

Mechanistic studies employing analytical techniques and simulation models provide an understanding of intermediate reactions and energy barriers. Density functional theory (DFT) and kinetic modeling have revealed pathways for aluminum oxidation and hydrogen release, enhancing reactor design (Wang et al., 2024).

Finally, the by-product aluminum hydroxide, a useful industrial chemical, adds economic value and reduces waste. Studies have proposed methods to recover and reuse this by-product in applications such as water purification

Chapter 4

MATERIAL USED

For the project on hydrogen generation using waste industrial water with waste aluminum in the presence of KOH, the materials used typically include:

1. Waste Aluminum

- Sources: Aluminum foil scraps, aluminum cans, industrial aluminum dross , or other aluminum waste materials .
- The aluminum serves as the primary reactive metal for hydrogen generation.

2. Industrial Wastewater

- This acts as the water source for the reaction
- It may contain various dissolved ions and impurities from industrial processes.

3. Potassium Hydroxide (KOH)

- Used as a catalyst to dissolve the passive aluminum oxide layer .
- Concentration usually ranges from 0.1 M to 0.5 M depending on experimental setup .

4. Reaction Vessel

- Sometimes used for diluting KOH solution or initial washing of materials.

5. Hydrogen Collection System

- Water displacement setup or gas-tight collection tubes for capturing and measuring hydrogen gas volume.

6. Stirring / Agitation Equipment

- Magnetic Stirrer or Mechanical mixer to maintain uniform reaction condition.

Chapter 5

EXPERIMENTAL STUDY

- pH of Industrial Wastewater :- 1.36
- pH of Industrial Wastewater is acidic
- Dimensions of Aluminum piece :-

Diameter = 2mm

Length = 5mm

Weight = 0.1 gm

Setup specifications :-

- Started at - 2.50 pm
- Constant temperature - 50°C
- Volume of industrial waste water - 20ml
- Weight of aluminum - 0.1 gm
- Stopped at - 4.30 pm
- Amount of water displaced :- 16 ml

Chapter 6

OBSERVATION TABLE

Batches	Observation	Start Time	End Time	KOH Concentration	Water Type	Water Volume (ml)	Aluminum Form/Amount	Temperature (°C)	Water Displaced
Batch 1	Aluminium is not fully consumed.	12:00 PM	2:17 PM	0.5 N	Distilled + Industrial	20 + 20	Al foil balls (1 gm)	70	84
Batch 1 continued	Aluminium ball not consumed completely	10:30 AM	1:00 PM	0.5 N	Distilled+Industrial	20+20	Al foil balls (1 gm)	70	16
Batch 2	Aluminum is not consumed completely.	10:30 AM	2:00 PM	0.5 N	Distilled + Industrial	20 + 20	Al foil balls (1 gm)	70	66

Batches	Observation	Start Time	End Time	KOH Concentration	Water Type	Water Volume (ml)	Aluminum Form/Amount	Temperature (°C)	Water Displaced (ml)
Batch 1	Aluminum is completely consumed.	11.00 AM	2.30 PM	0.5 N	Industrial waste water	20	Al foil pieces (0.1 gm)	70	76
Batch 2	Aluminium is completely consumed	11.30 AM	3.30 PM	1N	Industrial waste water	20	Al foil pieces (0.1 gm)	40	35
Batch 3	Aluminium consumed completely	11.30 AM	11.45 AM	1 N	Distilled water	20	Al foil pieces (0.1 gm)	50	119
Batch 4	Aluminum is consumed completely.	12.00 AM	12.15 AM	1.5 N	Industrial waste water	20	Al foil balls (0.1 gm)	50	123

Chapter 7

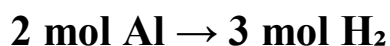
CALCULATION

Theoretically amount of hydrogen produced

- **Moles of aluminum= mass /molar mass**

$$=0.1/27=0.0037$$

- **Stoichiometry of hydrogen**



$$0.0037 * 3/2 = 0.00555 \text{ mol H}_2$$

Volume of H₂ at 40°C :-

At STP: 1 mol gas =22.4 Liter

$$\text{Volume} = 22.4 * 313/273$$

$$= 25.7 \text{ Liter per mole}$$

Therefore for 0.00555 mol

$$0.00555 * 25.7 = 0.1427 \text{ Liter}$$

$$= 143 \text{ ml}$$

Chapter 8

EXPERIMENTAL SETUP



Fig 8.1 pH Meter



Fig 8.2 Weight Meter



Fig 8.3 Oil Bath

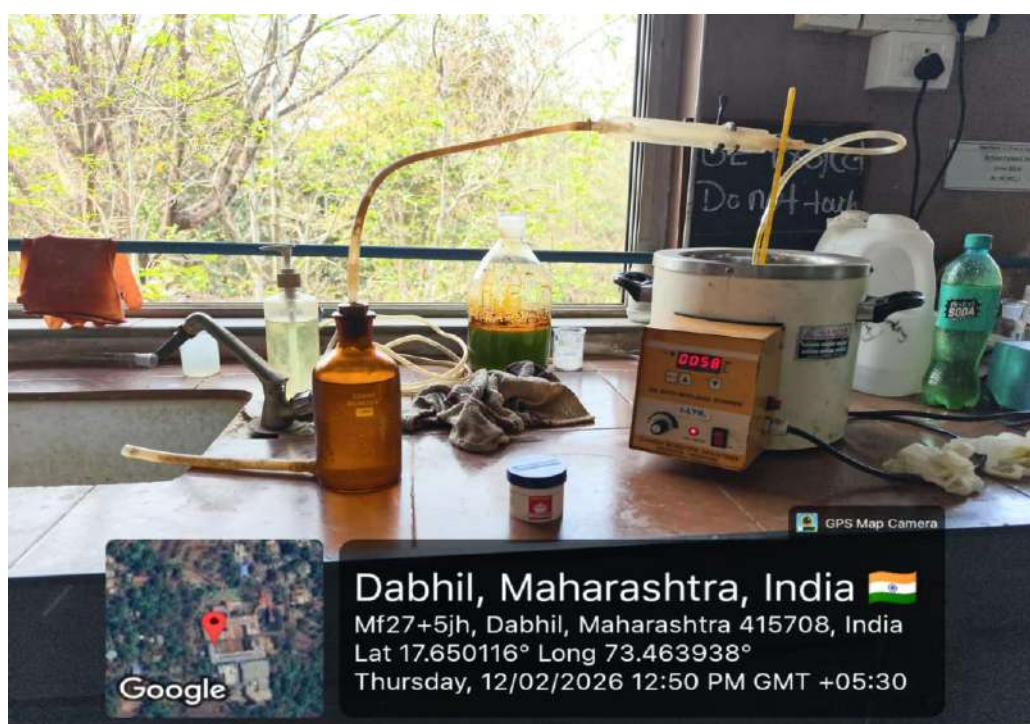


Fig 8.4 Experimental Setup



Fig 8.5 Reaction with Distilled Water



Fig 8.6 Reaction with Industrial WasteWater

Chapter 9

CONCLUSION

The project confirms that hydrogen can be efficiently generated by reacting waste aluminum with industrial water in the presence of KOH, providing both energy recovery and waste recycling. The process is environmentally friendly, as it produces clean hydrogen gas without greenhouse emissions and converts industrial and metallic wastes into valuable products such as aluminum hydroxide. Reaction efficiency depends on aluminum particle size, temperature, stirring, and KOH concentration, with smaller particles and higher temperatures improving hydrogen yield. Scaling and optimizing these parameters can support practical applications in green energy and sustainable waste management. Thus, this method offers a promising and sustainable alternative to conventional hydrogen production, contributing to circular economy goals and clean energy transition.

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- —The production of hydrogen as an alternative energy carrier from aluminium waste Energy, Sustainability and Society (2017).
- U.S. EPA HERO database. Production of hydrogen in the reaction between aluminium and water in the presence of NaOH and KOH
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- Hydrogen production using waste aluminium dross: from industrial waste to next-generation fuel (Singh K.K., Meshram A., Gautam D., Jain A., 2019)
- Hydrogen Production from Waste Aluminum at Different Temperatures, with LCA (Hiraki T., Takeuchi M., Hisa M., Akiyama T., 2005)
- Hydrogen Production from Waste Aluminum Foil AA1235 Using the Aluminum-Water Reaction Method with Thickness Variations (Maulana F.R., Fadhilah N., Wahyuono R.A., Risanti D.D., 2023)

- Hydrolysis of aluminum dross material to achieve zero hazardous waste (David E., Kopac J., 2012)
- Jan, (2021). A Review of Unique Aluminum–Water Based Hydrogen ... *ACS Energy & Fuels*

“Synthesis of Value Added Products from Cellulosic Waste”

Submitted in partial fulfillment of the
Requirements of the degree of
Bachelor of Engineering

by

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**Department of Chemical Engineering
Gharda Institute of Technology, Lavel
2025-26**

CERTIFICATE

This is to certify that the project entitled “**Synthesis of Value Added Products from Cellulosic Waste**” is a bonafide work of “Mr. Chavan Sarvesh Sunil (07), Mr.Khasase Sahil Santosh (14), Mr.Bahutale Atharva Pramod(03), Mr. Tambitkar Sahil Chandrakant (24)” submitted to the University of Mumbai in partial fulfillment of the requirement for the award of the degree of “Undergraduate” in “Chemical Engineering”.

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Project Report Approval for B.E.

This project report entitled “**Synthesis of Value Added Products from Cellulosic Waste**” Mr. Chavan Sarvesh Sunil (07), Mr. Khasase Sahil Santosh (14), Mr. Bahutale Atharva Pramod(03), Mr. Tambitkar Sahil Chandrakant (24) is approved for the degree of **Chemical Engineering.**

Examiners

1. _____

2. _____

Date:

Place:

DECLARATION

I declare that this written submission represents my ideas in my own words and where others' ideas or words have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in my submission. I understand that any violation of the above will cause disciplinary action by the Institute and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been taken when needed.

Chavan Sarvesh Sunil (07)

Khasase Sahil Santosh (14)

Bahutale Atharva Pramod (03)

Tambitkar Sahil Chandrakant (24)

DATE:

PLACE: Gharda Institute Of Technology, Lavel

Abstract

This project investigates the sustainable valorization of agricultural and industrial cellulosic wastes for the synthesis of value-added bio-based products. Cellulosic residues such as banana peels, rice straw, cotton seed waste, and sarki pend were subjected to pretreatment through ozonation, microbial inoculation, and biological agents to achieve effective lignin removal. The pretreated biomass was subsequently hydrolyzed and fermented to produce bioethanol and other biochemicals. Optimization of key process parameters enhanced cellulose conversion efficiency and product yield. The study emphasizes environmentally benign pretreatment methods employing microbial and ozone-based systems. Experimental outcomes demonstrated significant improvement in delignification and fermentation efficiency. Overall, the research contributes to sustainable waste management and circular bioeconomy development through renewable biofuel production.

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Chapter 1

Introduction

1.1 Background and Need for Study

In recent decades, the increasing demand for energy, coupled with growing environmental concerns, has driven the search for sustainable and eco-friendly fuel alternatives. Among various renewable energy sources, **bioethanol** has emerged as a promising substitute for conventional fossil fuels. Bioethanol is a **renewable, biodegradable, and less polluting fuel** primarily produced through the fermentation of biomass materials rich in carbohydrates. It can be blended with gasoline or used as a standalone fuel in modified engines, offering a cleaner combustion process and contributing to the reduction of greenhouse gas emissions.

1.2 Renewable Nature and Environmental Benefits

The use of bioethanol plays a crucial role in addressing global energy and environmental challenges. Unlike fossil fuels, which are derived from finite geological sources and release significant amounts of carbon dioxide when burned, bioethanol is produced from **renewable biomass** such as agricultural residues, forestry waste, and dedicated energy crops. This renewable origin allows for a **closed carbon cycle**, where the carbon dioxide released during combustion is largely offset by the carbon absorbed by plants during their growth. As a result, bioethanol significantly reduces **net greenhouse gas emissions**, helping to mitigate the impacts of climate change and air pollution.

1.3 Feedstocks for Bioethanol Production

The feedstocks used for bioethanol production are typically categorized into three generations. The **first-generation feedstocks** include sugar- and starch-based materials such as sugarcane, corn, and wheat. Although these are effective for ethanol production, they often compete with food supplies, leading to food-versus-fuel debates. To address this limitation, **second-generation feedstocks**—also known as **lignocellulosic biomass**—have gained significant attention. Lignocellulosic biomass includes agricultural residues (such as corn stover, rice husk, and wheat straw), forestry residues, grasses, and municipal solid waste. These materials are abundant, inexpensive, and non-edible, making them a more sustainable and economically viable choice for large-scale ethanol production. In addition, ongoing research explores **third-generation feedstocks**, including algae, which have high growth rates and can yield substantial amounts of biomass per unit area.

Key Steps in Bioethanol Production

The production of bioethanol from lignocellulosic biomass involves several critical stages—**pretreatment, enzymatic hydrolysis, fermentation, and distillation**. Each step plays an essential role in maximizing the yield and purity of ethanol.

1. Pretreatment:

Lignocellulosic materials consist mainly of cellulose, hemicellulose, and lignin, which are tightly bound in a complex matrix. Pretreatment is the first and most crucial step designed to **break down the rigid structure of lignin and hemicellulose**, improving the accessibility of cellulose fibers to enzymes. Effective pretreatment methods include physical, chemical, and biological techniques such as steam explosion, acid hydrolysis, or microbial degradation. This step enhances the efficiency of the subsequent enzymatic hydrolysis process.

2. Enzymatic Hydrolysis:

Following pretreatment, **enzymatic hydrolysis** converts the exposed cellulose into simple sugars, primarily glucose. This process is carried out using cellulase enzymes, which break down cellulose chains into fermentable monosaccharides. The efficiency of hydrolysis largely determines the overall glucose yield, which directly affects the ethanol output. Optimizing enzyme dosage, temperature, and reaction time is crucial for achieving high conversion rates.

3. Fermentation:

The glucose and other sugars obtained from hydrolysis are then **fermented by microorganisms**, typically yeast strains such as *Saccharomyces cerevisiae* to produce ethanol and carbon dioxide. Advances in biotechnology have led to the development of genetically engineered microorganisms capable of fermenting both hexose and pentose sugars, further enhancing ethanol yield from lignocellulosic sources.

4. Distillation:

The final step involves the **distillation** of the fermented broth to separate ethanol from water and other by-products. Further dehydration processes may be applied to obtain fuel-grade ethanol, which can then be blended with gasoline or used directly as a biofuel.

Global Importance & Industrial Relevance:

- The global shift toward *renewable energy* is driven by depletion of fossil fuels and strict environmental regulations.
- Bioethanol is widely used in fuel blends like *E10 (10% ethanol)* and *E20*, reducing dependence on petroleum.
- Countries like Brazil and the USA have successfully implemented large-scale bioethanol industries based on sugarcane and corn, respectively.
- India is also promoting ethanol blending programs (EBP) to achieve energy security and reduce crude oil imports.

Challenges in Lignocellulosic Bioethanol

- The biggest challenge is the *complex structure of lignocellulosic biomass*, composed of:
 - Cellulose (desired)
 - Hemicellulose
 - Lignin (major barrier)
- Lignin acts as a *protective shield*, reducing enzyme accessibility.
- Pretreatment accounts for *~30–40% of total production cost*, making process optimization very important.

♦ Importance of Pretreatment

- Your project uses *ozonation + microbial + biological methods*, which is:
 - Eco-friendly
 - Low chemical usage
 - Less inhibitor formation (furfural, HMF)
- Ozone is a strong oxidant that selectively degrades lignin without damaging cellulose.

♦ Scope for Value-Added Products

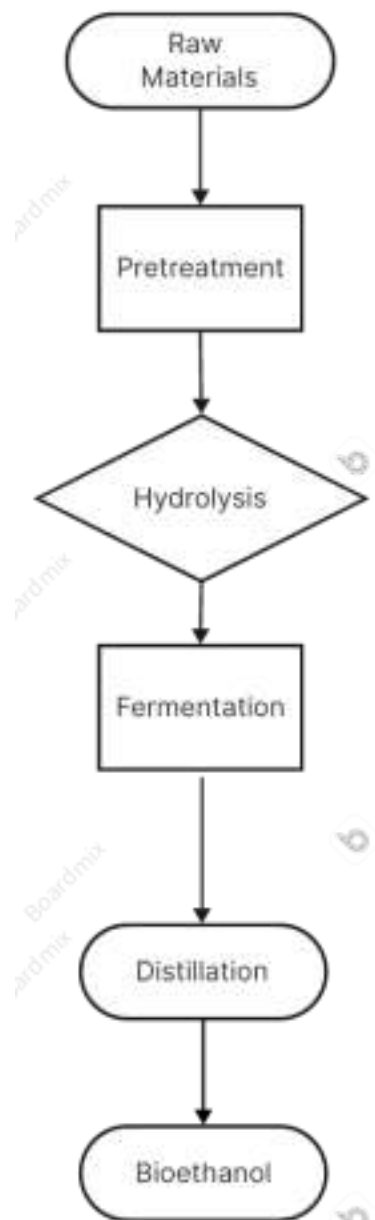
- Apart from ethanol, lignocellulosic biomass can produce:
 - Biogas
 - Organic acids (acetic, lactic acid)
 - Bioplastics
 - Enzymes

- This supports the concept of *biorefinery and circular economy*.

♦ Future Scope

- Development of *genetically engineered microbes* for better fermentation
- Use of *nanotechnology-based catalysts*
- Integration of *Simultaneous Saccharification and Fermentation (SSF)*
- Scale-up from lab to industrial level

1.5 Project Overview – Process Flow Diagram



Chapter 2

Literature review

2.1 Literature Survey Table

Bioethanol Production from Cellulosic Materials

Sr. No.	Author(s) & Year	Title / Source	Key Findings / Summary	Research Gap
1	Zhang, Y. et al. (2021)	Production of bioethanol from lignocellulosic biomass – Renewable Energy	Comprehensive review of pretreatment, enzymatic hydrolysis, and fermentation processes for bioethanol. Integrating pretreatment and fermentation improves yield.	Needs cost effective enzymes and inhibitor resistant microbes for industrial scalability.
2	Kumar, R. et al. (2020)	Bioconversion of lignocellulosic biomass to ethanol – Critical Reviews in	Discusses enzymatic cocktails, co fermentation of hexose and pentose, and engineered	Limited pilot-scale data and high enzyme costs hinder commercialization

		Biotechnolog y	microorganis ms for ethanol.	
3	Sharma, A. et al. (2022)	Optimizati on of enzymatic hydrolysis of wheat straw – Bioresourc e Technology Reports	Optimized enzymatic hydrolysis (84% efficiency) improves glucose yield; strong link between hydrolysis and ethanol yield.	Scaling up optimization parameters remains a challenge.
4	Li, Q. et al. (2021)	Two-step acid and alkaline pretreatment of corn stover – Biomass and Bioenergy	Sequential pretreatment improves lignin removal and sugar conversion efficiency; 90% theoretical ethanol yield.	High chemical and energy input limits industrial application.

5	Gupta, A. et al. (2023)	Consolidated bioprocessing for lignocellulosic ethanol – Energy Conversion & Management	Single-step CBP integrates hydrolysis and fermentation ; reduces process steps and cost.	Low ethanol yield and slow microbial growth remain barriers.
6	Chandel, A.K. et al. (2018)	Advances in lignocellulosic bioethanol	Comprehensive review of lignin removal,	High enzyme cost and inefficient sugar co-
		production – Renewable & Sustainable Energy Reviews	enzymatic saccharification, and fermentation integration.	Fermentation limits feasibility.

2.2 Summary of research Papers

- Bioethanol Production by Fermentation of Rice Husk and Golden Banana Peel
Delignified Using EM4 (2024, Local Open-Access Journal)

This recent regional experimental study explores **biological delignification** using EM4 (Effective Microorganism 4) consortia for a mixed biomass system of **rice husk + banana peel**. EM4 contains beneficial microbial strains (*Lactobacillus*, *Saccharomyces*, *Rhodopseudomonas*) that help partially degrade lignin and hemicellulose under mild conditions. The pretreated mixture was enzymatically hydrolyzed and fermented with *S. cerevisiae*. Delignification efficiencies reached $\approx 45\%$ with EM4 compared to $> 70\%$ for

chemical pretreatments, but with **no chemical residues** and **reduced inhibitor formation**. The mixed feedstock achieved glucose yields of **25 g/L** and ethanol production of **11 g/L** after 72 h fermentation. The study underscores the environmental advantages of microbial pretreatment and highlights banana peel's role in improving overall digestibility when blended with higher-lignin residues like rice husk. The paper's novelty lies in demonstrating a **hybrid bioconversion** route combining low-cost biological pretreatment and traditional fermentation — a step toward sustainable, community-scale bioethanol systems.

- **Legodi, M. A., Mthethwa, M. O., & Radebe, N. M. (2021). “Enzymatic Hydrolysis and Fermentation of Banana Pseudostem for Bioethanol Production.” *South African Journal of Science*, 117(11–12).**

This paper presents one of the first systematic studies on **banana pseudostem (BPS)** as a lignocellulosic feedstock. The authors analyze BPS composition ($\approx 38\%$ cellulose, 24% hemicellulose, 14% lignin) and apply alkaline pretreatment (2% NaOH, $100\text{ }^{\circ}\text{C}$, 1 h), followed by enzymatic hydrolysis using cellulase and hemicellulase cocktails. Glucose release reached **28 g/L**, with hydrolysis efficiency $> 70\%$. Fermentation using *S. cerevisiae* at $30\text{ }^{\circ}\text{C}$ for 72 h produced **12–14 g/L ethanol**, corresponding to $\approx 80\%$ of theoretical yield. Scanning electron microscopy (SEM) confirmed significant fiber disruption post pretreatment. The authors also explore nutrient supplementation (yeast extract, peptone) to improve fermentation kinetics and discuss the role of residual lignin on enzyme adsorption. The study is valuable for identifying BPS as a **renewable, high-biomass-yield residue** from banana cultivation with strong bioethanol potential.

- **A Comparative Study on Bioethanol Production from Rice Straw and Banana Pseudostem through Simultaneous Saccharification and Fermentation using *Kluyveromycesmarxianus* (May 2021, ResearchGate Preprint)**

This experimental work investigates two abundant lignocellulosic feedstocks—rice straw and banana pseudostem—for bioethanol production through **simultaneous saccharification and fermentation (SSF)**. The thermotolerant yeast *Kluyveromycesmarxianus* was employed due to its ability to ferment sugars efficiently at higher temperatures ($40\text{--}45\text{ }^{\circ}\text{C}$), thereby minimizing contamination and improving process kinetics. The study compares pretreatment requirements, saccharification efficiency, and ethanol yield for both feedstocks. Rice straw, having higher lignin ($\approx 18\text{--}20\%$) and silica

content, required more intensive alkaline pretreatment (NaOH, 2–4 %) to achieve adequate cellulose accessibility. Banana pseudostem, on the other hand, has lower lignin and a higher moisture fraction, allowing milder pretreatment and shorter enzymatic hydrolysis time. Enzyme dosages (10–30 FPU/g substrate) and SSF temperature were varied to identify optimal conversion conditions. Results show that the banana pseudostem achieved ethanol concentrations up to **24 g/L**, while rice straw yielded **18 g/L** under similar conditions, indicating better enzymatic digestibility. The study highlights that high-temperature SSF using thermotolerant yeasts can simplify process design by combining hydrolysis and fermentation steps, reducing cooling energy, and minimizing microbial contamination. However, enzymatic hydrolysis efficiency at elevated temperatures remains a limiting factor.

- **Lignocellulosic Bioethanol: Current Status and Future Perspectives” *Authors:* Limayem, A. & Ricke, S. (2012)**

This paper provides a comprehensive overview of bioethanol production from lignocellulosic biomass. It explains the structural complexity of biomass and highlights lignin as the major barrier to efficient conversion. Various pretreatment techniques such as acid, alkaline, steam explosion, and biological methods are compared in detail. The authors emphasize that enzymatic hydrolysis efficiency depends heavily on pretreatment effectiveness. The study also discusses fermentation challenges, especially the inability of traditional yeast to ferment pentose sugars like xylose. It highlights the role of genetically modified microorganisms in improving ethanol yield. Economic challenges such as high enzyme cost and energy consumption are also addressed. The paper suggests integrated biorefinery approaches as a future solution. Overall, it concludes that lignocellulosic ethanol is promising but requires technological advancements for commercialization.

- **“Pretreatment Methods for Lignocellulosic Biofuels Production”* *Authors:* Cheah, W.Y. et al. (2020)**

This review focuses on different pretreatment technologies used in bioethanol production. It compares physical, chemical, physicochemical, and biological methods in terms of efficiency and cost. Chemical methods like acid and alkaline pretreatment show high lignin removal but generate inhibitors. Biological methods using fungi are eco-friendly but slow. The paper highlights ozone pretreatment as an emerging green technology due to its selective lignin degradation. It also discusses how pretreatment affects downstream processes like enzymatic hydrolysis. The authors emphasize the importance of minimizing inhibitor formation for better

fermentation efficiency. Energy consumption and environmental impact are also analyzed. The study suggests hybrid pretreatment methods for better performance. It concludes that no single method is ideal, and process optimization is necessary.

- **Consolidated Bioprocessing for Bioethanol Production”* *Authors: Maleki, F. et al. (2021)***

This paper introduces the concept of Consolidated Bioprocessing (CBP), where enzyme production, hydrolysis, and fermentation occur in a single step. It reduces process complexity and cost compared to traditional multi-step methods. The study highlights the use of engineered microorganisms capable of producing cellulase enzymes and fermenting sugars simultaneously. However, current CBP systems suffer from low ethanol yield and slow microbial growth. The paper discusses metabolic engineering strategies to improve microbial efficiency. It also explains the importance of strain selection and genetic modification. The authors emphasize the potential of CBP for industrial-scale bioethanol production. Challenges such as stability and scalability are also addressed. Overall, CBP is presented as a future breakthrough technology.

- **Simultaneous Saccharification and Fermentation (SSF): A Review”* *Authors: Devos, R.J.B. et al. (2022)***

This review focuses on SSF, a process where enzymatic hydrolysis and fermentation occur simultaneously. It reduces enzyme inhibition by continuously converting glucose into ethanol. The paper explains how SSF improves overall process efficiency and reduces contamination risk. It discusses optimal conditions such as temperature compromise between enzymes and yeast. The study highlights the use of thermotolerant yeast strains for better performance. Substrate concentration and enzyme loading are identified as key parameters. The paper also compares SSF with SHF (Separate Hydrolysis and Fermentation). It concludes that SSF is more efficient but requires process optimization. Industrial applications and challenges are also discussed.

- **Advances in *Saccharomyces cerevisiae* for Xylose Fermentation”* *Authors: Wagner, E.R. et al. (2023)***

This paper focuses on genetic engineering of *Saccharomyces cerevisiae* to improve its ability to ferment xylose, a major sugar in lignocellulosic biomass. Normally, yeast ferments only glucose, limiting ethanol yield. The study explains metabolic pathways introduced to enable xylose utilization. It highlights improvements in ethanol productivity and yield. Challenges such as

cofactor imbalance and slow fermentation rates are discussed. The paper also explores adaptive evolution techniques to enhance strain performance. Industrial relevance is emphasized, as efficient xylose fermentation can significantly increase bioethanol production. The authors conclude that engineered yeast strains are essential for next-generation biofuels.

- **“Bioethanol Production from Agricultural Residues: A Review”* Authors: Jayakumar, M. et al. (2023)**

This paper reviews the use of agricultural residues like rice straw, wheat straw, and corn stover for bioethanol production. It highlights their abundance, low cost, and sustainability. The study explains the importance of pretreatment to remove lignin and improve cellulose accessibility. It compares different hydrolysis and fermentation techniques. The paper also discusses environmental benefits such as waste reduction and lower greenhouse gas emissions. Challenges like high processing cost and low efficiency are addressed. It suggests integrating waste management with biofuel production. The study concludes that agricultural residues are a promising feedstock for sustainable bioethanol.

- **Belal, E. B. (2013). “Bioethanol Production from Rice Straw Residues.” *BioResources*, 8(1), 1020–1036.**

Belal’s study is one of the most cited experimental papers exploring **pretreatment–hydrolysis–fermentation chains** for rice straw. It systematically compares **acid and alkaline pretreatments** (1 % H₂SO₄ vs 2 % NaOH) and evaluates their effects on sugar yield, inhibitor formation, and fermentation efficiency. Following pretreatment, enzymatic hydrolysis was conducted using cellulase from *Trichoderma reesei* under optimized pH 5.0 and 50 °C. The alkaline-pretreated biomass showed **75 % lignin removal** and glucose release up to **38 g/L**, outperforming acid pretreatment due to reduced inhibitor formation (notably furfural and HMF). Subsequent fermentation with *Saccharomyces cerevisiae* achieved ethanol yields approaching **0.46 g/g glucose** (≈90 % theoretical). The paper provides strong empirical support for alkaline pretreatment as an efficient route for rice-straw bioconversion. Energy and mass balances indicate the importance of pretreatment severity and washing steps in determining hydrolysate quality. Additionally, the study provides insight into the effect of particle size and solid loading on hydrolysis kinetics.

- **Bioethanol Production from Banana Peels — Multiple Experimental Studies (2016–2023, ResearchGate & Open Access)**

A body of research between 2016–2023 collectively evaluates **banana peel waste** as a low-cost feedstock for bioethanol. Banana peels, with **cellulose $\approx$ 15–25 %**, **hemicellulose $\approx$ 10–15 %**, and **lignin $\approx$ 8–12 %**, are easier to hydrolyze than typical agricultural residues. Various pretreatment approaches have been tested—hot water, dilute acid, alkali, and combined acid-enzymatic treatments—to enhance fermentable sugar yield. For example, A. O. Ademiluyi et al. (2017) reported a 60 % increase in glucose release after dilute acid pretreatment, while M. Patel et al. (2021) achieved ethanol yields of **0.42 g/g reducing sugar** using *S. cerevisiae* after alkaline pretreatment. Several studies utilize enzyme cocktails of cellulase and amylase to address the high starch fraction in fresh peels, obtaining up to **20–25 g/L ethanol**. Biological delignification using *Phanerochaete chrysosporium* has also been explored to reduce chemical use. Collectively, these studies demonstrate banana peels as a viable substrate for **fruit-waste valorization**, especially in decentralized or small scale settings where banana waste is abundant.

Chapter 3

Experimental setup& Procedure

3.1. Experimental setup:

3.1.1. Lignin separation:



Fig.3.1.1 Ozonation

- The main goal of pretreatment is to disrupt the complex structure of lignocellulosic biomass.
- It aims to break down lignin and hemicellulose, which form a protective layer around cellulose.
- This process helps to expose cellulose fibers, making them more accessible for enzymatic hydrolysis.
- As a result, the conversion of cellulose to glucose becomes easier, leading to higher ethanol yield.

3.1.2. Fermentation Setup:

Fermentation is the process of converting fermentable sugars (glucose, xylose) obtained from cellulosic waste into ethanol or other value-added products under anaerobic (oxygen-free) conditions.

· Microorganisms Used:

Commonly yeast (e.g., *Saccharomyces cerevisiae*) or bacteria are used for ethanol

fermentation.

· **Process Steps:**

1. Hydrolyzed cellulose releases simple sugars.
2. Sugars are fermented by microbes to produce ethanol, acids, or other biochemicals.
3. By-products include CO₂ and heat.

· **Parameters Affecting Fermentation:**

- o **Temperature:** Usually **30–35°C** for yeast activity.
- o **pH:** Maintained around **4.5–5.0**.
- o **Duration:** Typically **24–72 hours**, but we have fermented it for 6 days.
- o **Anaerobic conditions:** Essential for ethanol production.

Products Formed:

- o **Biofuel**



Fig. 3.1.2 Fermentation

3.1.3. Distillation Setup:



Fig. 3.1.3 Distillation

Chapter 4

(Chemicals & Raw materials)

4.1 Role of Chemicals & Raw materials

4.1.1 Role of Trichoderma in Lignin Separation:

1. Trichoderma is a **fungus** that helps in **biological lignin degradation and separation**.
2. It produces **ligninolytic enzymes** that break down complex lignin structures.
3. These enzymes **loosen the lignin–cellulose complex**, making cellulose more accessible.
4. Trichoderma also produces **cellulase and xylanase**, aiding in complete biomass breakdown.
5. Used in **biological pretreatment** of lignocellulosic biomass for **bioethanol production**.
6. Reduces lignin content **eco-friendly**, without using harsh chemicals.
7. Provides a **sustainable and cost-effective** alternative to chemical lignin separation methods.

4.1.2 Role of Alum in Lignin Separation:

1. **Alum ($\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$)** is a **chemical coagulant** used in lignin and color removal from pulp and paper wastewater.
2. It helps in **coagulation and flocculation** of lignin particles suspended in solution.
3. Effective in **reducing lignin content** and **color** of black liquor or effluents from paper mills.
4. Works best in **acidic to neutral pH (around 4–6)** for optimal coagulation.
5. Used as a **low-cost and efficient** chemical method for lignin removal.
6. Excessive alum can lead to **aluminum residue** and **pH imbalance**, requiring post-treatment.

4.1.3 Role of Microbes in Lignin Separation

1. Microbes (mainly fungi and bacteria) degrade lignin using **ligninolytic enzymes** like laccase, LiP, and MnP.
2. **White-rot fungi** (e.g., *Phanerochaete chrysosporium*, *Trametes versicolor*) are the most effective lignin degraders.
3. **Bacteria** such as *Pseudomonas* and *Bacillus* assist through oxidative enzyme activity.
4. They convert lignin into simpler aromatic acids (vanillic, syringic).

5. Enhances cellulose accessibility for enzymatic hydrolysis in **bioethanol production**.
6. An **eco-friendly** and low-energy biological pretreatment method.

4.1.4. Role of Ozone Purging in Lignin Separation

1. **Ozone (O_3)** oxidizes and breaks aromatic rings and ether bonds in lignin (ozonolysis). 2. Converts lignin into smaller, soluble, and colorless compounds — useful in pulp bleaching.
2. **Decomposes into oxygen (O_2)**, leaving no toxic residue.
3. Operates effectively under **mild and low-temperature conditions**.
4. Enhances cellulose exposure for **biofuel production**.
5. Considered a **clean and eco-friendly alternative** to chlorine bleaching.

4.1.5. Role of Potassium Dichromate in Lignin Separation

1. **Potassium dichromate ($K_2Cr_2O_7$)** oxidizes lignin's aromatic rings and side chains.
2. Helps **bleach pulp** by destroying colored chromophoric groups.
3. Improves **cellulose purity and brightness** in pulp processing.
4. Works efficiently in **acidic conditions** with sulfuric acid.
5. Produces **toxic Cr^{6+} residues**, making disposal environmentally hazardous.
6. Largely replaced by **greener oxidants** like ozone and hydrogen peroxide.

4.1.6. Role of Curd in Lignin Separation

1. Curd contains **lactic acid bacteria (LAB)** that produce organic acids breaking lignin–carbohydrate bonds.
2. The **acidic fermentation** softens lignocellulosic biomass and loosens lignin structure.
3. Enzymes from LAB aid partial lignin modification.
4. Acts as a **natural, eco-friendly biological pretreatment** method.
5. Reduces chemical usage and improves **biodegradability** of biomass.
6. Though less efficient, it supports **green, low-cost, sustainable processing**.

4.1.7. Role of Ozone Purging in Lignin Separation

1. **Ozone (O_3)** oxidizes and breaks aromatic rings and ether bonds in lignin (ozonolysis).
2. Converts lignin into smaller, soluble, and colorless compounds — useful in pulp bleaching.
3. **Decomposes into oxygen (O_2)**, leaving no toxic residue.

4. Operates effectively under **mild and low-temperature conditions**.
5. Enhances cellulose exposure for **biofuel production**.
6. Considered a **clean and eco-friendly alternative** to chlorine bleaching.

4.2. About Raw material:

- ▶ **Banana Peels:** Rich in carbohydrates and cellulose; easily available food waste.
- ▶ **Rice Straw:** Abundant residue; high in silica and lignin.
- ▶ **Cotton Seed Waste:** Contains cellulose and oil; industrial by-product.

All are low-cost, sustainable feedstocks for ethanol production.

❖ FEEDSTOCK AND RAW MATERIALS:

Identified the primary sources of cellulosic waste for ethanol production

- **Rice Straws:** Abundant agricultural residue left after grain harvest.
- **Lignocellulosic Biomass:** Complex plant materials containing cellulose and hemicellulose
- **Cellulosic Waste:** General organic waste from paper, pulp, and farming Value-Added

Products: Transforming low-cost waste into high-value biofuels

- **Rice Stem (Rice Straw):**

Agricultural waste with ~35– 40% cellulose.

Typical composition:

- **Cellulose:** 35–50%
- **Hemicellulose:** 20–35%
- **Lignin:** 10–25%

Rice straw was selected because:

It contains high cellulose (~40%). It produces higher fermentable sugar yield. It has lower fermentation inhibitors compared to mixed agro-waste. It gives better ethanol yield per gram of feed.

- **Biological Agents and Catalysts**

(The living organisms driving the chemical conversion process)

- **Microbes and Yoghurt**

Yoghurt acts as a source of beneficial bacteria Microbes assist in breaking down complex sugars

Natural catalysts for organic decomposition.

- **Yeast Cultures**

Primary driver for converting sugars to ethanol High tolerance to alcohol concentrations Essential for efficient fermentation cycles.

Chapter 5

Experimental Study

5.1 Lignin Separation:

Objective

To synthesize bioethanol and other value-added compounds from cellulosic waste materials such as banana peels, rice straw, cotton seed waste, and sarki pend through ozonation, microbial action, and fermentation.



Fig. 3.1.1 Ozonation

5.1.1 Batch A – Cotton Seed Experiment (27/07/2025)

Objective:

To examine the effect of microbial and ozone pretreatment on cotton seed waste for cellulose degradation.

Materials Used:

Fresh cotton seed waste – 30 g

Alum – 3 g

Curd – 2 g

Microbes – 3 g

Procedure:

1. The cotton seed waste was mixed thoroughly with alum, curd, and microbial

culture.

2. The mixture was subjected to ozonation for 2 hours using an ozone generator.

Purpose: Ozone helps break lignin and hemicellulose bonds, improving cellulose accessibility.

3. After ozonation, the material was stored in a sterile container for further microbial action.

Observation:

Material softened considerably, and a mild odor indicated partial degradation of organic matter.

5.1.2 Batch A – Rice Stem Experiment (27/07/2025)

Objective:

To pretreat rice stems with ozone and microbial mixture to enhance cellulose availability.

Materials Used:

Rice stems – 30 g

Alum – 3 g

Microbes – 3 g

Procedure:

1. Rice stems were chopped and blended with microbes and alum solution. 2. The mixture underwent 2-hour ozonation to enhance delignification. 3. Post-treatment, the sample was left to stand for microbial activity.

Observation:

Noticeable color change from light yellow to pale brown, indicating lignin oxidation.

5.1.3 Batch A – Rice Stem (07/08/2025)

Objective:

To evaluate the influence of a chemical oxidant (Potassium Dichromate) during ozonation on rice stems.

Materials Used:

Rice stems – 30 g

Alum – 2 g

Potassium Dichromate – 5 mL

Procedure:

1. Rice stems were mixed with alum and potassium dichromate solution.
2. The sample was ozonized for 2 hours.
3. It was then washed with alum water and dried at room temperature.

Purpose of Reagents:

Alum: Acts as a coagulant aiding in impurity separation.

$K_2Cr_2O_7$: Oxidizes lignin and improves cellulose exposure.

Observation:

Material became brittle and lighter, confirming oxidation and partial delignification.



Fig. 5.1.3 pretreated rice stem

5.1.4 Batch A – Banana Peel Experiment (07/08/2025)**Objective:**

To pretreat banana peel waste using biological agents and ozone for enhanced fermentation efficiency.

Materials Used:

Banana peels – 30 g

Alum – 39 g

Curd – 15 g

Microbes – 3 g

Procedure:

1. Banana peels were cleaned and cut into small pieces.
2. Alum, curd, and microbes were mixed uniformly with the peels.
3. The mixture was ozonized for 2 hours to promote lignin oxidation and cellulose exposure.

Observation:

Peels softened significantly; microbial activity began within 24 hours, producing mild fermentation odor.



Fig. 5.1.4 pretreated banana peels

5.1.5 Batch B – Sarki Pend (14/08/2025)**Objective:**

To study the effect of Trichoderma inoculation on ozonized sarki pend for cellulose degradation.

Materials Used:

Sarki pend – 20 g

Alum – 5 g

Trichoderma culture – 1 ml

Curd – 2 g

Procedure:

1. Sarki pend and alum were first ozonized for 2 hours.
 2. After ozonation, the material was dried and inoculated with Trichoderma and curd. 3.
- The mixture was left to stand under aerobic conditions for 48 hours to initiate enzymatic degradation.

Observation:

Visible fungal growth of Trichoderma observed within 2 days, confirming successful inoculation.



Fig. 5.1.5 pretreated sarki pend

Common Observations and Inferences:

Ozonation effectively disrupted lignin bonds, improving cellulose exposure. Microbial and enzymatic action (from curd and Trichoderma) led to partial hydrolysis of cellulose. Alum helped in maintaining a suitable pH and clarifying the broth during later separation. Samples became progressively softer and more porous after treatment, ideal for fermentation.

5.2 Fermentation:

Feed Composition:

- 50 g banana peels
- 50 g rice straw

50 g sarki pend

750 mL water

100 g curd

5 g microbes

Process Steps:

1. Biomass was pretreated with an effective way which was a mixture of biomass 100gm curd 5gm microbes then we kept it for 6 hour ozonation.
2. The mixture was then fermented anaerobically for 10 days.
3. After 10 days, 1.5 g yeast was added to enhance ethanol production.
4. Fermentation continued for 6 more days at ~25°C.
5. Expected Outcome: Conversion of cellulose into glucose via enzymatic and microbial hydrolysis, followed by ethanol production through yeast fermentation.
6. We have used a simple distillation for separating the ethanol from the mixture. It starts at 3.15 to 4.15. We did it for 1 hour. The first drop of product came after 10 min from starting.

Rice Straw Experiments:

05/02/2026

1. Reactor volume taken: **2 L**
2. Added **85 g rice straw** to the reactor
3. Added **750 mL distilled water**
4. The mixture was subjected to **ozonation for 6 hours** (Pretreated)
5. After ozonation, added:
6. **50 g curd**
7. **5 g microbial culture**
8. The mixture was allowed to **ferment for 10 days**
9. After 10 days, **1.5 g yeast** was added
10. Then we have waited for 6 days to convert the fermented sugar into bioethanol
11. The mixture was then **distilled for 1 hour**
12. Distillation temperature maintained at **80–85 °C**

10/03/2026

1. Reactor volume taken: **1 L**
2. Added **40 g rice straw** to the reactor
3. Added **350 mL distilled water**
4. The mixture was subjected to **ozonation for 6 hours** (Pretreated)
5. After ozonation, added:
6. **25 g curd**
7. **2.5 g microbial culture**
8. The mixture was allowed to **ferment for 10 days**
9. After 10 days, **1.5 g yeast** was added
10. The mixture was then **distilled for 1 hour**
11. Distillation temperature was maintained at **80–85 °C**

16/03/2026

1. Reactor volume taken: **2 L**
2. Added **80 g rice straw** to the reactor
3. Added **750 mL distilled water**
4. The mixture was subjected to **ozonation for 6 hours**
5. After ozonation, added: **50 g curd , 5 g microbial culture**
6. The mixture was allowed to **ferment for 11 days**
7. After 11 days, **1.5 g yeast** was added
8. The mixture was further kept for **6 days** to convert fermentable sugars into bioethanol
9. The mixture was then **distilled for 1 hour**
10. Distillation temperature was maintained at **80 °C**

16/03/2026

2. Reactor volume taken: **2 L**
3. Added **160 g rice straw** to the reactor
4. Added **1500 mL distilled water**
5. The mixture was subjected to **ozonation for 6 hours**
6. After ozonation, added: **100 g curd , 10 g microbial culture**
7. The mixture was allowed to **ferment for 11 days** (in BOD incubator to maintain temperature at 23°C)
8. After 11 days, **3 g yeast** was added

9. The mixture was further kept for **6 days** to convert fermentable sugars into bioethanol
10. The mixture was then **distilled for 1.5 hour**
11. Distillation temperature was maintained at **80-82 °C**

10/04/2026

2. Reactor volume taken: **2 L**
3. Added **160 g rice straw** to the reactor
4. Added **1500 mL distilled water**
5. The mixture was subjected to **ozonation for 6 hours** (Pretreated)
6. After ozonation, added:
7. **100 g curd**
8. **10 g microbial culture**
9. The mixture was allowed to **ferment for 10 days**
10. After 10 days, **3 g yeast** was added
11. The mixture was then **distilled for 1 hour**
12. Distillation temperature was maintained at **80–85 °C**

Fermenter:



Distillation Setup:



Chapter 6

Results and Discussion

The experimental study on bioethanol production from rice straw demonstrated successful conversion of lignocellulosic biomass into ethanol through pretreatment, fermentation, and distillation processes.

1. Effect of Pretreatment (Ozonation)

- Ozonation for **6 hours** resulted in effective disruption of lignin structure.
- Visible changes such as **color fading and softening of biomass** were observed, indicating partial delignification.
- This observation is consistent with earlier findings in the report, where ozonation improved cellulose accessibility .

2. Fermentation Performance

- The addition of **curd (lactic acid bacteria)** and **microbial culture** initiated biological degradation.
- After **11 days of fermentation**, the mixture showed:
 - Development of **fermentation odor**
 - Increased **liquidity and breakdown of solid biomass**
- Upon addition of **1.5 g yeast**, further fermentation for **6 days** enhanced ethanol formation.
- Gas evolution (CO₂) and change in consistency confirmed active fermentation.

3. Ethanol Formation

- Conversion of fermentable sugars into ethanol was achieved after the yeast fermentation stage.
- The extended fermentation period (total ~17 days) improved sugar conversion compared to earlier batches (10-day fermentation).
- However, the rate of conversion was relatively slow due to:
 - Absence of enzymatic hydrolysis step
 - Dependence on natural microbial activity

4. Distillation Results

- Distillation was carried out at **80 °C for 1 hour**.
- The **first drop of distillate appeared after ~10 minutes**, confirming ethanol presence.
- The collected distillate contained **bioethanol along with water impurities**, indicating simple distillation limitations.

5. Yield Observation

- The overall ethanol yield was **low (~10%)**, as also noted in the conclusion of the report .
- Reasons for low yield:
 - Incomplete lignin removal
 - Limited sugar availability
 - Non-optimized fermentation conditions
 - Lack of controlled temperature and pH

6. Overall Inference

- The combined process of **ozonation + microbial pretreatment + fermentation** is feasible and eco-friendly.
- The experiment successfully demonstrated the **conversion of agricultural waste (rice straw) into bioethanol**, but efficiency needs improvement.
- The process supports the concept of **sustainable biofuel production from lignocellulosic biomass**.

7. Key Result

- Bioethanol was successfully produced from rice straw.
- Pretreatment improved biomass degradation.
- Fermentation and distillation confirmed ethanol formation.
- Yield obtained was low (~10%), indicating scope for optimization.

Chapter 7

Conclusion

The project successfully demonstrated the conversion of **cellulosic waste** such as banana peels, rice straw, cotton seed waste, and sarki pend into **bioethanol**. The combined use of **ozonation, microbial treatment, and fermentation** broke down lignin and enhanced cellulose accessibility. We got the results but not in the expected quantity. The yield is 10% from the performed batches.

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A

Project

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In

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2025-26

CERTIFICATE

This is to certify that the project entitled “**CATALYTIC TRANSFORMATION OF RENEWABLE FEEDSTOCKS TO USEFUL CHEMICALS**” is a bona fide work of DHANASHRI SURESH MAHAKAL (15), SIDDHI DASHARTH KHARATH (13), APURVA ANIL SHINDE (23) submitted to the University of Mumbai in partial fulfillment of the requirement for the award of the degree of “**Undergraduate**” in “**Chemical Engineering**”.

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Project Report Approval for B.E.

This project report entitled “**CATALYTIC TRANSFORMATION OF RENEWABLE FEEDSTOCKS TO USEFUL CHEMICALS**” by Prof. Y. V. Thakare is approved for the degree of **Chemical Engineering.**

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DECLARATION

We declare that this written submission represents our ideas in our own words and where others ideas or words have been included; we have adequately cited and referenced the original sources. We also declare that we have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in our submission. We understand that any violation of the above will because for disciplinary action by the Institute and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been taken when needed.

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ABSTRACT

This project is based upon synthesis of biodiesel from waste cooking oil (WCO) from heterogeneous catalyst. Demand for diesel continues to increase due to rapid population growth, which could contribute to fossil fuel exhaustion. Biodiesel has been widely developed as a replacement for conventional diesel to resolves the issue. Biodiesel production from waste cooking oil (WCO) was carried out via the trans-etherification process using of egg shell catalyst. Here in this project, we aimed to map the effect of use of catalyst on the effective carriage of reaction. Various papers have been published in the synthesis of catalyst from various sources, and variation of other conditions. There is extensive research carried out in this field. Here we will also check the effect of regenerated catalyst on the reaction. We study the reaction with catalyst, without catalyst and reuse of catalyst. Study of the catalyst is made on Trans-etherification reaction at different Molar Ratios.

Key Words: Egg shell Catalyst, Trans-etherification

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CHAPTER-1
INTRODUCTION

1. INTRODUCTION

General Introduction

As pricing of petroleum is increasing and significant decrease in the amount of fossil fuel making us to find better substitution. Biodiesel, a clean renewable fuel has recently been the best candidate for a diesel substitution because it can be used in any compression ignition without the need of modification. Production of biodiesel is the important process and it is done by Trans-etherification. Trans-etherification of vegetable oil with simple alcohol has long been a preferred method for preparing fatty esters. Currently, biodiesel is produced by performing trans-etherification reaction, a triglyceride reacts with an alcohol such as KOH or NaOH dissolved in methanol. The major disadvantage of homogenous catalyst is that they cannot be reused or regenerated, because the catalyst is consumed in the reaction. Moreover separation of catalyst from mixer is difficult and requires more equipment with result in higher production cost. In addition, crude glycerol by product has very low value due to high impurities such as catalyst, soap, methanol and water. The use of heterogeneous catalyst is a key technology to overcome such problems.

What is Catalyst?

Any substance that changes the rate of a reaction without itself being consumed. Enzymes are naturally occurring catalysts responsible for many essential biochemical reactions. Most solid catalysts are metals or the oxides, sulfides, and halides of metallic elements and of the semi-metallic element boron, aluminum, and silicon. Gaseous and liquid catalysts are commonly used in their pure form or in combination with suitable carriers or solvents; solid catalysts are commonly dispersed in other substances known as catalyst supports. Enzymes and other biocatalysts are often considered as a third category. Catalysis is ubiquitous in chemical industry of all kinds. Estimates are that 90% of all commercially produced chemical products.

Types of Catalyst:

A) Heterogeneous Catalyst:

In chemistry, heterogeneous catalysis is a catalyst where the phase of catalysts differs from that of the reactants or products. The process contrasts with homogeneous catalysis where the reactants, products and catalyst exist in the same phase. Phase distinguishes between not only solid, liquid, and gas components, but also immiscible mixtures (e.g. oil and water), or anywhere an interface is present. Heterogeneous catalysis is very important because it enables faster, large-scale production and the selective product formation. Approximately 35% of the world's GDP is influenced by catalysis. The production of 90% of chemicals (by volume) is assisted by solid catalysts. The chemical and energy industries rely heavily on heterogeneous catalysis. For example, the Haber–Bosch process uses metal-based catalysts in the synthesis of ammonia, an important component in fertilizer; 144 million tons of ammonia were produced in 2016.

B) Homogeneous Catalyst:

Advantages:

Homogeneous catalysts are generally more selective than heterogeneous catalysts. For exothermic processes, homogeneous catalysts dump heat into the solvent. Homogeneous catalysts are easier to characterize precisely, so their reaction mechanisms are amenable to rational manipulation.

Disadvantages:

The separation of homogeneous catalysts from products can be challenging. In some cases, involving high activity catalysts, the catalyst is not removed from the product. In other cases, organic products are sufficiently volatile that they can be separated by distillation. Homogeneous catalyst has limited thermal stability compared to heterogeneous catalysts. Many organometallic complexes degrade <100 °C. Some pincer-based catalysts, however, operate near 200 °C.

C) Biocatalyst:

Bio-catalysis is defined as the use of natural substances that include enzymes from biological sources. Enzymes have pivotal role in the catalysis of hundreds of reactions that include production of alcohols from fermentation and cheese by breakdown of milk proteins. Recent advances in the field of scientific research have helped to understand the structure and functional activities of enzymes, which has in turn led to an increase in their stability, activity, sustainability, and substrate specificity. Currently, there are hundred different bio-catalytic processes that have been implemented in various pharmaceuticals, chemical, food, and agriculturally based industries. Bio-catalytic processes are similar to conventional processes in many ways and the major factors to be accounted for are reaction kinetics and stability for both single and multistep reactions. Biocatalyst engineering requires rational design and directed evolution. The impact of bio-catalysis in the future will be precisely this: The increasing ability to use enzymes to catalyze chemical reactions in industrial processes, including the production of drug substances, flavors, fragrances, electronic chemicals.

Trans-etherification Reaction:

Trans-etherification reaction is a reaction process by means of which triglyceride molecules present in animal fats or vegetable oils react with an alcohol in general to form esters and glycerol. It has been evidently clear that the growing concerns of depletion in the sources of fossil fuels which are categorized as non-renewable energy resources as well as increase in crude oil prices and concerns to reduce the environmental pollution has resuscitated the production of biodiesel hence several processes for the production of biodiesel has been developed in which Trans-etherification acts as a common integral reaction. Trans-etherification so called alcohols is, can be described as the displacement of alcohol from an ester by another alcohol in a process similar to hydrolysis, except that an alcohol is employed instead of water. Suitable Alcohols used in this reaction include Methanol, Ethanol, propanol, butanol and amyl alcohol. Methanol and Ethanol are more frequently utilized. The importance of this process is that it reduces the viscosity of triglycerides, from which biodiesel is derived; there by this reduction enhances the physical properties of renewable fuels to improve engine performance. Generally, this process is carried out in the presence of catalyst which is necessary to increase the reaction rate and the trans-etherification reaction yield.

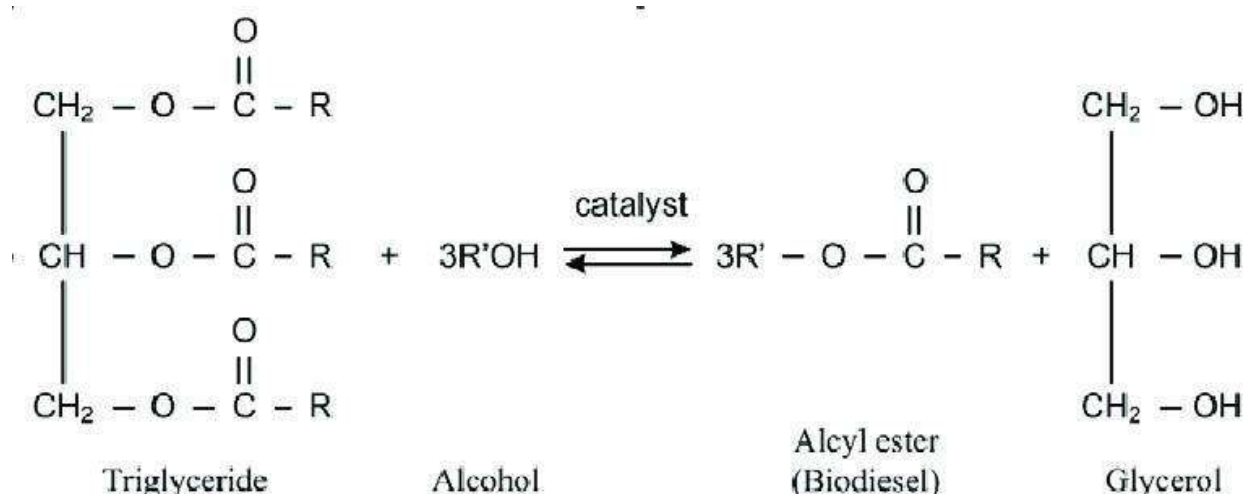


Fig1.1: Trans-Etherification Reaction

This is a trans-etherification reaction in its general form. Catalyst used can be classified attending to its chemical presence in the trans-etherification reaction: homogeneous or heterogeneous catalysts. Many ways and methods have been under investigations to determine the right method that can be adapted for biodiesel manufacturing because based on studies and research due to uncertain amount offered fatty acids (FFA) in triglycerides, if the FFA is low it leads to a reaction with the homogeneous catalysts that are mostly basic in nature forming soaps. The soap formation consumes the catalyst and decreases the ester yield. Moreover, the soap formation also affects this partition of glycerol from the biodiesel which is considered to be major failure from the industrial point of view. The disadvantages of homogeneous catalyzed trans-etherification reaction have motivated the study and research towards heterogeneous catalysts. In general, heterogeneous biodiesel production process has a smaller number of unit operations, with simple product separation and purification steps.

CHAPTER-2

LITERATURE REVIEW

2. LITERATURE REVIEW

The work has carried out by Moraine Hamzah etc. Al, this research paper is focusing on Production of biodiesel from cooking oil using egg shell catalyst, (Noraini Hamzah etc. Al, 2021). This paper addressed the influence of the reaction temperature on the yield of biodiesel. The oil's viscosity is more significant at a higher temperature, resulting in a higher mixing step and faster salvation of glycerol. Few other parameters will be affecting the yield of biodiesel, including the type of catalyst, type of solvent, and availability of free fatty acids (FFAs). Heterogeneous catalysts are known to enhance the trans-etherification process relative to the homogeneous process by removing increased production costs associated with homogeneous catalysis and reducing contaminants 'generation. Heterogeneous catalysts help fast recovery, reusability and a cost-effective green operation. These catalysts withstand high levels of FFAs and moisture content. Methanol as a solvent can facilitate higher reaction rates and higher biodiesel's yield than other alcohol due to its high solubility. The presence of FFAs content also significantly affects biodiesel production through as the formation of gels and foams will hinder the separation of glycerol from biodiesel, causing low yield of biodiesel.

The work has carried out by Wisdom C. Ulakpaet Al. this research paper is focusing on Statistical optimization of biodiesel synthesis from waste cooking oil using NaOH/ egg shell impregnated catalyst,(WisdomC.Ulakpaet.Al,2022).The aim of the investigation presented in this research synthesis of NaOH/egg shell catalyst, testing its efficiency in producing biodiesel from used cooking oil, and identify the ideal conditions for production. The creation of a heterogeneous catalyst supported on egg shell for the synthesis of biodiesel is the unique contribution of this work. Despite being affordable and widely accessible, very little research has been done on the use of betonies clay as a catalytic support in the synthesis of biodiesel.

The work has carried out by Pisitpong Intaraponget al, this research paper is focusing on the trans-etherification of palm oil using KOH supported on betonies in a continuous reactor, (Pisitpong Intarapongteal, 2014). In this study, KOH/betonies catalysts, prepared by the

Impregnation method, were studied for trans-etherification in a continuous reactor. The catalysts were characterized using X-ray diffraction, Fourier transform infrared spectrophotometer, scanning electron microscopy, nitrogen adsorption/desorption, CO₂ temperature programmed desorption, and X-ray fluorescence. The results showed that the addition of 20 wt% K on the egg shell catalyst at 60°C reaction temperature, methanol/oil molar ratio of 15:1, and flow rate of 0.3 ml/min gave a biodiesel content exceeding 75%. With increasing basicity, the presence of K₂O was observed, and the biodiesel content was improved. In addition, there was negligible loss in activity when the catalyst was operated at 60°C for 7 days. Aluminum level of potassium leaching on the 20 wt% egg shell was observed during the run. In this present work, various amounts of KOH on betonies were introduced in a continuous reactor to investigate the catalytic performance in biodiesel production under optimum conditions. The influence of potassium loading and stability of the catalysts were also investigated.

CHAPTER-3
OBJECTIVES OF RESEARCH WORK

3. OBJECTIVES OF RESEARCH WORK

The Project work was done with the following Objectives:

- 1) To Synthesis and modification of egg shell catalyst Heterogeneous catalyst.
- 2) To study the effect of catalyst on production of biodiesel using various parameters such as:
 - I. Effect of catalyst loading on conversion of Limiting reactant.
 - II. Effect of Molar Ratio on Conversion of Limiting Reactant.
 - III. Effect of Reaction Temperature on conversion of Limiting reactant.
 - IV. Effect of Reaction Time on conversion of Limiting reactant.
 - V. Effective catalyst reusability.

CHAPTER-4
MATERIALS AND METHODS

4. MATERIALS AND METHODS

Waste cooking oil was collected from the local cafeteria. Methanol is used in this experiment And egg shell clay, utilized throughout the study, was acquired from a store.



Fig.4.1: Furnace Heating



Fig.4.2: Eggshell Powder



Fig.4.3: Mixing With KOH

Experimental Setup:



Fig.4.4: Oil Bath with Magnetic stirrer

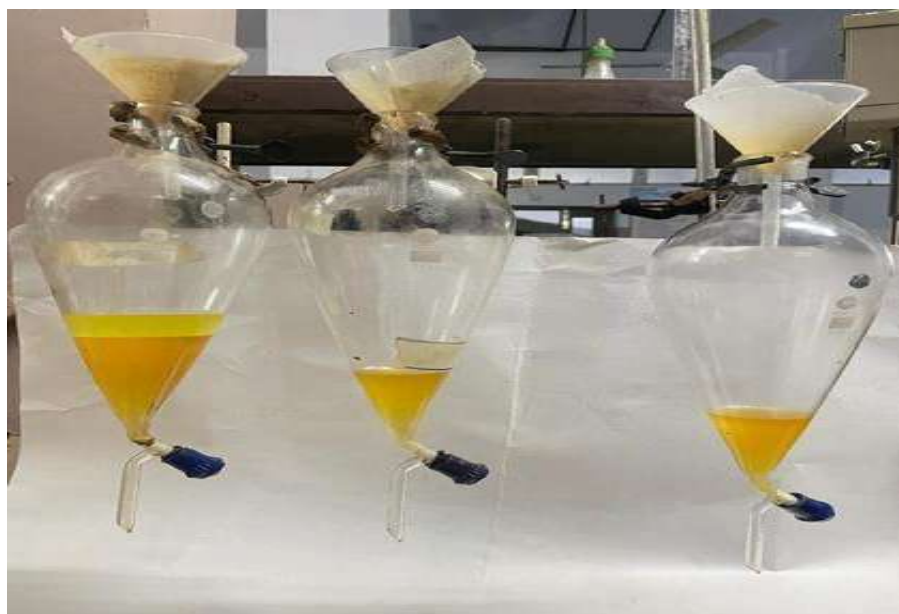


Fig.4.5: Separation

Experimental procedure for trans-etherification:

1. The etherification reaction was carried out in a round bottom (RB) glass flask (500 cm³) fitted with a water-cooled condenser.
2. The temperature was maintained using an oil bath with magnetic stirrer.
3. The reactants were taken directly into the round bottom flask along with the catalyst.
4. The reaction mixture was continuously stirred during the reaction using a magnetic stirrer.
5. The reaction was carried out for a definite period of time after which the catalyst was separated from the reaction mixture.
6. Experiments were designed by varying the amount of the catalyst, the molar ratios of the reactants, the reaction temperature and the reaction period.

CHAPTER-5
RESULT AND DISCUSSION

Methanol: Oil Ratio	Biodiesel Yield (%)
9:1	65%
12:1	78%
20:1	92%
30:1	95%(Maximum)
36:1	93%
40:1	90%

Table.5.1: Transesterification of Sunflower Oil with Methanol

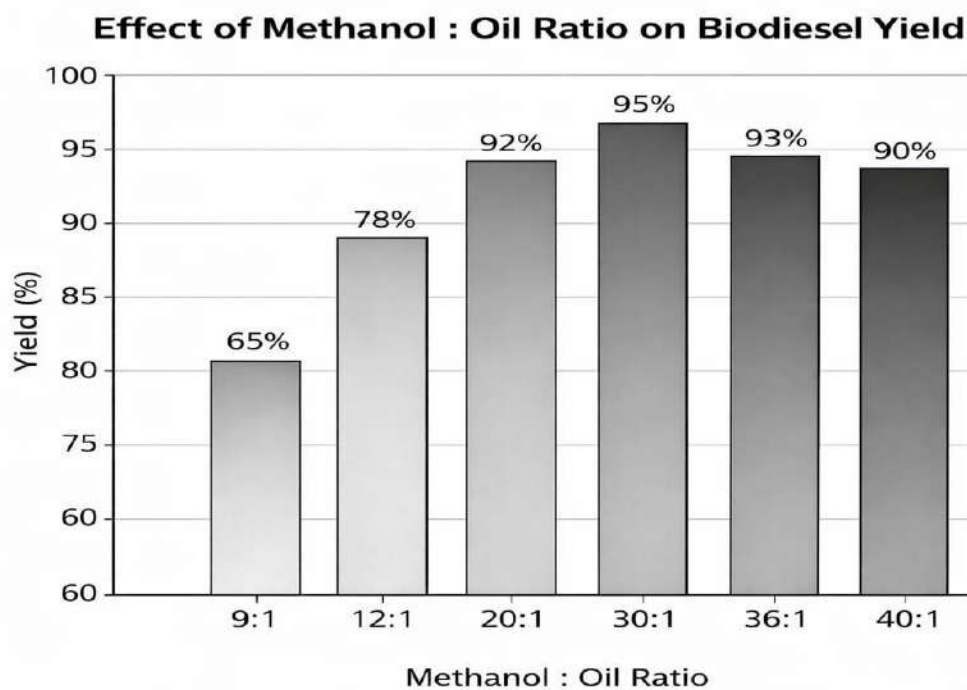


Fig.5.1: Effect of Methanol: Oil Ratio on Biodiesel Yield

Ethanol: Oil Ratio	Biodiesel yield (%)
9:1	55%
12:1	63%
20:1	75%
30:1	85%
36:1	90%
40:1	88%

Table. 5.2: Transesterification of Vegetable Oil with Methanol

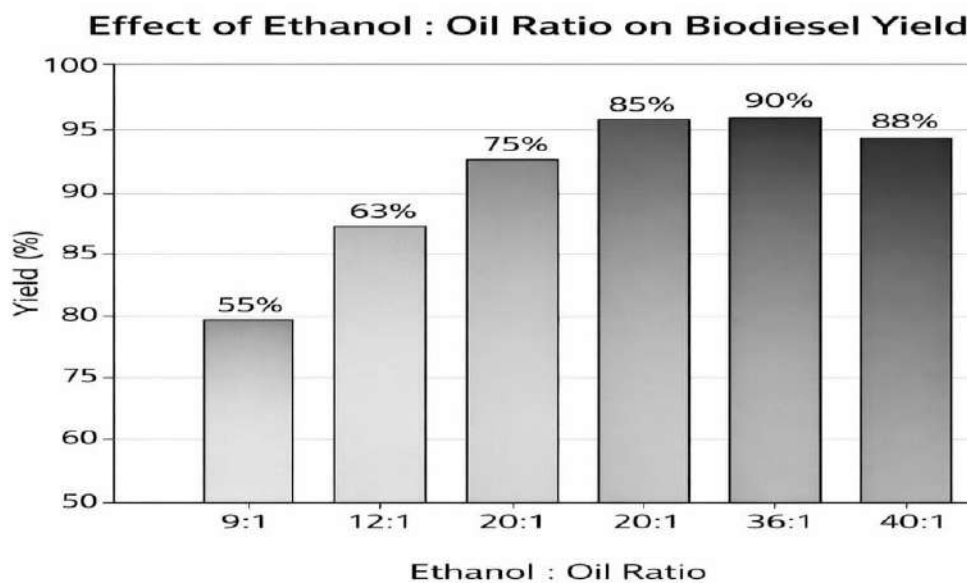


Fig.5.2: Effect of Ethanol: Oil Ratio on Biodiesel Yield

Methanol: Oil Ratio	Biodiesel yield (%)
9:1	72%
12:1	82%
20:1	91%
30:1	95%
36:1	93%
40:1	90%

Table.5.3: Transesterification of Vegetable Oil with Ethanol

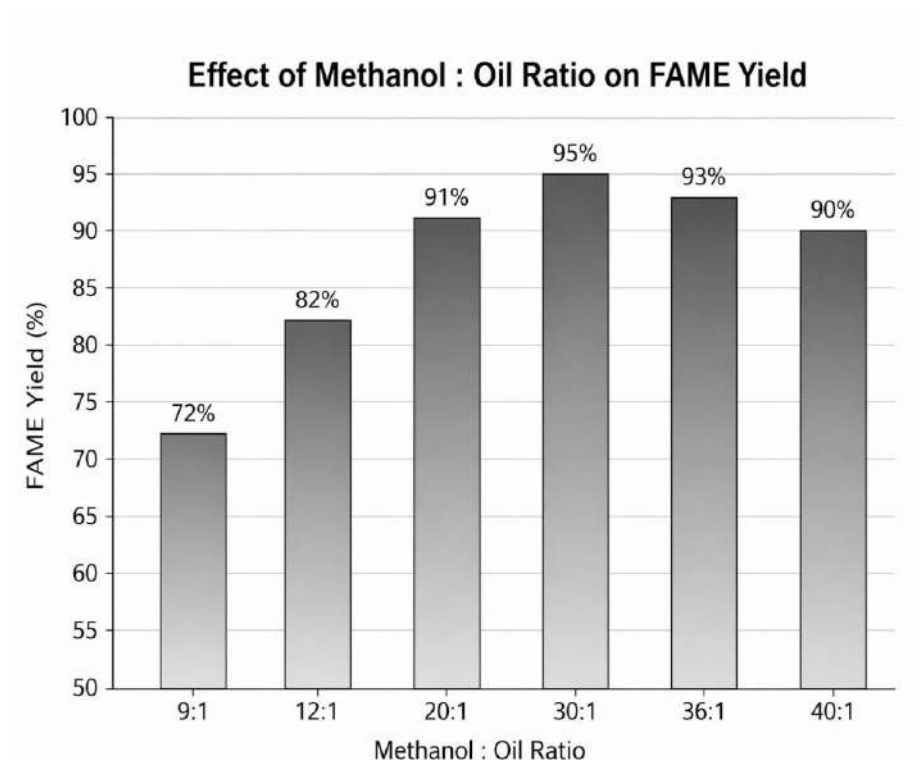


Fig.5.3: Effect of Methanol: Oil Ratio on Biodiesel Yield

CHAPTER-6
CONCLUSION

6. CONCLUSION

The outcomes of the studies suggest that factors including the molar ratio of methanol to oil, reaction time and catalyst loading significantly influenced the trans-etherification of waste cooking oil (WCO) to methyl ester. The impacts of the key operating actors on the development of biodiesel from used cooking oil were effectively studied. The methanol to oil molar ratio, temperature, and catalyst loading are the three most important variables. The optimal yield for the synthesis of fatty acid methyl esters from used cooking oil laws attained at 87.5% at 5-gram catalyst, methanol to oil molar ratio 9:1, temperature 100°C, and reaction time 4hr.

CHAPTER-7

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A
Major Project Report
on
“Synthesis of cellulose product from waste material by novel method for lignin
Separation”

Submitted in partial fulfillment of the
Requirements of the degree of

Bachelor of Engineering in Chemical Engineering (Sem VIII)

By
Mr. Pranay G. Baikar (Roll No. 05)
Mr. Aditya S. More (Roll No. 17)
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Maharashtra University of Mumbai

2025-26



CERTIFICATE

This is to certify that the project entitled **“Synthesis of cellulose product from waste material by novel method for lignin Separation”** is Bonafide work **“Aditi A. Vichare (25), Om G. Patil (19), Pranay G. Baikar (05), Aditya S. More (17)”** submitted to the University of Mumbai in partial fulfillment of the requirement for the word of the degree of **“Bachelor of engineering”** in **“Chemical Engineering”**.

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Project Guide

(S. J. Kulkarni)
HOD

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Principal

Internal Examiner

External Examiner

Project Report Approval For B.E.

This project report entitled (**Synthesis of cellulose product from waste material by novel method for lignin Separation**) by (**Aditi A. Vichare, Om G. Patil, Aditya S. More, Pranay G. Baikar**) of **Chemical Engineering**.

Examiners

1. _____

2. _____

Date: 25/04/2026

Place: Lavel

DECLARATION

We declare that this written submission represents my ideas in my own words and where other's ideas or words have been included, we have adequately cited and referenced the original sources. We also declared that We have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in my submission. Understand that any violation of the above will cause disciplinary action by the institute and can also evoke penal action from the sources which have thus not been cited or from whom proper permission has not been taken when needed.

Mr. Om G. Patil

Mr. Aditya S. More

Mr. Pranay G. Baikar

Ms. Aditi A. Vichare

Date: / /

ABSTRACT

This project generally aims to explore the sustainable production of microcrystalline and nanocrystalline cellulose from lignocellulosic waste materials such as sawdust and sugarcane trash. The biomass has been pretreated prior to extraction with size reduction, washing and drying to expand their surface area and to remove external dirt from the raw material. Pre-treatment is completed on the raw material by applying 4% NaOH solution and heating to 80–85 °C for 1.5 hrs in order to solubilize the lignin and hemicellulose. The resultant product is filtered and washed to remove any remaining alkaline solutions. Bleaching of the cellulose was completed using sodium hypochlorite (NaClO) 1.5%, heated to 65–70 °C and held for 1-hour in order to remove any residual lignin. All samples were subsequently washed several times until a neutral pH was achieved prior to being dried at 60 °C to produce the final cellulose product. The extraction of cellulose was confirmed through the use of Fourier Transform Infrared (FTIR) Spectroscopy to investigate the chemical structure and functional groups of the extracted cellulose. The FTIR spectrum reflects the cellulose molecules an O–H stretching band (3463 cm^{-1}), C–H stretching band (2917 cm^{-1}) for wood dust. And for Sugarcane Bagasse it shows O–H stretching band (3408 cm^{-1}), C–H stretching band (2902 cm^{-1}). FTIR spectrum shows that these both the sample detect the cellulose.

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CHAPTER 1

INTRODUCTION

1.INTRODUCTION:

The growing interest in sustainable materials and the need to decrease environmental pollution is leading to an exponential interest in valorizing agricultural and industrial waste. Cellulose and lignin are found in high abundance as biopolymers on Earth, comprising lignocellulosic biomass, which is often regarded as a waste and overlooked, but holds enormous potential. The facile separation of lignin and cellulose is important for converting this biomass to valuable products. This project plans to develop cellulose-based products from waste using a novel separation for lignin. Most delignification methods typically utilize harsh chemicals that are sometimes energy-intensive, degrading the cellulose, in addition to having environmental impacts. In this paper, it discusses a novel delignification method that is environmentally friendly, cost-effective, and scalable, in consideration of greener methodologies for biomass processing. This procedure promotes a circular bioeconomy by extracting pure cellulose from waste feedstocks (agricultural waste, paper sludge, and sawdust) and also recovering lignin as a value-added co-product. The cellulose could find markets in applications like biodegradable packaging or in a medical capacity, and the lignin could serve as a precursor in biofuels, adhesives, and carbon fiber. For example, sugarcane bagasse, which is the cellulosic residue from extracting juice from the sugarcane plant is comprised of 37-50% cellulose, 20-30% hemicellulose, and 20-26% lignin (al., 2011) wood dust, which is a byproduct from milling is on average 40-50% cellulose, 25-35% hemicellulose, and 20-30% lignin (the composition is variable depending on the specification of the wood) (Budiyanoro & Yudhanto, 2024). Sugarcane bagasse contains slightly less lignin than wood dust; nevertheless, both could serve as feedstocks for bioplastics, biofuels, and composite materials with wood dust being a superior feedstock for strength-properties and sugarcane bagasse being superior for availability and handling properties. Cellulose-based products have increased exponentially in their importance due to their versatility and sustainability. Being a very common organic compound, cellulose is a renewable resource-based, biodegradable material, which makes its usage viable in many applications, including food packaging, absorbent materials, drugs, etc., and many more (Abolore et al., 2023). The cellulose available for making non-toxic and biodegradable polymers offers a viable option for a sustainable future in place of traditional petroleum-based plastics. The versatility of cellulose material varies from its usage as a structural element and water retention material in absorbent materials to its usage as a core material in drug recipes (Hubbe et al., Jafari et al., 2024). However, lignin separation from biomass is a very difficult process, with significant challenges making cellulose extraction a complex process. Lignin, a

complex aromatic polymer, is intimately mixed with both hemicellulose and cellulose in lignocellulosic biomass, which gives rigidity to plant cell walls (Abolore et al., 2023). To extract cellulose, lignin separation is a must, which is traditionally carried out using high energy-intensive and environmentally harmful processes involving chemical-based processes (Hubbe et al., 2019). Effective lignin removal is a key factor in the economic viability and sustainability of biomass processing in industries that aim to produce valuable cellulose-based materials (Lan & Luterbacher, 2019). The focus of recent research is on the development of green pretreatment methods for biomass processing with a focus on sustainability while maintaining the value of lignin for future use (Abolore et al., 2023). The complex nature of lignin and its variability from biomass source to source are challenges that are yet to be overcome for efficient removal (Mateo et al., 2025). The value of lignin for use in high-value materials, such as aromatic chemicals and high-carbon content materials, is a key driver for overcoming these challenges.

To evaluate the capability of the separation process and cellulose product of the method various advanced characterization techniques are used:

- FTIR (Fourier Transform Infrared Spectroscopy) is the method used to evaluate the functional groups and confirm the chemical modification of cellulose and lignin.
- XRD (X-ray Diffraction) is the technique to inform the crystalline structure and confirm the degree of crystalline of the cellulose that was separated.
- SEM (Scanning Electron Microscopy) is used to characterize the surface morphology and to visualize the microstructural changes from the separation.

The analytical methods provide a comprehensive understanding of the material properties of cellulose and objectively confirm sufficient support of the effectiveness of the new method with promise for scale-up and potentially for industrial application.

1.1 About wood dust:

Wood Dust Wood dust is an abundant by-product of sawmills, carpentry shops, and furniture making shops. Wood dust is usually viewed as waste, and can lead to environmental or health concerns if not disposed of properly, or inhaled. Wood dust contains a high potential percentage of cellulose (up to 60% depending on species and processing), making it suitable for cellulose extraction. The fibrous, porous and similar nature of wood dust, combined with the compatibility of an alkaline and bleach treatments and high purity cellulose extraction, allow for lignin to easily be removed. Using wood dust as a raw material for cellulose extraction not

only creates added value from industrial waste, but develops a sustainable material at low cost.



Fig.1.1.1 wood dust

1.2 About Sugarcane bagasse:

Sugarcane Bagasse Sugarcane bagasse is a fibrous residue that remains after extracting the juice of sugarcane to produce sugar. Bagasse is readily available in sugarcane producing regions, is generally underutilized, and is often burned-to-pollute the environment. Sugarcane bagasse contains about 40-50% of cellulose, while also containing some hemicellulose and lignin, both with high value, and demonstrates excellent biomass valorization potential. The fibrous structure of bagasse is very porous, which allows the reagent to be easily absorbed during chemical treatment processes and potentially leads.



Fig.1.2.1 sugarcane bagasse

1.3 comparison of raw material selection:

Wood dust and sugarcane bagasse were selected as raw materials because both are abundant waste sources rich in cellulose (~40–50%), contain relatively low ash, and are highly compatible with novel green delignification techniques such as ozonation and alkaline treatments. They offer an excellent balance between yield, purity, cost, and sustainability compared to other lignocellulosic wastes.

Table 1.3.1 comparison of raw material:

Sr. no.	Raw material	Yield%	Composition%	Justification
1.	Wheat straw	35–40	20-30	Cheap but high ash, lower fiber strength, moderate yield.
2.	Wood dust	40–50	25–30	Waste, low silica, strong fibers, sustainable industrial by-product.
3.	Rice husk	35-40	25-30	High silica content complicates purification, low yield.
4.	Cotton	85-95	<5	Almost pure cellulose but costly and less sustainable as raw waste.
5.	Bamboo	30-40	20-30	Fast-growing renewable source; denser fibers; requires mechanical pretreatment.
6.	Sugarcane bagasse	40–50	25–30	Agro-industrial waste, easy to process, high yield, suitable for green extraction.

1.3 Application:

Cellulose from trash and a new, effective way to get lignin will be used as raw material in a number of industries. Cellulose made from wood waste and sugarcane bagasse, using a new way to get lignin, will be used to make high-value products in many different fields. In the packaging industry, for instance, cellulose will be turned into biodegradable films or containers that can be used instead of plastics made from petroleum. In the textile industry, purified celluloses would be turned into feedstock for making sustainable fibers and specialty papers. High-quality cellulose would be processed to be a biocompatible and non-toxic feedstock for drug delivery systems, wound dressings, and tissue engineering scaffolds in pharmaceuticals and biomedical applications.

1.5 Objective:

1. To remove cellulose from agricultural waste products like wood dust or sugarcane bagasse.
2. To use chemical treatment techniques or methods to eliminate the lignin and hemicellulose from the raw material.
3. To create and implement a novel technique or methods for effective lignin separation using the least amount of chemicals.
4. To produce purified cellulose with a higher quality and yield.
5. To use Fourier Transform Infrared Spectroscopy (FTIR) to investigate and verify the existence of cellulose in a product.
6. To study the impact of lignin removal of bleaching and alkali treatment.
7. To efficiently use waste materials and transform them into products with increased value.
8. To create an economical and environmentally friendly beneficial cellulose extraction method.

CHAPTER 2

LITERATURE REVIEW

2.LITRATURE REVIEW:

The increasing dependence on non-renewable fossil fuels has resulted in notable environmental challenges such as air pollution and the greenhouse effect, highlighting the urgency for alternative energy sources. Cellulose products have grown massively in relevance because of their versatility and sustainability (Rusdin et al., 2024). As a very common organic compound. Cellulose is renewable resource-based and biodegradable, making it suitable for several uses ranging from food packaging, absorbent materials, and drugs to others lignin separation from biomass is exceedingly difficult, with major challenges making cellulose extraction highly complex. Lignin, a complex aromatic polymer, is entangled with hemicellulose and cellulose in lignocellulosic biomass, adding structural strength to plant cell walls Cellulose isolation requires lignin removal, which is conventionally achieved using high-energy-demanding and environmentally harmful methods based on chemical use Effective lignin separation is fundamental to the economic feasibility and sustainability of biomass processing in industries that seek to make valuable cellulose-derived products (Jaturapiree et al., 2024). The potential of lignin, however, to be used in high-value products, e.g., the generation of aromatic chemicals and high-carbon content materials, underscores the need to address these challenges. The main advantages of using these products from natural resources are bio-degradability and ease of handling. Deforestation of trees for the production of cellulose can be reduced when cellulose is extracted from agricultural waste. Sugarcane is a well-known material which produces sugar. Sugarcane bagasse, a fibrous by-product of sugarcane processing, is traditionally used as disposed of waste. Also, it is highlighted in science that it is rich in cellulose. A high amount of SCB is generated annually which can impact the environment such as if it is burning causing air pollution, groundwater contamination, greenhouse gas emission (Chen et al., 2022). The forestry and wood processing sectors produce significant amounts of wood dust, which often poses a disposal challenge. composition is mainly cellulose, lignin, and hemicellulose, but also includes numerous extractives such as resins and terpenes, which vary significantly by wood species. The severity of the risk, therefore, is directly proportional to both the concentration of airborne dust and the specific species being processed, with hardwood dust generally considered more hazardous. Consequently, industrial safety regulations emphasize the use of Local Exhaust Ventilation (LEV) systems and strict exposure limits to minimize the

accumulation of both airborne dust (for respiratory health) and settled dust, which poses a significant combustible dust explosion hazard (Chen et al., 2022).

2.1 Cellulose, Hemicellulose, Lignin %:

Sugarcane bagasse (SCB) a fibrous waste material with a high cellulose content (40–50%) is usually used for the production of cellulose. Wood dust and sugarcane bagasse are lignocellulosic biomasses most found for their applications in sustainable material production and bioenergy production because they are rich in cellulose, hemicellulose, and lignin (Mhd. Ramle et al., 2024). Wood dust, as a waste product, typically contains 40–50% cellulose, 25–35% hemicellulose, and 20–30% lignin, although the composition depends on wood specification. Wood hemicellulose consists of branched polysaccharides that can be easily broken down than cellulose while lignin responsible for rigidity and hydrophobicity (Kumar et al., 2008). On the other hand, sugarcane bagasse, the cellulosic residue remaining after the juice extraction process, contains 37–50% cellulose, 20–30% hemicellulose, and 20–26% lignin. The bagasse hemicellulose is made up of arabinoxylan, which is rich in xylose and arabinose and making it valuable for fermentation-based bioprocesses. Lignin in bagasse is strongly linked with the carbohydrate matrix, and pretreatment is essential for effective enzymatic hydrolysis. Even though sugarcane bagasse contains slightly less lignin than wood dust, it is a more sustainable option because it is so abundant as a crop residue. Both are promising sources of feedstocks for bioplastics, biofuels, and composite materials, where wood dust is the preferred choice because of its structural strength and sugarcane bagasse due to its availability and ease of handling (Springer, 2024).

The cellulose microfibrils were isolated from the SCT by a two-step chemical treatment process (alkali pretreatment with sodium hydroxide followed by sodium hypochlorite bleaching) at different operating parameters. Also did an ozonation method using an ozonator machine to remove residue of lignin. Ozone preferentially attacks the aromatic rings and double bonds found in lignin molecules. The primary effect of ozone pre-treatment is to make the remaining cellulose more accessible for subsequent steps, such as enzymatic hydrolysis, which is necessary to convert cellulose into high-value materials. By removing the lignin barrier, ozone significantly increases the surface area and porosity of the remaining biomass (MDPI, 2023).

Different characterization techniques such as Fourier Transform Infrared spectroscopy (FTIR),

X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) were used for the analysis of surface functional groups, crystallinity index, surface morphology, and the internal structure of the raw as well as chemically treated biomass respectively (Josh 2024). The quantification results show the optimum process conditions for the alkali pretreatment as 5% (w/v) alkali (NaOH) concentration, and 6 h process time and for bleaching as 20% NaClO₂ and 8 h process time. This indicates the effective removal of hemicellulose and lignin. This confirms lignin removal and detects cellulose. The cellulose is generally used in pharmaceutical, food, cosmetic, paper and plastic industry industries.

2.2 Removal of lignin:

Agricultural waste products like rice husk, wheat straw, sugarcane bagasse and corn stalks (known as lignocellulosic biomass) have been identified as cheap renewable resources of lignocellulosic biomass. The majority of lignocellulosic biomass consists of cellulose, hemicellulose and lignin, but the main component for future applications will be cellulose. Although it is possible to extract cellulose from these materials, it is not an easy process due to the fact that cellulose is locked inside of a rigid matrix caused by the bonding of lignin to cellulose. Historically, the removal of lignin has been accomplished by utilizing high-temperature and/or high-pressure methods that utilize harsh chemicals (acids or bases), which accomplish the removal of lignin but can also result in environmental contamination and damage to the structural integrity of cellulose. Because of the disadvantages of using chemical methods for the removal of lignin, there has been an increasing focus on the use of more environmentally friendly and effective lignin removal methods using deep eutectic solvents, ionic liquids, organosolv processes and/or microorganisms. These alternative methods allow for the removal of lignin in a selective manner while preserving the structural integrity of cellulose.

2.3 How FTIR analysis works:

After the removal of lignin, the remaining cellulose is typically washed, bleached and purified so that a more usable and higher quality product can be achieved. One protocol for verifying the successful extraction of cellulose is the use of Fourier Transform Infrared (FTIR) spectroscopy as an analytical method. FTIR spectroscopy measures the way different types of chemical bonds absorb energy when infrared light is shone through a sample. The result of this process is a spectrum composed of distinct peaks that define the type and number of chemical bonds contained within the sample being analyzed. For example, there are distinct peaks associated

with the presence of cellulose. In FTIR specimens of cellulose, there is a broad peak between the wavenumbers of 3330 and 3400 cm^{-1} , which corresponds to the stretching of O–H functional groups. There is also a peak at approximately 2900 cm^{-1} , which corresponds to C–H stretching vibrations. Additional peaks that provide evidence for the presence of cellulose in FTIR spectra occur at 1160 cm^{-1} and between 1030 and 1050 cm^{-1} , which are associated with C–O–C and C–O bonds in the cellulose polymer; specifically, the β -1,4-glycosidic linkage. One of the most important peaks in a FTIR spectrum of cellulose is the peak that occurs near 897 cm^{-1} , which is often referred to as the characteristic peak of cellulose. The removal of lignin can be established by observing a reduction or the disappearance of peaks associated with aromatic compounds that usually appear in the FTIR spectra at approximately 1510 cm^{-1} and 1600 cm^{-1} , respectively, and a peak associated with carbonyl (C=O) groups that usually appears in FTIR spectra at approximately 1730 cm^{-1} . Therefore, by comparing these modifications of the FTIR spectrums, it is possible to assess both the existence of cellulose and the extent to which lignin has been removed from cellulose.

2.4 Various methods of synthesis:

1. Alkaline Treatment method:

Using sodium hydroxide (NaOH) to treat agricultural waste is the most common treatment method. Sodium hydroxide breaks down lignin and hemicellulose in the waste. Bleaching, with either hydrogen peroxide or sodium hypochlorite, removes any remaining impurities from the agricultural waste after treatment. This method is simple and effective, but it is not very environmentally friendly due to the use of chemicals.

2. Acid hydrolysis method:

Strong acids such as sulfuric acid, H_2SO_4 , may be utilized in this way to eliminate amorphous areas and to partially decompose hemicellulose and lignin. This technique has the most utility during production of microcrystalline or nanocellulose. This method can be very harsh on the cellulose and may lead to cellulosic degradation if not closely monitored.

3. Organogold Method:

The above method incorporates the use of organic soluble such as ethanol or methanol to dissolve lignin, which is performed with the aid of either an acid catalyst or without an acid catalyst. High-purity lignin can be recovered through this process as well as improving the overall physical integrity of cellulose. Furthermore, it is considered an environmentally friendly alternative to conventional methods using chemicals.

1. Ultrasound assisted method:

Ultrasound waves create cavitation (tiny bubbles), which disrupts the biomass structure and enhances the penetration of chemicals, improving lignin separation.

2. Ionic liquid method:

Biomass contains very strong bonded structures that can be disrupted using ionic liquid salts in their liquid state. Ionic liquids include an effective lignin solvent, allowing for the easy separation of cellulose once dissolved; however, due to the expense associated with the process, the method is not applicable at a large scale.

2.5 Why we used Alkaline treatment:

Because of its effectiveness at removing lignin from agricultural waste without causing significant degradation to cellulose, alkaline treatment was the method you selected in this case. Sodium hydroxide (NaOH) breaks down the bonds between cellulose and lignin and dissolves much of the hemicellulose when applied to the biomass. The structure becomes more open and allows for increased separation of the cellulose fibers, thereby producing higher yield and purity of cellulose. Another reason why alkaline treatment is convenient is that it is more affordable and much easier to perform than newer techniques, such as ionic liquids or deep eutectic solvents, on a laboratory scale. It can be conducted without specialised equipment and common chemicals are used; therefore, it is a highly preferred method for many experimental and/or small-scale studies. Finally, alkaline treatment generally produces fast and repeatable results, which is essential when establishing a method for reproducible results in your project. The process substantially prepares your sample for subsequent processing, such as bleaching and FTIR, by providing clear evidence of lignin removal and presence of cellulose in the sample.

CHAPTER 3

MATERIALS AND METHODOLOGY

3.MATERIALS:

Institute of Wood Science and Technology Bangalore, Karnataka, India has kindly presented wood powder and cellulose. Sugar Cane Bagasse and wood dust were gathered in the vicinity of Davangere, Karnataka, India. Sodium hypochlorite (NaClO), and sodium hydroxide (NaOH) were acquired from SD Fine Chemicals. All chemicals were used without any further purification and were 98.0% pure.

3.1 Raw Materials:

3.1.1 Wood Dust

Wood Dust used to make cellulose because they are easy to find and do not cost a lot of money and also have a lot of components. These materials are known as Lignocellulosic Biomass. This biomass is made up of cellulose, hemicellulose and lignin. This cellulose content is usually 40 to 50 percent. Therefore, it is good for making cellulose after they have been treated with chemicals. Wood dust is a material to use because it is very fine, so, it has a more surface area. When it has a surface area chemicals like sodium hydroxide and sodium hypochlorite can get in and do their job better. This makes it easier to remove lignin and other things that are not wanted. This process is called Delignification. The thing about wood dust is that it comes from different types of wood. It can also have some things in it that are not wanted, like resins or oils.

3.1.2 Sugarcane bagasse

Sugarcane bagasse is the leftover part after juice is extracted from sugarcane in sugar industries. It is a raw material because it is available in large quantities and is renewable. Also has a fibrous structure. This helps reagents to penetrate easily during treatment. As a result, cellulose can be separated efficiently from lignin and hemicellulose. Sugarcane bagasse is a waste from farming. It is produced in amounts but often not used properly. This makes it an important raw material for sustainable research. Wood dust and sugarcane bagasse are chosen for two reasons. Firstly, they have a lot of cellulose. Secondly, they help in making use of waste. They convert waste from farms and industries into products. Using these materials supports sustainability. These raw materials are preferred for reasons. They are available, affordable and suitable for chemical processing. They also have benefits. They help in waste valorization by converting industrial residues into valuable products. Their use supports the principles of sustainability. Aligns with modern approaches, in Green Chemistry, where waste materials are converted into useful resources.

3.2 Chemicals:

3.2.1. Sodium Hydroxide (NaOH)

Sodium hydroxide is very important to extract cellulose from Lignocellulosic Biomass. The use Sodium hydroxide because it is a chemical that helps to remove lignin and some of the hemicellulose from the biomass. Lignin is a material that acts like a glue holding the cellulose fibers together and making the plant strong. The biomass with Sodium hydroxide at the temperature and concentration the Sodium hydroxide breaks the bonds in the lignin, which makes it break down and dissolve in the solution. At the time the hemicellulose, which dissolves easily in alkaline conditions is also partially removed. Sodium hydroxide also helps to swell the fibers, which means it goes into the cellulose structure and makes the fibers spread out more, increasing the surface area. This change in the structure of the cellulose makes it easier to treat the cellulose such as when we bleach it. After we treat the biomass as shown in fig 3.2.1, the solid part that is left is mostly cellulose and the lignin and other impurities that dissolved form a liquid called black liquor. So Sodium hydroxide is necessary for breaking down the biomass and removing most of the parts that're not cellulose. Sodium hydroxide plays a role in this process and without Sodium hydroxide it would be hard to extract the cellulose from the Lignocellulosic Biomass.



3.2.2. Sodium Hypochlorite (NaClO)

sodium hypochlorite is used in the bleaching stage to purify cellulose. It helps remove the lignin that is left after alkali treatment. It helps to enhance or brighten the colored of cellulose. The fig 3.2.2 shows it works really well because it breaks down lignin. The mix of sodium hypochlorite with water turns into acid which help to make cellulose white. It does this by reacting with the parts of lignin that make it colored. These parts get broken down into pieces that can dissolve in water. Therefore as a result the cellulose becomes really white and pure.

The bleaching process makes the cellulose look better and also makes it better for making things like paper and clothes. We have to be careful when using sodium hypochlorite. If the use much of it or its too hot or we use it for too long it can hurt the cellulose. This can make it weak and not useful anymore. Also, when we use sodium hypochlorite it can make some chemicals. These chemicals can hurt the environment so we have to be careful when we get rid of them. Sodium hypochlorite is really useful for making cellulose pure and white. It should be use in a way that's safe for the environment.



Fig.3.2.2 NaClO

3.3 Experimental Setup:

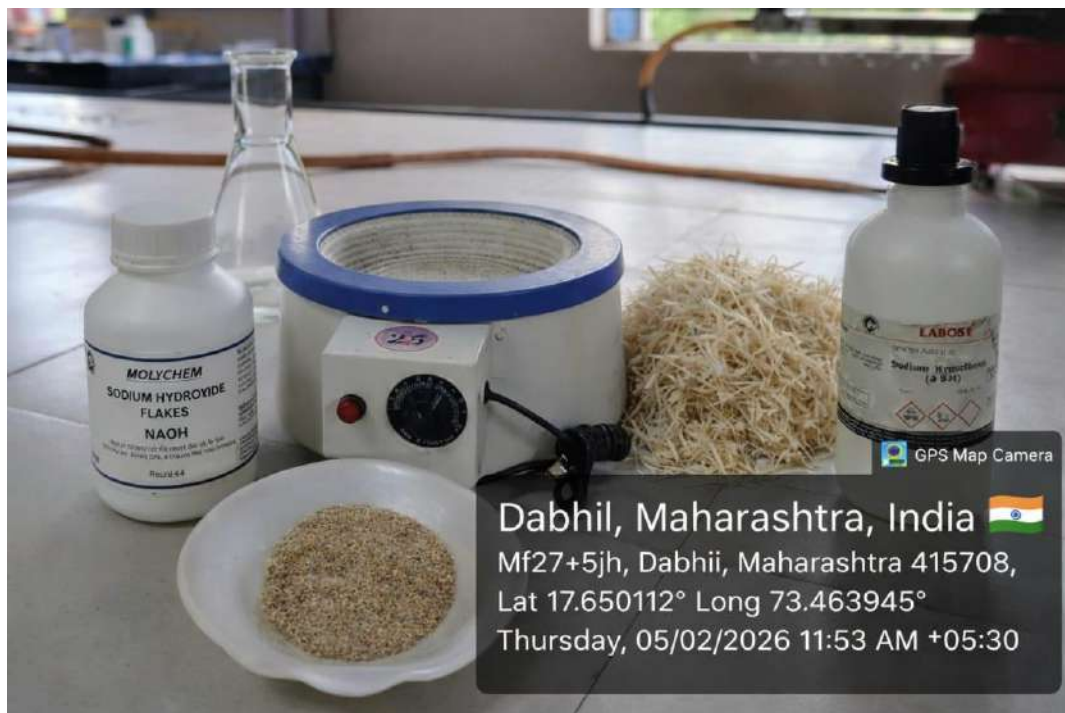


Fig.2.1 Experimental setup

- **Sugarcane bagasse:**
Sugarcane bagasse is the raw product for this project. It is readily available and it contains a high proportion of cellulose.
- **Wood dust:**
Wood dust is the raw product for this project. It is readily available and it contains a high proportion of cellulose.
- **NaOH Flakes:**
We use NaOH to treat lignocellulosic biomass (e.g. wood dust, sugarcane bagasse). It breaks down the complex structure of material. It helps to remove lignin
- **NaClO:**
It is used as a bleaching agent after lignin removal. It helps to get the colour white and purify the cellulose by oxidizing residual lignin.
- **Heating machine:**
Used to accelerate the lignin breakdown. Uses at common temperature to 60–100°C
- **Conical Flask:**
Used to store the solution.

- **Stirrer:**
To mix the solution properly.

3.4 Methodology:

3.4.1. Wood dust:-

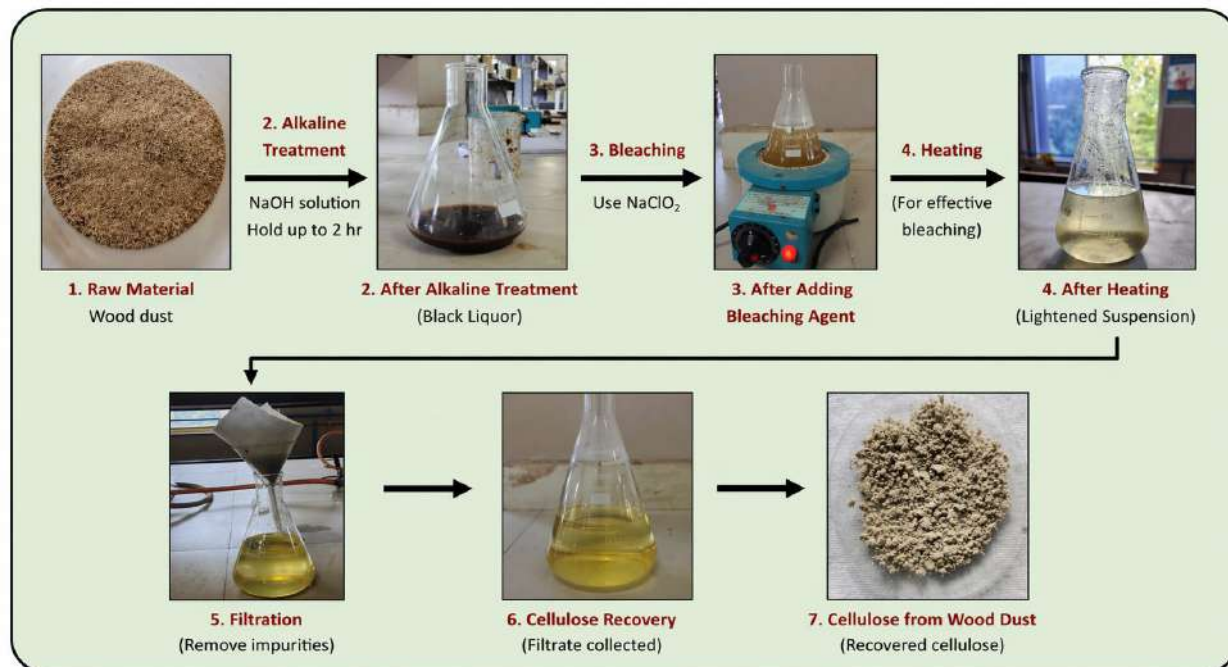


Fig. 3.4.1 cellulose work flow process from wood dust

The fig 3.4.1 presents a step-by-step isolation process of cellulose from the raw material, wood dust, involving chemical treatments and purification procedures. First, the raw material is put under an alkaline treatment by a sodium hydroxide (NaOH) solution; in this way, the substance stays in the alkaline solution for 2 hours. As a result of this process, lignin and hemicellulose are decomposed, resulting in dark liquid known as "black liquor." After that, sodium hypochlorite (NaClO) is used as a bleaching agent to remove the remaining lignin. In this case, heating is applied to enhance the process of oxidation, hence light suspension proves the purity of cellulose. After that, the solution with the dissolved components is filtered to obtain only the fibers of cellulose without any other substances dissolved in the liquid. The obtained filtrate is a suspension that contains purified cellulose. Finally, cellulose is dried at 60°C to obtain dry cellulose, as presented in the last stage. Thus, the procedure includes not only chemical but also thermal treatment of the substance, as well as mechanical filtration.

Overall, this method produces cellulose with better structural integrity and lower impurity content. This makes it suitable for further applications like biodegradable materials, composites, or chemical modification. The process also shows a cost-effective way to use lignocellulosic waste, like wood dust. This contributes to developing sustainable materials and making better use of waste.

3.4.2 Sugarcane bagasse:

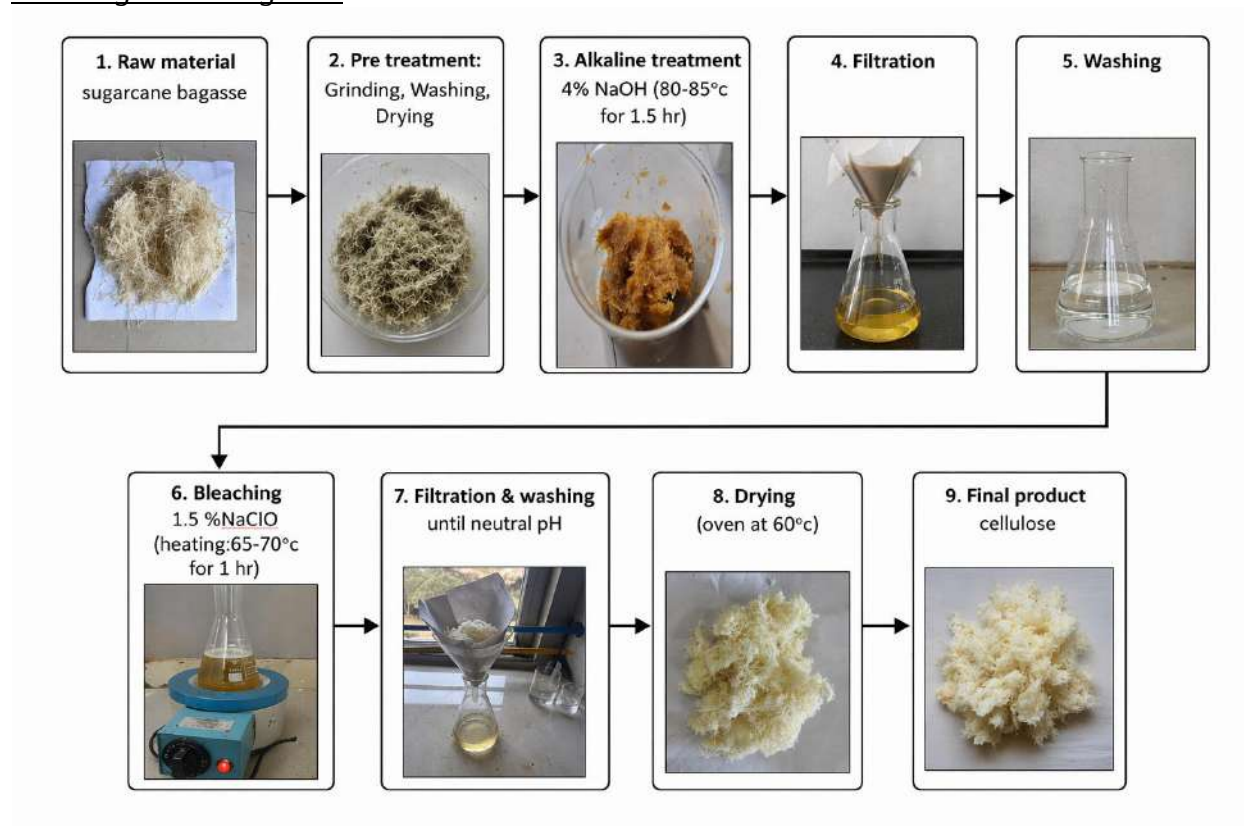


Fig 3.4.2 cellulose workflow process from sugarcane bagasse

Sugarcane bagasse is the material we start with. It has to go through some steps which are shown in fig 3.4.2. These steps are grinding, washing and drying. The grinding makes the particles smaller which increases the surface area of the sugarcane bagasse. The washing gets rid of the dirt and other impurities that can dissolve in water. The drying removes the moisture from the sugarcane bagasse so it is ready for the steps. Next the sugarcane bagasse that has been pre-treated goes through a treatment. This treatment uses 4% NaOH at a temperature of 80°C to 85°C for 1½ hours. This step is important because it breaks down the lignin and hemicellulose in the sugarcane bagasse. These are parts of the sugarcane bagasse that're not cellulose. The treatment loosens up the fibers. Increases the amount of cellulose in the sugarcane bagasse. After this treatment the mixture is filtered to separate the fibers from the impurities that have dissolved. The fibers that are left are washed well to remove any remaining alkali and other substances that can dissolve. After that the sugarcane bagasse material goes through a bleaching process. This process uses 1.5% NaClO at a temperature of 65°C to 70°C for 1hr. This step helps get rid of any lignin and coloring substances that're still in the sugarcane bagasse. This makes the cellulose material lighter and purer. The bleached sugarcane bagasse sample is filtered again that shows in fig 3.4.2. Washed many times until it has a neutral pH. This ensures that all the remaining chemicals are removed from the sugarcane bagasse. Finally the purified cellulose from the sugarcane bagasse is dried in an oven at 60°C. This removes the moisture. Produces the final solid product. This whole process changes the sugarcane bagasse, which's a waste product into purified cellulose. It does this through a series of treatment, purification and drying steps that are applied to the sugarcane bagasse.

Preparation of cellulose:

Delignification Reaction:

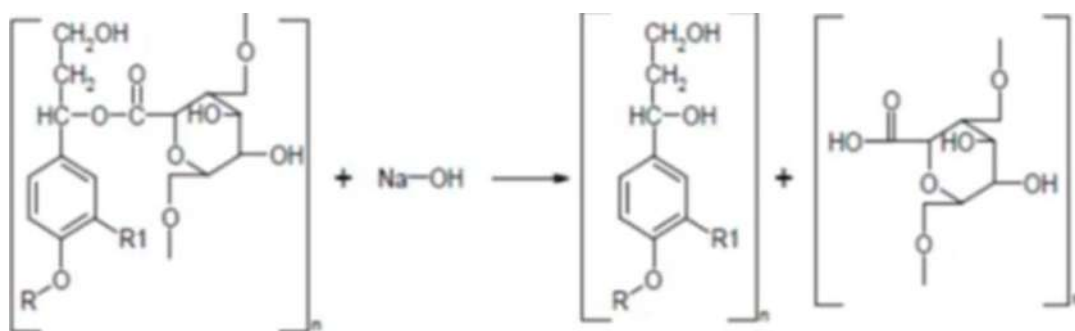


Fig 3.4.3 delignification

Delignification is the process of removing lignin from plant-based materials, primarily wood, to isolate cellulose for various applications. This process is essential in industries such as papermaking and biorefining, where lignin acts as a natural binder, providing rigidity and structural integrity. Used to extract cellulose.

Bleaching Reaction:

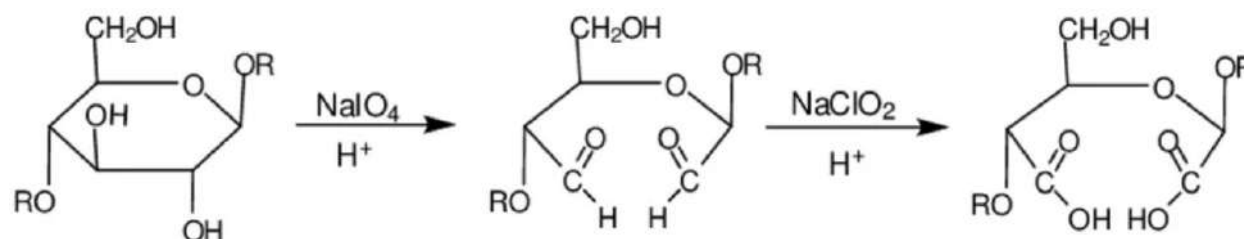


Fig 3.4.4 Bleaching

Bleaching is a chemical process used to whiten or remove color from materials and to disinfect surfaces by altering or breaking down colored compounds.

CHAPTER 4

RESULT AND DISCUSSION

4.RESULT:

Data for wood dust:

Weight of raw material (sawdust), $\square_1 = 35$ g

Weight of cellulose obtained, $\square_2 = 12$ g

Method used: Bleaching and conventional alkali treatment

Formula:

Yield =

$$\frac{\text{Weight of isolated cellulose}}{\text{Weight of initial sawdust}}$$

$$= \frac{12}{35} * 100$$

$$= 34.29 \%$$

Yield of cellulose from wood dust=34.29 %

A 34.29 % yield from 35 g of sawdust is reasonable. This means about one-third of the raw mass became isolated cellulose.



fig .4.1 cellulose from wood dust

Data for Sugarcane Bagasse:

Weight of Sugarcane bagasse, $\square_1 = 50$ g Weight
of cellulose obtained, $\square_2 = 23$ g

$$\frac{\text{Weight of isolated cellulose}}{\text{Weight of initial sawdust}}$$

$$= \frac{23}{50} * 100$$

$$= 46 \%$$

Yield of cellulose from sugarcane bagasse is 46%.

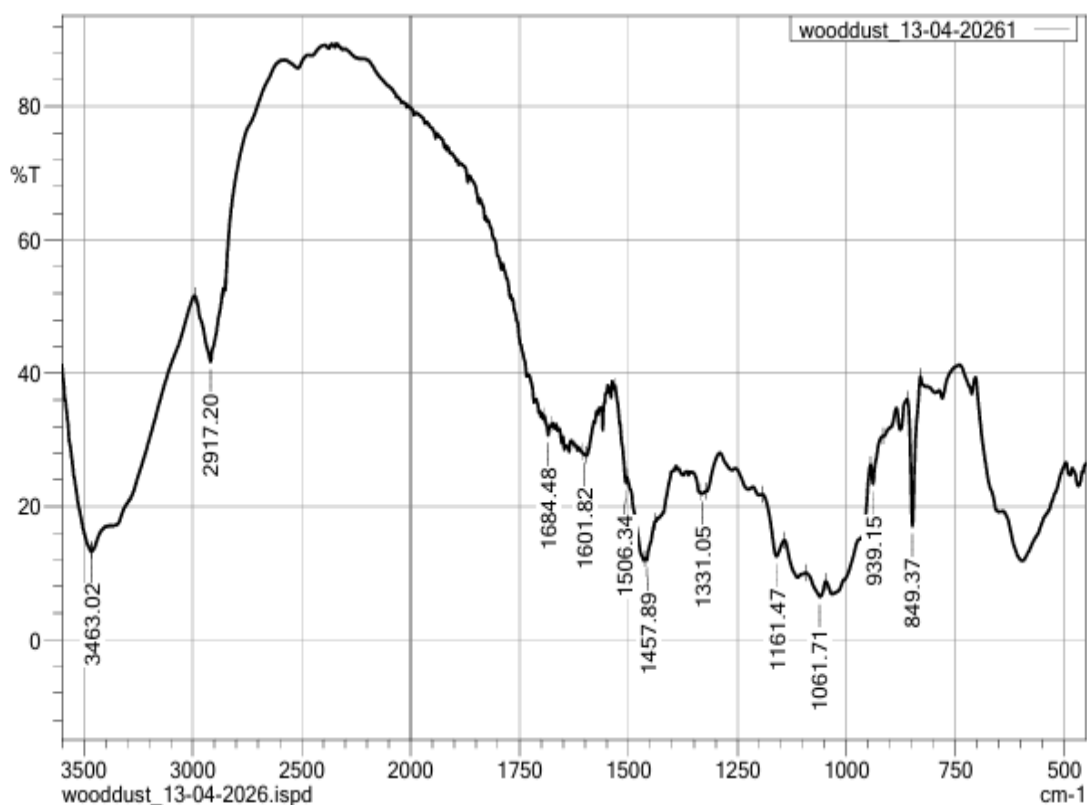


Fig.4.2 cellulose from sugarcane bagasse

4.1 DISCUSSION:

4.1.1 Wood dust

SHIMADZU



D:\25-26\wooddust_13-04-20261.ispd

wooddust_13-04-2026.ispd

	Item	Value
2	Sample name	wooddust
3	Sample ID	wooddust_13-04-2026
4	Option	
5	Intensity Mode	%Transmittance
6	Apodization	Happ-Genzel
9	No. of Scans	45
10	Resolution	4 cm ⁻¹

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area	Comment
1	849.37	17.08	20.32	860.77	830.84	2098.558	237.218	
2	939.15	23.41	3.74	944.85	916.35	2063.378	23.702	
3	1061.71	6.48	2.68	1094.49	1047.46	4319.319	59.229	
4	1161.47	12.55	4.88	1194.24	1142.94	4280.877	94.044	
5	1331.05	21.92	0.33	1336.75	1322.50	1110.263	2.446	
6	1457.89	11.99	1.25	1462.16	1439.36	1950.226	11.830	
7	1506.34	23.63	2.19	1530.57	1503.49	1882.000	19.786	
8	1601.82	27.74	0.20	1607.53	1598.97	616.302	0.801	
9	1684.48	30.71	2.21	1693.03	1677.36	1064.592	13.874	
10	2917.20	41.72	10.51	2991.31	2855.92	7164.120	689.118	
11	3463.02	13.36	0.11	3464.44	3461.59	246.786	0.150	

The FTIR spectrum of the wood dust sample shows peaks that indicate the presence of lignocellulosic components like cellulose, hemicellulose and lignin.

A broad peak around 3463 cm^{-1} shows O–H stretching vibrations, which're mainly from hydroxyl groups in cellulose. This peak means there is hydrogen bonding in the cellulose structure.

The peak near 2917 cm^{-1} represents C–H stretching vibrations of groups, which are commonly found in cellulose, hemicellulose and lignin but are particularly significant in cellulose and hemicellulose

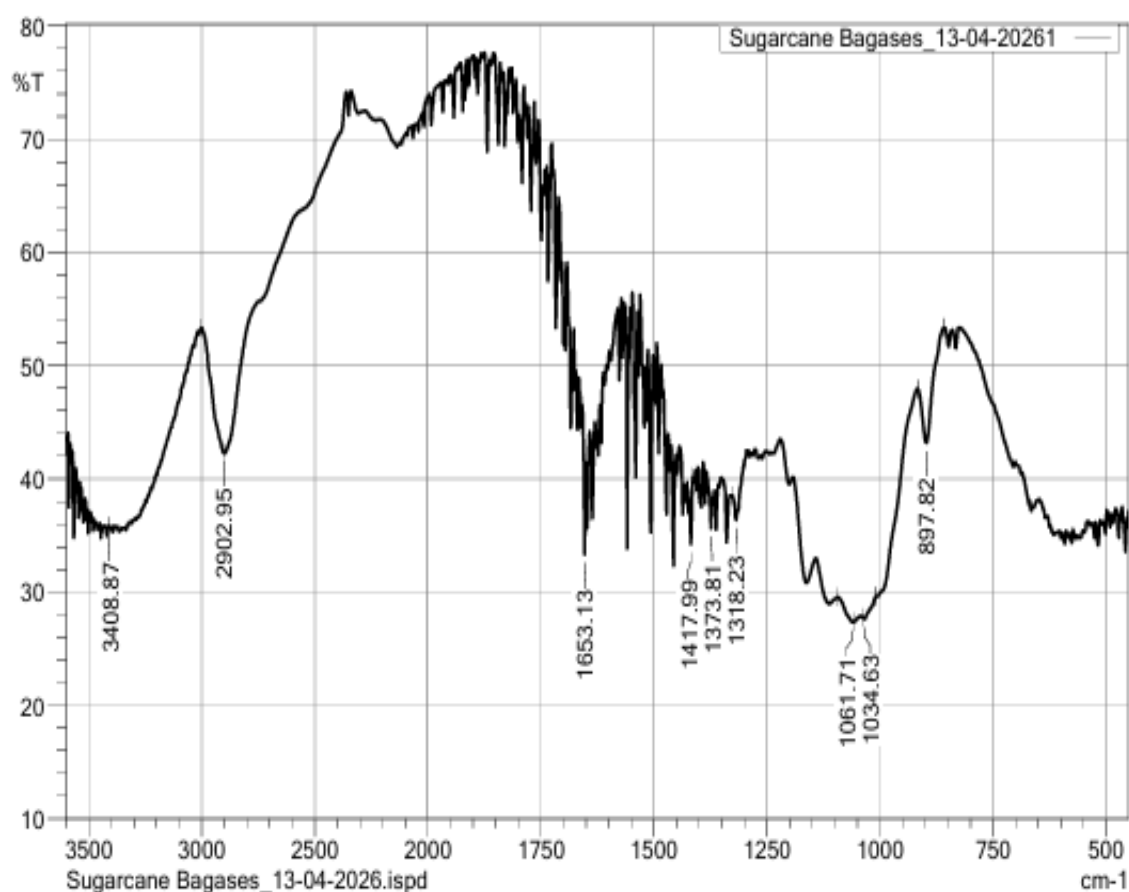
.In the wavenumber region the peaks at 1161 cm^{-1} and 1061 cm^{-1} are important as they are related to C–O–C and C–O stretching vibrations confirming the presence of cellulose especially the β -1,4-glycosidic linkage in cellulose.

The peak around 939 cm^{-1} further supports the presence of cellulose due to its association with bonds in cellulose.

These peaks clearly indicate that cellulose is a component, in the wood dust sample and cellulose hemicellulose and lignin are present. The FTIR spectrum shows that the wood dust sample contains cellulose, hemicellulose and lignin.

4.1.2 Sugarcane bagasse

SHIMADZU



D:\Y25-26\Sugarcane Bagases_13-04-20261.ispd

Sugarcane Bagases_13-04-2026.ispd

	Item	Value
2	Sample name	Sugarcane Bagases
3	Sample ID	Sugarcane Bagases_13-04-2026
4	Option	
5	Intensity Mode	%Transmittance
6	Apodization	Happ-Genzel
9	No. of Scans	45
10	Resolution	4 cm-1

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area	Comment
1	897.82	43.18	6.48	916.35	857.92	3030.757	147.407	
2	1034.63	27.63	0.23	1036.06	1008.98	1936.064	5.484	
3	1061.71	27.33	0.23	1094.49	1058.86	2550.367	2.292	
4	1318.23	36.38	3.13	1326.78	1298.28	1745.912	40.669	
5	1373.81	35.65	2.81	1376.66	1370.96	358.369	7.614	
6	1417.99	34.26	4.70	1420.84	1413.71	449.990	16.455	
7	1653.13	33.34	9.80	1658.83	1650.28	506.764	27.900	
8	2902.95	42.30	0.16	3001.28	2901.53	5240.299	35.582	
9	3408.87	35.78	0.05	3410.29	3407.44	182.977	0.074	

The sugarcane bagasse was analyzed using FTIR analysis. This was done to see what functional groups are in the sugarcane bagasse. The test was done in the range of 4000–500 cm^{-1} .

The results show a peak at 3408 cm^{-1} . This peak is because of the O–H stretching vibrations. This means that there are hydroxyl groups in the cellulose and hemicellulose of the sugarcane bagasse. These groups are also connected by hydrogen bonds.

There is another peak at 2902 cm^{-1} . This peak is because of the C–H stretching of groups. These groups include methyl and methylene groups.

The sugarcane bagasse also shows a peak at 1653 cm^{-1} . This peak is due to the C=O stretching vibrations. This could be because of the lignin in the sugarcane bagasse or because of water that is absorbed.

The peaks at 1517 cm^{-1} and 1417 cm^{-1} are because of the ring vibrations. This confirms that the sugarcane bagasse contains lignin. The peak at 1373 cm^{-1} is due to the C–H bending. This shows that the cellulose in the sugarcane bagasse has a structure.

The peak at 1318 cm^{-1} is because of the C–O stretching vibrations of alcohol groups.

The sugarcane bagasse also shows peaks at 1061 cm^{-1} and 1034 cm^{-1} . These peaks are because of the C–O–C stretching vibrations and ether linkages. These are present in the cellulose and hemicellulose of the sugarcane bagasse.

The peak at 897 cm^{-1} shows that there are β glycosidic linkages between glucose units. This confirms that the sugarcane bagasse contains cellulose.

So the FTIR spectrum of sugarcane bagasse shows that it contains cellulose, hemicellulose and lignin. This means that sugarcane bagasse is a biomass. It is suitable for use in different industries. The sugarcane bagasse is a material for these industries because it is a lignocellulosic biomass.

CHAPTER 5

CONCLUSION

5.CONCLUSION:

This project proves that cellulose products can be produced from waste biomass with a novel method of lignin separation. The lignin separation process created was more effective, inexpensive, and eco-friendly compared to conventional chemical separation methods of lignin. The end-cellulose products can have uses in the future like: biodegradable items and paper products, and split into distinct uses as bio-composites created from cellulose. The project in general emphasized the possibilities for processing technologies to transform waste and other substrates to cellulose products, by means of innovative and environmentally friendly processing technologies and enable waste-to-wealth programs and initiatives for circular bio-economy.

As a result, we get a cellulose structure with better physical and chemical properties. The fact that lignin was removed effectively is clear from the FTIR analysis, which shows that the characteristic peaks of lignin are reduced or gone while the peaks that are typical of cellulose remain unchanged. This means that the structure of cellulose stays intact during the treatment process. The new method used in this research has advantages over traditional methods of separating lignin. It reduces the need for chemicals, is better for the environment and takes less time and energy to process. These features make the method more sustainable and cost-effective. Also the treatment makes it easier to access the hydroxyl groups in cellulose, which can make it more reactive and suitable for chemical changes.

In conclusion the research provides a sustainable and innovative way to produce cellulose from waste materials supporting the idea of recovering resources and green chemistry. The cellulose product made from waste biomass using the method is a significant step towards sustainable development and has many potential applications.

CHAPTER 6

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“Industrial wastewater treatment by using Acoustic Cavitation and H₂O₂ Additive”

Submitted in partial fulfillment of the
Requirement of the degree of
Bachelor of Engineering
by

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2025-26

CERTIFICATE

This is to certify that the project entitled **“Industrial wastewater treatment by using acoustic cavitation and H₂O₂ Additive”** is a bonafide work of Mr. Ambre Vedant Anant (Roll no.02) ,Mr. Bahutule Tejas Sanjay (Roll no.04), Mr. Kalambate Aditya Baliram (Roll no.12), Mr. More Prathamesh Sandesh (Roll no.18) submitted to the University of Mumbai in partial fulfillment of the requirement for the award of the degree of “Undergraduate” in “Chemical Engineering”.

(Dr.S.D.Ayare)
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Project Report Approval for B.E.

This project report entitled “**Industrial wastewater treatment by using acoustic cavitation and H₂O₂ Additive**” by Mr. Ambre Vedant Anant(02),Mr. Bahutule Tejas Sanjay(04), Mr. Kalambate Aditya Baliram(12),Mr. More Prathamesh Sandesh(18) is approved for the degree of **Chemical Engineering**

Examiners

- 1.
- 2.

Date:

Place: Lavel

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DECLARATION

I declare that this written submission represents my ideas in my own words and where others' ideas or words have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in my submission. I understand that any violation of the above will because of disciplinary action by the Institute and can also evoke penal action from the sources which have thus not been properly cited or from whom proper permission has not been taken when needed.

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ABSTRACT

This project focuses on the treatment of industrial wastewater using a combination of acoustic cavitation and hydrogen peroxide (H_2O_2) as an advanced oxidation process. Industrial wastewater contains harmful organic pollutants, high COD, and toxic chemicals that are difficult to remove by conventional methods. In this study, acoustic cavitation was applied using ultrasonic waves, which generate microbubbles that collapse and produce high temperature and pressure, forming highly reactive hydroxyl radicals ($\bullet\text{OH}$). The addition of H_2O_2 further increases the production of these radicals, enhancing the degradation of complex pollutants. The combined method showed higher efficiency compared to individual processes, resulting in significant reduction in COD, color, and toxic compounds. Optimum conditions such as ultrasonic power, H_2O_2 dosage, and reaction time played an important role in achieving better results. This hybrid treatment is simple, efficient, and eco-friendly, producing minimal secondary pollution. Therefore, it is a promising and sustainable method for industrial wastewater treatment. .

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CHAPTER - I

Introduction

1. Overview of Industrial Wastewater

Industrial wastewater is generated from a wide range of manufacturing and processing activities, and its composition varies depending on the type of industry. For example, textile industries release colored dyes and chemicals, pharmaceutical industries discharge complex organic compounds, and food processing units generate wastewater rich in organic matter. Each type of wastewater has different characteristics, making treatment more challenging and requiring specialized methods.

One of the major concerns with industrial wastewater is the presence of toxic and hazardous substances. These may include heavy metals such as lead, mercury, and cadmium, as well as synthetic chemicals that are resistant to natural degradation. When discharged without proper treatment, these pollutants can contaminate soil and water bodies, leading to long-term environmental damage and health risks for humans and animals.

Water pollution caused by industrial discharge affects aquatic ecosystems significantly. Pollutants reduce dissolved oxygen levels in water, harming fish and other aquatic organisms. Some chemicals can also bioaccumulate in the food chain, meaning their concentration increases as they move from smaller organisms to larger ones, eventually affecting human health through consumption of contaminated water or food.

In many developing countries, including India, rapid industrial growth has increased the volume of untreated or partially treated wastewater being released into rivers and lakes. Inadequate infrastructure, lack of strict regulations, and high treatment costs are some of the reasons for improper wastewater management. As a result, major rivers and water bodies are facing severe pollution problems.

To address these challenges, governments and environmental agencies have introduced strict discharge standards and guidelines for industries. There is also a growing emphasis on adopting cleaner production techniques and recycling wastewater within industrial processes. However, due to the complexity of pollutants, conventional treatment methods alone are often insufficient, highlighting the need for advanced and more efficient technologies like acoustic cavitation combined with chemical oxidation processes.

2. Limitations of Conventional Treatment Methods

Traditional wastewater treatment methods include:

- Biological treatment
- Chemical oxidation
- Adsorption
- Coagulation and flocculation

However, these methods have several limitations:

- Ineffective for complex and toxic pollutants
- High operational cost
- Slow reaction rates
- Production of sludge
- Incomplete degradation of contaminants

Modern industrial effluents contain complex chemical structures that are resistant to conventional treatment processes.

3. Need for Advanced Treatment Technologies

To overcome these limitations, advanced treatment technologies are required. One of the most promising methods is Advanced Oxidation Processes (AOPs).

AOPs work by generating highly reactive species like:

- Hydroxyl radicals ($\bullet\text{OH}$)
- Hydrogen peroxide (H_2O_2)

These radicals are powerful oxidants that can break down complex organic pollutants into simpler and less harmful compounds.

1.1. Advanced Methods for Wastewater Treatment 1.

1.1.1. Advanced Oxidation Processes (AOPs)

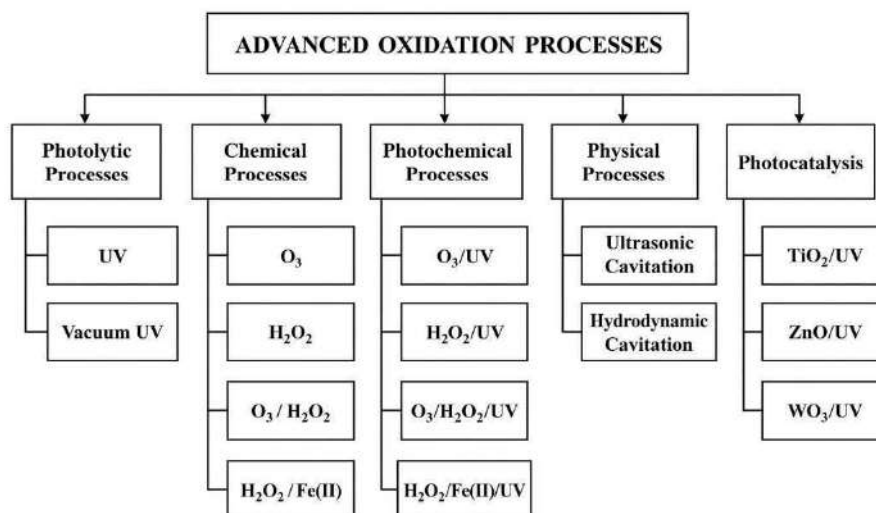


Fig.no.1.1.1(Advanced Oxidation Processes)

Advanced Oxidation Processes (AOPs) are among the most effective modern methods for degrading complex and non-biodegradable organic pollutants in wastewater. These processes rely on the generation of highly reactive hydroxyl radicals ($\bullet\text{OH}$), which have strong oxidizing power and can mineralize a wide range of contaminants into harmless end-products such as carbon dioxide and water. Common AOPs include ozonation (O_3), ultraviolet/hydrogen peroxide ($\text{UV}/\text{H}_2\text{O}_2$) systems, Fenton and photo-Fenton reactions, and photocatalysis using titanium dioxide (TiO_2). These methods are especially useful for treating industrial wastewater containing dyes, pesticides, pharmaceuticals, and phenolic compounds. Although AOPs are energy-intensive and may require careful control of pH and oxidant dosage, they offer rapid reaction rates and high treatment efficiency compared to conventional chemical treatments. The AOP has been segmented into various categories. The most successful of these are those that involve an oxidizer, such as hydrogen peroxide, combined with a catalyst and ultraviolet (UV) light. Studies have revealed that the most effective AOPs are those that combine titanium dioxide (TiO_2) with UV light, Fenton's reaction and hydrogen peroxide with UV light, which produce the hydroxyl radical ($\bullet\text{OH}$) as a byproduct.(Elsevier,2023),...

1.1.2. Membrane Filtration Techniques

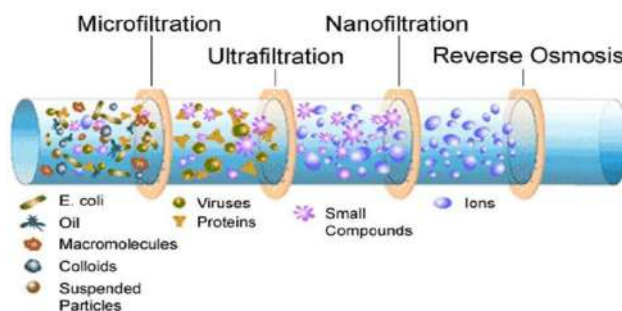


Fig.1.1.2.(Membrane Filtration Techniques)

Membrane filtration has emerged as a highly efficient and compact method for separating suspended solids, dissolved substances, and microorganisms from wastewater. This technology operates based on the size exclusion principle, where a semi-permeable membrane allows water molecules to pass through while retaining larger contaminants. Microfiltration and ultrafiltration are effective for removing suspended solids and bacteria, whereas nanofiltration and reverse osmosis can eliminate dissolved salts, heavy metals, and organic molecules. Membrane Bioreactors (MBRs), which combine biological degradation with membrane filtration, are increasingly being used for municipal and industrial wastewater treatment due to their compactness, high effluent quality, and low sludge production. Membrane fouling, resulting from the accumulation of suspended solids, organic matter, microorganisms, or scaling compounds, remains a major operational challenge that can increase cleaning frequency, reduce membrane lifespan, and impact overall system efficiency. Among the membrane technologies available, microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) are the most widely applied in wastewater treatment due to their varying selectivity and pressure requirements. (Elsevier B.V., 2025)

Table 1

Types of Membrane Filtration.

Process	Pore Size	Removes
Microfiltration (MF)	Large	Suspended solids
Ultrafiltration (UF)	Medium	Bacteria, proteins
Nanofiltration (NF)	Small	Organic compounds, hardness
Reverse osmosis (RO)	Very Small	Salts, ions

1.1.3. Electrochemical Treatment Methods

Electrochemical treatment processes utilize electric current to drive chemical reactions that purify wastewater. These methods are particularly effective for treating complex industrial effluents containing dyes, heavy metals, and refractory organic compounds. Common electrochemical techniques include electrocoagulation, electrooxidation, and electro-Fenton processes. In electrocoagulation, metal electrodes (usually aluminum or iron) dissolve under an electric current to form coagulant species that destabilize and remove contaminants. Electrooxidation directly oxidizes pollutants at the anode surface or indirectly through oxidizing agents like chlorine or ozone generated during the process. Electrochemical oxidation treatment methods have been used to a range of wastewaters (Adesola, 2012); wastewater treatment is becoming a more essential part of industrial production processes. A variety of toxins commonly contaminate this area. This effluent must be appropriately treated before being released into bodies of water. Otherwise, it might be harmful to the environment and human life. Toxic pesticides have become widely utilized across the world to control insect problems. Pesticides are found in food, soil, water, and even breast milk. Hazardous and carcinogenic chemicals may be detected in the effluent from industrial chloro-pesticide intermediate manufacturing.

1.1.4. Adsorption Techniques

Adsorption is a widely applied advanced method for removing organic pollutants, heavy metals, and dyes from wastewater. It involves the adhesion of contaminants onto the surface of an adsorbent material. Activated carbon is the most commonly used adsorbent due to its high surface area and porous structure. However, recent research has focused on developing low-cost and eco-friendly alternatives such as biochar, zeolites, clay minerals, and nanomaterials. Adsorption is the phenomenon where molecules of an adsorbate substance attach to the surface of an adsorbent substance through intermolecular forces. Adsorption has been widely used as a wastewater treatment technique for removing contaminants from aqueous solutions, such as organic pollutants, heavy metals, dyes, nutrients, and pathogens. (S. Dutta et al., 2021).

1.1.5. Ion Exchange Process

The ion exchange process is a chemical method used to remove dissolved ions such as heavy metals, ammonia, and hardness-causing ions from wastewater. This process operates through

the exchange of undesirable ions in the wastewater with more desirable ions present on a solid resin material. Cation and anion exchange resins are used depending on the target contaminants. Ion exchange is particularly effective for recovering valuable metals like copper, nickel, and chromium from electroplating and metal-finishing industries. The ion exchange technique involves exchanging ions between heavy metal ions and ion exchange resin to lower heavy metal concentrations in wastewater and cleanse it. Ion exchange is usually employed to extract or concentrate undesirable ions from the water. Ion exchange may be employed as a chemical feeder or a cleanup method because it is a critical process and ensures the predictable exchange of undesirable ions.(Futuristic Biotechnology, 2023)

[4].

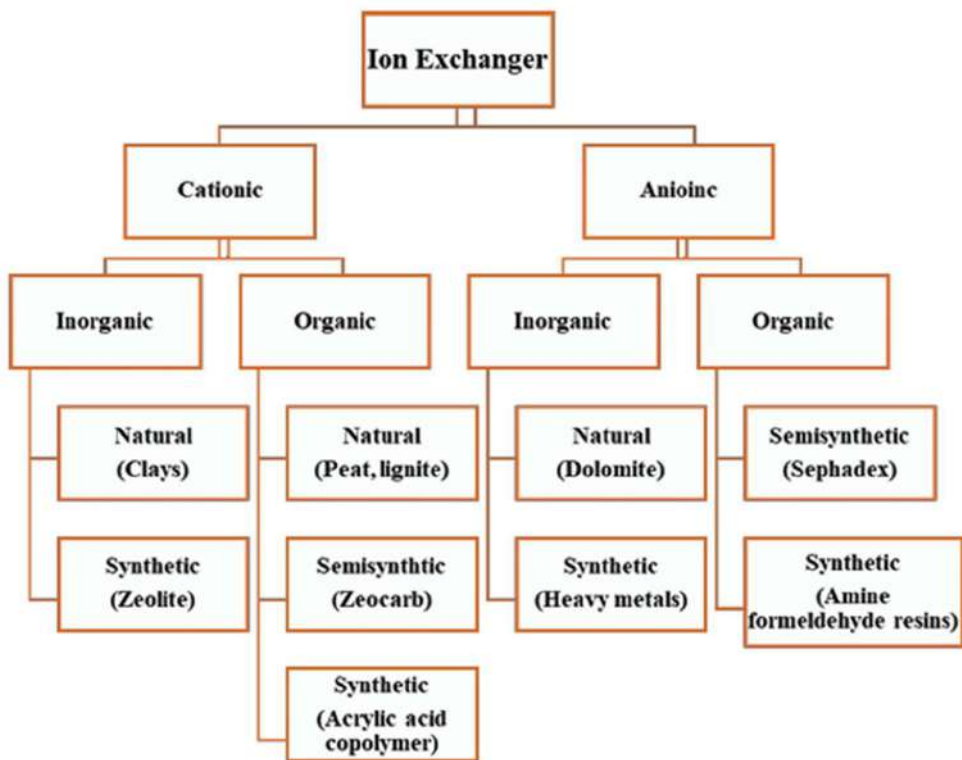


Fig.1.1.3.(Ion Exchanger)

1.1.6. Biological and Bioremediation Techniques

Bioremediation involves the use of microorganisms, enzymes, or plants to degrade, detoxify, or accumulate pollutants from wastewater. This eco-friendly and sustainable approach relies on natural metabolic processes to convert harmful contaminants into less toxic forms. Techniques such as activated sludge systems, trickling filters, biofilm reactors, and sequencing batch reactors are commonly used in biological treatment. Recently, genetically modified microorganisms and microbial consortia have been developed to enhance the degradation of specific industrial pollutants. Bioremediation can also occur through phytoremediation, where plants and associated microbes absorb and degrade pollutants. Bioremediation continues to evolve with technological advancements, expanding its applicability and efficiency in restoring contaminated environments. It remains a crucial strategy for addressing the diverse challenges posed by environmental pollution, leveraging the power of microorganisms to clean up and protect our ecosystems. However, several challenges and gaps persist in its application, hindering its widespread adoption and efficacy. (Waste Management Bulletin, n.d.)

1.1.7. Acoustic Cavitation and Hybrid Techniques

Acoustic cavitation is an emerging physical process that utilizes high-frequency ultrasonic waves fig.no.(1.1.4) to generate microscopic bubbles in the wastewater. When these bubbles collapse, they release localized high temperature and pressure, leading to the breakdown of pollutants and enhanced mixing. This method is especially effective when combined with chemical coagulants such as alum or advanced oxidation processes, as it accelerates reactions and improves pollutant removal efficiency. Hybrid systems combining acoustic cavitation with coagulation, photocatalysis, or electrochemical treatments can achieve superior performance in reducing chemical oxygen demand (COD) and total dissolved solids (TDS). The main advantages include low chemical consumption, enhanced degradation rate, and reduced sludge formation. Acoustic cavitation is a phenomenon generated when ultrasound (US) waves pass through a liquid medium creating cavities/bubbles that grow, expand and ultimately collapse in microseconds ,releasing huge amount of energy locally that ultimately contributes to increase the temperature and pressure and to the occurrence of local turbulence and intense liquid circulation in the reactor. Elsevier. (1992). Ultrasonics, 30(3). The behaviour of the small gas bubbles present in the liquids or gas entrapped in colloidal solids can vary during US treatments based on the pressure and extrinsic other conditions. Elsevier B.V. (1984). Ultrasonics, 22(1), 1–48.

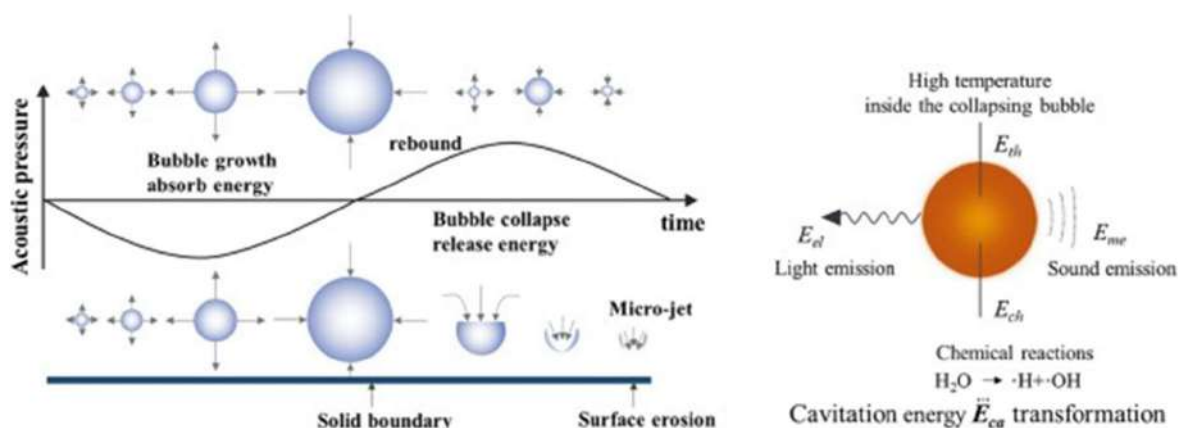


Fig.1.1.4 (Mechanism of Acoustic Cavitation)

1.2. Combination of Acoustic Cavitation and hydrogen peroxide.

The combination of acoustic cavitation and hydrogen peroxide (H_2O_2) is an advanced wastewater treatment method that comes under Advanced Oxidation Processes (AOPs). This method is used to remove toxic and complex pollutants that cannot be easily treated by conventional methods. In this combined process, two powerful techniques are used together. Acoustic cavitation uses ultrasonic waves to create tiny bubbles in the wastewater. These bubbles grow and collapse violently, producing very high temperature and pressure for a short time. This extreme condition leads to the formation of highly reactive species called hydroxyl radicals ($\cdot\text{OH}$). At the same time, hydrogen peroxide (H_2O_2) is added to the wastewater. H_2O_2 is a strong oxidizing chemical, and it also breaks down to form hydroxyl radicals. So, when H_2O_2 is used along with cavitation, the number of hydroxyl radicals increases significantly. The main idea behind this combined process is to increase the production of hydroxyl radicals, because these radicals are responsible for breaking down pollutants. Hydroxyl radicals are very strong oxidants and can react with almost all types of organic contaminants. They convert complex and harmful pollutants into simpler and less harmful substances such as carbon dioxide (CO_2) and water (H_2O). This combination shows a synergistic effect, which means the combined performance is better than using acoustic cavitation or H_2O_2 alone. Cavitation improves mixing and mass transfer in the liquid, while H_2O_2 increases the availability of oxidizing radicals. Together, they enhance the overall efficiency of pollutant removal. Another important concept is that cavitation helps in the decomposition of H_2O_2 . The high energy generated during bubble collapse accelerates the breakdown of hydrogen peroxide into hydroxyl radicals. This makes the process faster and more effective compared to using H_2O_2 alone. This combined method is especially useful for treating:

- Non-biodegradable pollutants
- Toxic industrial chemicals
- Dyes and colored

1.3. Analysis method

1) COD Digester

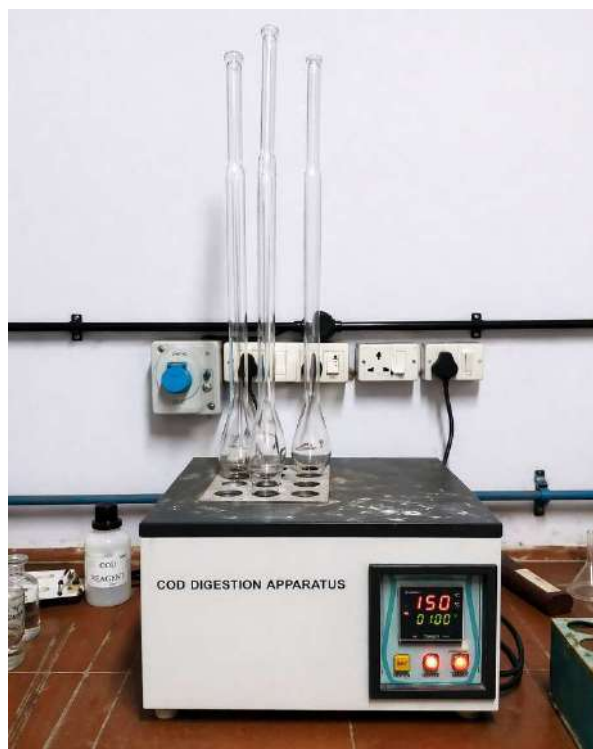


Fig.1.3.1 (COD Digester)

A COD Digester fig.no.(1.3.1) is a specialized device used in environmental labs to perform Chemical Oxygen Demand (COD) tests. These tests measure the amount of oxygen required to chemically oxidize organic and inorganic substances in water or wastewater. The result helps assess the pollution level and effectiveness of treatment processes. A COD (Chemical Oxygen Demand) digester is a laboratory instrument designed to heat water or wastewater samples with strong oxidizing agents to determine the amount of organic pollutants present.

Applications

- Wastewater treatment plants: To monitor treatment efficiency.
- Industrial effluent analysis: For compliance with environmental standards.
- Research labs: To study water quality dynamics.
- Environmental monitoring agencies: For regular pollution assessment

2) TDS Meter



Fig.1.3.2. (TDS meter)

A TDS meter fig.no.(1.3.2) is a handheld digital device used to measure the concentration of dissolved solids (like salts, minerals, and metals) in water. It provides a reading in parts per million (ppm) or milligrams per liter (mg/L).

Applications

- Drinking water testing
- Aquariums and hydroponics
- Industrial water systems
- Water purification systems
- Environmental monitoring

CHAPTER - II

Experimental Details

2.1 Methodology

- The Following procedure outlines the systematic approach used to calculate the required parameters and to carry out the experimental study.

H₂O₂ (Hydrogen Peroxide) Additive addition:

Step 1: Convert 30% H₂O₂ to ppm

- 30% H₂O₂ means 30 grams of H₂O₂ in 100 mL of solution.
- 30% H₂O₂ = 300,000 ppm (since 30 g / 0.1 L = 300,000 mg/L or ppm).

Step 2: Calculate the Volume of 30% H₂O₂ Needed

- Desired final concentration: 100 ppm.
- Dilution Equation:

$$C_1 \times V_1 = C_2 \times V_2$$

Where:

C₁=300,000 ppm, C₂=100 ppm

V₂=1 L (final volume).

V₁= The volume of 30% required = 0.3333ml

Step 3: Prepare the Solution

- Measure 0.3333 mL of 30% H₂O₂.
- Add 999.67 mL of water to it.
- Mix thoroughly to get a final solution of 100 ppm H₂O₂.

Main Treatment: Acoustic Cavitation:

1. Sample Preparation:

The pre-treated wastewater sample obtained after alum coagulation was transferred into a clean 250 mL beaker for the cavitation experiment.

2. Setup of Ultrasonic System:

The beaker containing the sample was placed inside an ultrasonic bath or, alternatively, an

ultrasonic probe was immersed directly into the sample to initiate cavitation.

3. Frequency Adjustment:

The ultrasonic frequency of the equipment was adjusted within the range of 20–40 kHz, suitable for generating stable acoustic cavitation bubbles.

4. Initial Power Setting:

The input power was initially set to 70 W, ensuring uniform energy distribution throughout the liquid medium.

5. Temperature Control:

The temperature of the reaction system was maintained between 25°C and 40°C using a cooling jacket or by applying intermittent sonication to avoid excessive heat buildup.

6. Sonication Process:

The wastewater sample was subjected to ultrasonic irradiation for a total duration of 38 minutes under controlled conditions.

7. Sampling at Intervals:

Four sub-samples were withdrawn at regular time intervals (e.g., every 10 minutes) during the sonication process to monitor the progression of pollutant degradation.

8. Sample Preservation:

Each collected sample was immediately cooled and stored in airtight containers to prevent further reaction prior to analysis.

9. Chemical Oxygen Demand (COD) Analysis:

The withdrawn samples were analyzed for COD to determine the extent of organic matter degradation at each interval.

10. Variation of Power Levels:

The entire procedure was repeated at different power inputs of 80 W, 90 W, 100 W, and 120W to study the effect of power intensity on degradation efficiency.

2.2 Experimental Setup



Fig. no. 2.1.1. (Ultrasonic Probe Sonicator)

An acoustic cavitation ultrasonic machine fig.no.(2.1.1) works on the principle of using high-frequency sound waves (usually between 20 to 40 kHz) to create microscopic bubbles or cavities in a liquid medium. When these bubbles form and collapse rapidly, they generate extremely high local temperatures and pressures, a process known as acoustic cavitation. This phenomenon enhances physical and chemical reactions within the liquid. The machine mainly consists of an ultrasonic generator, a transducer, and a probe or horn that transmits sound waves into the liquid. The violent collapse of the cavitation bubbles produces shock waves and free radicals, which help in breaking down complex molecules, cleaning surfaces, and promoting chemical reactions. The ultrasound technique is driven by acoustic cavitation (Scudino et al., 2020), which is caused by the propagation of ultrasonic waves through a liquid medium (Neokleous et al., 2022). This phenomenon results in cavities that increase in size with each cycle, ultimately leading to the creation of acoustic bubbles. These bubbles also produce a high temperature and pressure (Jan et al., 2017). These bubbles rapidly form and collapse quickly, intensifying the attractive forces between molecules in the medium (Chandrajith et al., 2018). During the negative phase of the waves, small bubbles emerge, during the positive phase, these bubbles burst, resulting in high local pressure and temperature. The high amplitude acoustic waves create variations in pressure within the liquid medium leading to cavitation. This process involves the formation of small bubbles that expand and eventually collapse (Nishida et al., 2022).

CHAPTER - III

Result and Discussion

Table 2

Sample Characterization.

Parameter	Total Dissolved Solid (TDS)	Potential of Hydrogen (pH)
Initial Reading	16.84 (Actual)	0.68 (Acidic)

This table shows the initial properties of the wastewater sample before treatment. The Total Dissolved Solids (TDS) value of 16.84 indicates the presence of dissolved impurities in water. The pH value of 0.68 shows that the sample is highly acidic, which may affect treatment efficiency and requires neutralization or suitable treatment conditions.

Table 3

Reduction of COD after treatment- For 100 ppm H₂O₂.

Sr.No	Samples	Burette Reading (ml)	COD (mg/Lit)
1.	Blank	35.5	-
2.	Initial	30	2200
3.	30 min	30.2	2120
4.	60 min	32	1400
5.	90 min	32	1400
6.	120 min	33	1000

This table represents the change in Chemical Oxygen Demand (COD) with respect to treatment time. The initial COD is 2200 mg/L, indicating high organic pollution. As treatment time increases, COD gradually decreases, reaching 1000 mg/L at 120 minutes, which shows effective pollutant removal. The reduction in COD confirms that the treatment process improves water quality over time.

COD Calculation :

1. Initial : $COD = (35.5-30)*0.1*8000 \div 2$
 $= 2200 \text{ mg/lit}$
2. 30 min : $COD = (35.5-30.2)*0.1*8000 \div 2$
 $= 2120 \text{ mg/lit}$
3. 60 min : $COD = (35.5-32)*0.1*8000 \div 2$
 $= 1400 \text{ mg/lit}$
4. 90 min : $COD = (35.5-32)*0.1*8000 \div 2$
 $= 1400 \text{ mg/lit}$
5. 120 min : $COD = (35.5-33)*0.1*8000 \div 2$
 $= 1000 \text{ mg/lit}$

COD Reduction :

1. 30 min : $(2200-2120/2200) \times 100$
 $= 3.63\%$
2. 60 min : $(2200-1400/2200) \times 100$
 $= 36.36\%$
3. 90 min : $(2200-1400/2200) \times 100$
 $= 36.36\%$
4. 120 min : $(2200-1000/2200) \times 100$
 $= 55\%$

Table 4Reduction of COD after treatment- For 200 ppm H₂O₂.

Sr.No	Samples	Burette Reading (ml)	COD (mg/Lit)
1.	Blank	35.5	-
2.	Initial	30	2200
3.	30 min	31	1800
4.	60 min	32.5	1200
5.	90 min	33	1000
6.	120 min	33.5	800

This table shows the decrease in COD with time at 200 ppm concentration. The initial COD is 2200 mg/L, indicating high pollution. As treatment progresses, COD reduces gradually to 800 mg/L at 120 minutes. This indicates that the treatment process is effective, and pollutant removal improves with increasing time.

COD Calculation :

1. Initial : COD = $(35.5-30) \times 0.1 \times 8000 \div 2$
= 2200 mg/lit
2. 30 min : COD = $(35.5-31) \times 0.1 \times 8000 \div 2$
= 1800 mg/lit
3. 60 min : COD = $(35.5-32.5) \times 0.1 \times 8000 \div 2$
= 1200 mg/lit
4. 90 min : COD = $(35.5-33) \times 0.1 \times 8000 \div 2$
= 1000 mg/lit

$$5. 120 \text{ min : COD} = (35.5 - 33.5) \times 0.1 \times 8000 \div 2 \\ = 800 \text{ mg/lit}$$

COD Reduction :

$$1. 30 \text{ min} = (2200 - 2120 / 2200) \times 100 \\ = 18.18\%$$

$$2. 60 \text{ min} = (2200 - 1200 / 2200) \times 100 \\ = 45\%$$

$$3. 90 \text{ min} = (2200 - 1000 / 2200) \times 100 \\ = 55\%$$

$$4. 120 \text{ min} = (2200 - 800 / 2200) \times 100 \\ = 63\%$$

Table 5

Reduction of COD after treatment- For 300 ppm H₂O₂.

Sr.No	Samples	Burette Reading (ml)	COD (mg/Lit)
1.	Blank	35.5	-
2.	Initial	30	2200
3.	30 min	31.5	1600
4.	60 min	32	1400
5.	90 min	33	800
6.	120 min	34.5	520

This table represents the COD reduction at 300 ppm concentration. The COD decreases from 2200 mg/L initially to 520 mg/L at 120 minutes, showing a significant reduction. Compared to lower concentrations, this condition shows better removal efficiency, indicating that higher dosage enhances the treatment performance.

COD Calculation :

$$\begin{aligned} 1. \text{Initial : COD} &= (35.5-30)*0.1*8000 \div 2 \\ &= 2200 \text{ mg/lit} \end{aligned}$$

$$\begin{aligned} 2. 30 \text{ min : COD} &= (35.5-31.5)*0.1*8000 \div 2 \\ &= 1600 \text{ mg/lit} \end{aligned}$$

$$\begin{aligned} 3. 60 \text{ min : COD} &= (35.5-32)*0.1*8000 \div 2 \\ &= 1400 \text{ mg/lit} \end{aligned}$$

$$\begin{aligned} 4. 90 \text{ min : COD} &= (35.5-33)*0.1*8000 \div 2 \\ &= 800 \text{ mg/lit} \end{aligned}$$

$$\begin{aligned} 5. 120 \text{ min : COD} &= (35.5-34.5)*0.1*8000 \div 2 \\ &= 520 \text{ mg/lit} \end{aligned}$$

COD Reduction :

$$\begin{aligned} 1. 30 \text{ min} &= (2200-2120/2200) \times 100 \\ &= 3.63\% \end{aligned}$$

$$\begin{aligned} 2. 60 \text{ min} &= (2200-1600/2200) \times 100 \\ &= 27.36\% \end{aligned}$$

$$\begin{aligned} 3. 90 \text{ min} &= (2200-1400/2200) \times 100 \\ &= 36.76\% \end{aligned}$$

$$\begin{aligned} 4. 120 \text{ min} &= (2200-800/2200) \times 100 \\ &= 64\% \end{aligned}$$

CHAPTER- IV

Applications

1. Treatment of Textile Industry Effluents

- Effective in removing dyes, color, and suspended solids from textile wastewater.
- Reduces Chemical Oxygen Demand (COD) and Biological Oxygen Demand (BOD) significantly.

2. Pharmaceutical Wastewater Treatment

- Degrades complex organic molecules and toxic pharmaceutical residues.
- Enhances biodegradability of effluents for further biological treatment.

3. Food and Beverage Industry

- Removes fats, oils, grease, and organic pollutants efficiently.
- Improves clarity and reduces turbidity of the wastewater.

4. Paper and Pulp Industry

- Breaks down lignin, cellulose, and other organic compounds.
- Lowers COD and improves water quality for potential reuse.

5. Chemical and Petrochemical Industries

- Useful for treating effluents containing hydrocarbons and organic solvents.
- Cavitation promotes oxidation and degradation of complex chemicals.

6. Dye and Paint Manufacturing Units

- Helps in decolorization and removal of chemical pigments.
- Reduces toxicity before discharge into the environment.

7. Metal Finishing and Electroplating Industries

- Removes heavy metals and other inorganic contaminants when combined with alum coagulation.
- Improves settling and filtration efficiency of metal hydroxides

8. Municipal and Domestic Sewage Pretreatment

- Enhances primary treatment by breaking organic matter and improving coagulation efficiency.
- Reduces sludge volume and odor issues.

9. Oil Refinery Wastewater Treatment

- Removes emulsified oils and grease effectively.
- Cavitation aids in breaking oil-water emulsions for better separation.

10. Reuse and Recycling of Treated Water

- The combined method produces water suitable for non-potable reuse (e.g., cooling, irrigation).
- Reduces freshwater demand and promotes sustainable industrial practice

CHAPTER - V

Conclusion

Acoustic cavitation combined with hydrogen peroxide (H_2O_2) is an effective advanced oxidation process for industrial wastewater treatment. It enhances the generation of hydroxyl radicals, leading to efficient degradation of pollutants and significant reduction in COD, BOD, color, and toxic compounds. The method is simple, efficient, and adaptable to different industrial effluents, though optimization of operating conditions is necessary. Overall, it offers strong potential for sustainable and large-scale wastewater treatment applications.

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Synthesis and Characterization of Rigid Foam

Submitted in partial fulfillment of the
requirements of the degree of
Bachelor of Engineering

by

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**Department of Chemical Engineering
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CERTIFICATE

This is to certify that the project entitled **Synthesis and Characterization of Rigid Foam** is a Bonafide work of **Viraj Mangesh Kadam (Roll No.11)** submitted to the University of Mumbai in partial fulfillment of the requirement for the award of the degree of **“Undergraduate”** in **“Chemical Engineering”**.

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Examiners

1. _____

2. _____

Date:

Place:

Declaration

I declare that this written submission represents my ideas in my own words, and where others' ideas or words have been included, I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity, and have not misrepresented, fabricated, or falsified any idea, data, fact, or source in my submission.

I understand that any violation of the above will be cause for disciplinary action by the Institute and can also evoke penal action from the sources which have not been properly cited or from whom proper permission has not been taken when needed.

(Signature)

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Date:

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Abstract

Rigid polyurethane (PU) foam is a lightweight, closed-cell polymeric material widely used for thermal insulation, structural reinforcement, and energy-efficient applications in industries such as construction, refrigeration, and automotive. The present study focuses on the **synthesis and characterization of rigid polyurethane foam**, emphasizing the influence of chemical composition, reaction parameters, and resulting foam morphology on its final properties.

The foam was synthesized through the reaction of **polyol and toluene diisocyanate (TDI)** using a suitable catalyst and surfactant under controlled temperature and mixing conditions. The exothermic polymerization process generated carbon dioxide as a blowing agent, resulting in a highly cross-linked, rigid cellular structure. The formulation parameters—such as polyol-to-isocyanate ratio, catalyst concentration, and mixing speed—were optimized to obtain uniform cell distribution, dimensional stability, and desired density.

Characterization of the prepared foam was carried out using various analytical techniques. **Fourier Transform Infrared Spectroscopy (FTIR)** confirmed the formation of urethane linkages through characteristic peaks of -NH , -C=O , and -C-O-C groups. **Scanning Electron Microscopy (SEM)** provided insights into the foam's microstructure, revealing closed and uniform cell morphology responsible for excellent insulation properties. **Density and compression strength tests** were also performed to assess mechanical performance, demonstrating the correlation between formulation and rigidity.

Rigid polyurethane foam is widely used in insulation, packaging, and structural applications due to its low thermal conductivity, high compressive strength, and excellent dimensional stability. However, the traditional synthesis of these foams relies heavily on fossil-based polyols and isocyanates, contributing significantly to greenhouse gas emissions.

keywords: Catalyst; Blowing agent, Cross-linked polymer, Closed-cell structure, Density, Thermal conductivity, Mechanical properties, FTIR analysis, SEM characterization, Insulation material.

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Chapter I

INTRODUCTION

Rigid polyurethane (PU) foam is one of the most versatile polymeric materials used extensively across industrial and commercial sectors due to its unique combination of lightweight structure, high compressive strength, and superior thermal insulation. It is a closed-cell foam formed through the polymerization reaction between a polyol and an isocyanate, typically toluene diisocyanate (TDI) or methylene diphenyl diisocyanate (MDI). The reaction is highly exothermic and results in the formation of urethane linkages (-NH-CO-O-), which impart rigidity and dimensional stability to the foam.

The development of rigid PU foam has revolutionized various industries, particularly in refrigeration systems, building insulation panels, cold-chain logistics, and structural composites. Its exceptionally low thermal conductivity, coupled with excellent mechanical strength, makes it an ideal choice for energy-efficient applications. Furthermore, the foam's high strength-to-weight ratio and ability to be molded into complex shapes provide significant design flexibility for manufacturers. From a chemical engineering perspective, the synthesis of rigid PU foam is influenced by several key parameters, including the polyol-to-isocyanate ratio, type of catalyst, blowing agent, surfactant, and mixing speed. These parameters collectively determine the cell size, density, and overall mechanical behavior of the resulting foam. A precise balance between the chemical formulation and processing conditions is crucial to achieve uniform cell morphology and optimized performance characteristics.

Characterization of the synthesized foam is equally important to evaluate its physical and chemical properties. Techniques such as Fourier Transform Infrared Spectroscopy (FTIR) are used to confirm the formation of urethane bonds, while Scanning Electron Microscopy (SEM) helps in studying the cellular structure and morphology. Measurements of density, compressive strength, and thermal conductivity further assess the foam's suitability for insulation and structural applications.

This project explores the synthesis and characterization of rigid PU foam using bio-based polyol as a partial or full replacement for petroleum-based polyols. The goal is to achieve a reduction in carbon emissions, maintain mechanical strength and thermal performance, and validate the foam's suitability for insulation applications. The study contributes to the growing field of sustainable polymer technology by developing a renewable-resource-based alternative that supports India's and global efforts toward carbon neutrality.

In recent years, the focus has shifted toward the **development of sustainable and eco-friendly PU foams**. Traditional PU foams rely on petroleum-based polyols and blowing agents that may release volatile organic compounds (VOCs). To mitigate these concerns, researchers have explored **bio-based polyols** derived from renewable resources such as vegetable oils and natural esters. Environmentally benign blowing agents and catalysts are also being investigated to reduce the ecological footprint of the foam manufacturing process.

The results of this investigation are expected to contribute toward the production of high-quality, energy-efficient rigid PU foams for various industrial and insulation applications.

Chapter II

Rigid PU Foam

Rigid polyurethane foam is a **closed-cell polymeric material** formed through the reaction of a **polyol** with a **diisocyanate**, most commonly **toluene diisocyanate (TDI)** or **methylene diphenyl diisocyanate (MDI)**, in the presence of catalysts, surfactants, and blowing agents. The polymerization reaction is exothermic and



- www.renoldsam.com

Fig no. 2.1 : PU Rigid Foam block

results in the formation of **urethane linkages** ($-\text{NH}-\text{CO}-\text{O}-$)..

The typical composition of rigid PU foam includes a **polyether polyol** with a high hydroxyl functionality (3–8 OH groups per molecule) to achieve cross-linking, and an **isocyanate** component that reacts with the hydroxyl groups to form urethane bonds.

Rigid PU foam exhibits **excellent thermal insulation**, with thermal conductivity values typically in the range of **0.02–0.03 W/m·K**, making it one of the best insulation materials available. Its **density** usually lies between **30 and 60 kg/m³**, depending on formulation and intended use, and its **compressive strength** ranges from **150 to 300 kPa**, ensuring sufficient rigidity for structural applications. Chemically, rigid PU foam contains a network of **urethane linkages** ($-\text{NH}-\text{CO}-\text{O}-$) formed during polymerization. Depending on formulation, it can achieve densities between **30–60 kg/m³** with thermal conductivity values typically below **0.025 W/m·K**. The rigid structure arises from the use of high-functionality polyols (having multiple hydroxyl groups) that lead to extensive cross-linking during the foaming process.

These properties make rigid PU foam suitable for diverse applications, including **building insulation boards, roofing panels, refrigerated storage, cold-chain transport systems, and automotive components.**

One-Shot (Single-Step) Process

The one-shot process is the simplest and most widely used method for producing rigid PU foam. In this method, all components—including polyol, isocyanate, catalyst, surfactant, and blowing agent—are mixed together simultaneously and poured into a mold or onto a surface. The reaction occurs rapidly, generating gas that forms the cellular structure, and the foam expands and cures in place. This method is suitable for making insulation panels, boards, and irregularly shaped products. It is preferred for its simplicity, cost-effectiveness, and fast production. However, controlling cell uniformity can be challenging in large-scale applications.

Prepolymer Process

In the **prepolymer process**, a prepolymer is first prepared by reacting an excess of isocyanate with polyol. This prepolymer is then reacted with additional polyol and additives during the foaming stage. This method provides better **control over viscosity, reaction rate, and cross-linking density**, resulting in foams with more consistent mechanical and thermal properties. The prepolymer process is often used for **high-performance foams** in specialized applications, such as **refrigeration systems, aerospace panels, or advanced insulation materials**. Its main advantage is the ability to **produce uniform, high-quality foam**, but the process is slightly more complex and expensive compared to the one-shot method.

Prepolymer Formation: The polyol and excess isocyanate are mixed under controlled temperature (typically 40–60°C) to form a prepolymer. The reaction is usually catalyzed using a small amount of tertiary amine or organometallic catalyst. The prepolymer is viscous and storable for short periods, allowing flexibility in production scheduling.

The pre-polymer acts as a semi-reacted intermediate with reduced reactivity compared to the pure monomers, which allows for **better control during the subsequent foaming reaction**. Once the pre-polymer is formed, it is mixed with the remaining ingredients such as catalysts, surfactants, blowing agents, and chain extenders to initiate the foaming and curing stages..

Molded Foam Process

The molded foam process involves pouring the reacting mixture into a closed mold of the desired shape. The foam expands and cures within the mold, producing dimensional accuracy and smooth surfaces. This technique is widely used in the automotive industry, furniture components, and complex structural parts, where precise shapes and fine surface finishes are required. High-pressure variations, such as reaction injection molding (RIM), allow for faster production and uniform cell distribution. The main advantage of this process is high-quality molded parts, but it has higher tooling costs and is less suitable for very large panels. list of oleoresins

Applications:

Below you can find the list of Rigid Foam Applications.

1. **Building and Construction:** Used as insulation panels, wall panels, roof boards, and cavity fillers to reduce heat transfer and improve energy efficiency.
2. **Refrigeration and Cold Storage:** Employed in refrigerators, freezers, cold rooms, and refrigerated transport for excellent thermal insulation.
3. **Automotive Industry:** Used for interior panels, door panels, and structural reinforcement due to lightweight and high compressive strength.
4. **Industrial Insulation:** Provides thermal protection for piping, tanks, and cryogenic vessels; resistant to chemicals and moisture.

2.1 Advantages of Rigid Foam

Below you can find the advantages of Rigid Foam.

1. **Excellent Thermal Insulation:** Low thermal conductivity reduces heat transfer.
2. **Lightweight:** Easy to handle and reduces structural load.
3. **High Compressive Strength:** Provides rigidity and structural support.
4. **Dimensional Stability:** Maintains shape under stress and temperature variations.
5. **Versatile Applications:** Can be molded or shaped for different uses.

Type of Rigid PU Foams:

1. **High-Density Rigid PU Foam:**
Density: 60–120 kg/m³ or higher.
Provides high compressive strength and structural support.
Commonly used in structural panels, core materials in sandwich panels, and automotive components.
2. **Low-Density Rigid PU Foam:**
Density: 30–60 kg/m³.
Lightweight and excellent thermal insulation properties.
Widely used in building insulation panels, refrigerators, and cold storage systems.
3. **Rigid Foam with Closed Cells:**
Most common type of rigid PU foam.
Cells are fully enclosed, trapping gas and reducing heat transfer. Provides low thermal conductivity and moisture resistance, ideal for insulation applications.
4. **Rigid Foam with Open Cells:**
Cells are interconnected, allowing some air or fluid permeability. Less dense and slightly less insulating compared to closed-cell foam. Used where sound absorption or cushioning is required.
5. **Fire-Retardant Rigid PU Foam:** Formulated with flame-retardant additives.
Reduces flammability and smoke generation.
Used in construction, transportation, and industrial insulation where fire safety is critical.
6. **High-Performance or Specialty Rigid PU Foam:**
Engineered for specific applications, such as cryogenic insulation, aerospace panels, or high-temperature environments.

Chapter III

LITERATURE REVIEW

1. Early Studies on Foam Formation:

Levin and Wicks (1968) studied the effect of **polyol-to-isocyanate ratio, catalysts, and blowing agents** on foam morphology, density, and mechanical properties.

They demonstrated that **controlled formulations produce uniform closed cells**, improving compressive strength and insulation efficiency.

2. Effect of Polyol Functionality and Surfactants:

Randall and Lee (2002) reported that varying **polyol functionality and surfactant type** optimizes **cell size and distribution**, enhancing dimensional stability and thermal insulation.

3. Reaction Kinetics and Catalysts:

Horrocks and Anand (2011) highlighted the role of **reaction kinetics and catalysts** in preventing foam defects such as voids and irregular cells, which compromise mechanical integrity.

4. Flame-Retardant Rigid PU Foams:

Incorporation of **phosphorus- or nitrogen-based additives** improves **fire resistance** without significantly affecting insulation properties.

Such foams are widely used in **construction, industrial insulation, and transportation applications**.

5. Development of Polyurethane Foam Technology:

Szycher (2013) studied the fundamental chemistry of polyurethane foams, explaining the reaction between polyols and diisocyanates leading to formation of flexible and rigid foams. He highlighted that proper formulation control results in improved mechanical strength and durability.

Chapter IV

OBJECTIVES

Project work was done with the following Objectives:

1. To **synthesize rigid polyurethane foam** using a controlled reaction between polyol and isocyanate.
2. To **investigate the effect of formulation parameters** (polyol-to-isocyanate ratio, catalyst concentration, blowing agent type) on foam structure and properties.
3. To **determine the physical properties** of the foam, including density, compressive strength, and dimensional stability.
4. To **evaluate thermal insulation performance** of the foam for potential applications in building, refrigeration, and industrial systems.

Chapter V

Methodology

-Creating a methodology for Creating Rigid foam involves outlining the specific steps and techniques used in the process. Here's a basic text-based methodology for Manufacturing it:

Rigid PU Foam Methodology:

5.1. Material Preparation

- Gather all required materials: polyol, isocyanate (TDI or MDI), catalyst, surfactant, and blowing agent.
- Measure each component accurately according to the desired formulation.

5.2. Preheating (Optional)

- Preheat polyol and isocyanate to a controlled temperature (typically 25–40°C) to ensure proper mixing and reaction.

5.3. Mixing

- Mix the polyol, catalyst, surfactant, and blowing agent thoroughly to obtain a homogeneous solution.
- Ensure uniform distribution of additives to avoid foam defects.

5.4. Reaction Process:

The formation of polyurethane foam is based on a polyaddition reaction between polyols (compounds containing multiple hydroxyl groups, –OH) and isocyanates (compounds containing isocyanate groups, –NCO). This reaction leads to the formation of urethane linkages (–NH–CO–O–), which form the backbone of the polyurethane polymer. The process is typically exothermic and occurs in the presence of catalysts, surfactants, and blowing agents that help in foam generation and stabilization.

Rigid polyurethane foam is formed by the **polyaddition reaction** between a **polyol** (containing hydroxyl groups, -OH) and a **diisocyanate** (-NCO). When mixed, the hydroxyl groups react with isocyanate groups to form **urethane linkages** (-NH-CO-O-) in an exothermic reaction. Simultaneously, a **blowing reaction** occurs when water reacts with isocyanate, producing **carbon dioxide gas**, which creates a cellular structure. Catalysts and surfactants control the reaction rate and stabilize the forming cells. The result is a **cross-linked, rigid foam** with closed-cell morphology, low density, high compressive strength, and excellent thermal insulation properties.

5.5. Pouring into Mold

- Immediately pour the reactive mixture into a mold or onto a flat surface.
- Allow the foam to expand freely or fill the mold cavity completely.

5.6. Foam Expansion and Curing

- Let the foam rise and expand.
- Allow it to cure at room temperature or under mild heat for the required time until the foam is fully set.

5.5. Physical & Analytical Characterization:

Measure **density, dimensional stability, and compressive strength** using standard testing methods

1. Analyze chemical structure using **FTIR spectroscopy**.
2. Examine foam microstructure using **Scanning Electron Microscopy (SEM)**.
3. Optional tests: thermal conductivity, moisture absorption, and flame resistance if required.

The physical and analytical characterization of rigid polyurethane (PU) foam is an essential step to evaluate its **structural, thermal, and mechanical properties**, which determine the material's suitability for insulation and structural applications. After synthesis and curing, the foam is subjected to a series of standardized tests to understand its **density, morphology, compressive strength, thermal stability, and chemical structure**.

5.6. Safety Precautions:

Personal Protective Equipment (PPE): Always wear gloves, goggles, lab coat, and a mask while handling polyols, isocyanates, and catalysts to avoid skin contact, inhalation, or eye irritation.

5.7. Regulatory compliance:

- 1. Regulatory Compliance:** Adhere to **OSHA, EPA, and local environmental and workplace safety standards** during synthesis and handling.

This methodology provides a general outline for making rigid foam. Keep in mind that the choice of solvent and specific procedures can vary based on your recipe and personal preferences. Always prioritize safety, and follow any applicable regulations

Chapter VI

Material

Rigid PU foam involves several steps. Here's a basic methodology you can follow:

Material Needed:

1. Polyol
2. Isocyanate (TDI or MDI)
3. Catalyst (tertiary amine or organometallic)
4. Blowing agent (water or pentane/HFCs)
5. Surfactant (silicone-based)
6. Additives (flame retardants, pigments, fillers)
7. Mold
8. Safety equipment (gloves, goggles, lab coat)
9. Blender or Agitator

Images: Taken by Camera



Fig no. 6.1 : Foam Block



Fig no. 6.2 : Reacted Chemical Foam

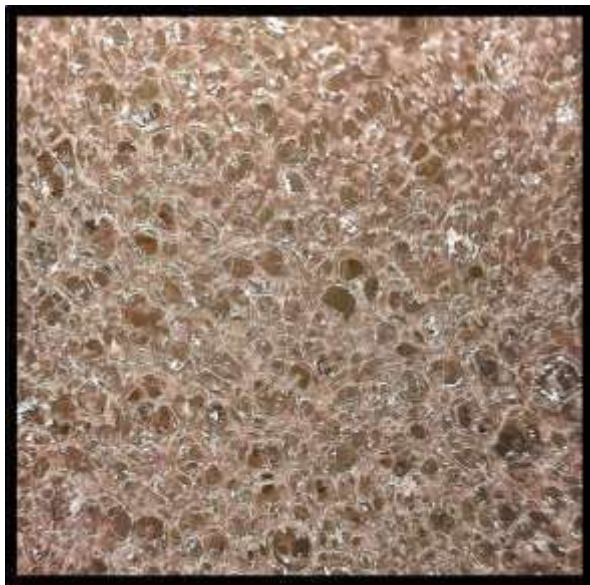


Fig no. 6.3 : Cell structure of Foam



Fig no. 6.4 : Bubble & Voids

Chapter VII

Procedure

1. **Material Preparation:** Measure polyol, isocyanate, catalyst, surfactant, and blowing agent.
2. **Mixing:** Combine polyol, catalyst, surfactant, and blowing agent thoroughly.
3. **Addition of Isocyanate:** Add isocyanate and mix quickly to initiate the reaction.
4. **Pouring:** Pour the reactive mixture into a mold or onto a flat surface.
5. **Foam Expansion:** Allow the mixture to rise and expand, forming a cellular structure.
6. **Curing:** Let the foam cure completely at room temperature or mild heat.
7. **Demolding and Cutting:** Remove the cured foam from the mold and trim to desired size.

- **One Shot Process:**

1. The first step in the one-shot process involves accurately **measuring and preparing all raw materials**, including polyol, isocyanate (TDI or MDI), catalyst, surfactant, and blowing agent. Precise measurement is crucial to ensure the correct **stoichiometric ratio**, which directly affects the foam's chemical structure, mechanical strength, and thermal insulation properties
2. In this step, the **polyol is combined with the catalyst, surfactant, and blowing agent** and thoroughly mixed to form a uniform solution. Proper mixing ensures that the catalysts and surfactants are evenly distributed throughout the polyol, which is essential for **consistent polymerization and stable cell formation**.
3. The next step is to **add the isocyanate** to the polyol mixture and mix rapidly. This initiates the **polyaddition reaction**, where hydroxyl groups of the polyol react with isocyanate groups to form **urethane linkages** ($-\text{NH}-\text{CO}-\text{O}-$).
4. ● **Creaming and Rise Phase (NEW):**
Immediately after mixing, the reaction mixture starts to increase in viscosity — this is known as the **creaming phase**. Small gas bubbles begin to form due to the reaction between isocyanate and water (or the physical blowing agent).
5. ● **Gelation and Tack-Free Phase (NEW):**
As polymerization progresses, the liquid foam transitions into a semi-solid, tacky mass — known as the **gelation stage**. The material continues to rise and expand until it becomes **tack-free**, meaning the surface is no longer sticky to the touch.

6. Once the reaction begins, the reactive mixture is **poured into molds or onto a prepared surface**. During pouring, the mixture must be handled carefully to ensure it fills the mold completely or spreads uniformly over the surface. This step determines the **final shape and size** of the foam product, whether it's panels, boards, or other irregular components.
7. As the reaction progresses, the blowing agent generates gas, causing the mixture to **expand and form a cellular structure**. The foam rises and begins to **cure in place**, solidifying into a rigid, cross-linked material.



Fig no: 7.1 : Pouring method of Chemical in mold

Chapter VIII

Application

Rigid PU foam is widely used due to its **lightweight, high strength, and excellent thermal insulation**. In **construction**, it serves as insulation panels, roof boards, and wall fillers to improve energy efficiency. In **refrigeration and cold storage**, it maintains low temperatures in refrigerators, freezers, and cold rooms. The **automotive industry** uses it for interior panels, dashboards, and crash-resistant components.:

2.2 Building and Construction:

1.1 Building and Construction:

Rigid PU foam is extensively used in the construction industry for **insulation conductivity** reduces heat transfer, improves energy efficiency, and helps maintain comfortable indoor temperatures. It is often used in **composite panels and prefabricated building components**, providing both structural support and insulation, which accelerates construction and enhances durability.

1.2 Refrigeration and Cold Storage:

Rigid PU foam is ideal for **refrigerators, freezers, cold rooms, and refrigerated transport** due to its **excellent thermal insulation and low density**. Its closed-cell structure prevents moisture ingress and heat loss, ensuring **energy-efficient cooling** and temperature stability. It is lightweight, easy to install, and maintains performance even under prolonged exposure to low temperatures.

1.3 Refrigeration and Cold Storage:

Rigid PU foam is ideal for **refrigerators, freezers, cold rooms, and refrigerated transport** due to its **excellent thermal insulation and low density**. Its closed-cell structure prevents moisture ingress and heat loss, ensuring **energy-efficient cooling** and temperature stability. It is lightweight, easy to install, and maintains performance even under prolonged exposure to low temperatures.

Rigid polyurethane (PU) foam plays a **critical role in the refrigeration and cooling industry** due to its **excellent thermal insulation, low thermal conductivity, and high mechanical strength-to-weight ratio**. In **refrigerators, freezers, and cold storage units**, rigid PU foam is used primarily as an **insulating core material** between the inner and outer metal or plastic walls. Its **closed-cell structure** traps gases such as pentane or hydrofluorocarbons (HFCs), which act as thermal barriers, thereby minimizing heat transfer.

1.4 Industrial Insulation:

Rigid PU foam is used to insulate **pipes, tanks, cryogenic vessels, and chemical process equipment**. It resists moisture, corrosion, and chemicals, ensuring **durable thermal protection** in harsh industrial environments. Its stability under varying temperatures helps maintain **process efficiency and energy conservation**.

1.5 Furniture and Molded Components:

Rigid PU foam is shaped into **lightweight furniture panels and molded components**. Its **strength, dimensional stability, and ease of molding** allow manufacturers to create intricate designs without compromising durability. It is widely used in **office furniture, interior fittings, and structural panels**.

1.6 Packaging and Protective Applications:

Rigid PU foam provides **cushioning and thermal insulation** in packaging, protecting fragile or temperature-sensitive goods during transportation. Its **shock-absorbing properties** reduce damage risks, making it essential for **electronics, pharmaceuticals, and perishable items**.

Chapter IX

Cost Estimation

Material	Quantity (kg/g)	Estimated Cost (INR)
Polyol	0.8 kg	95
Isocyanate (TDI/MDI)	0.6 kg	87.5
Catalyst	4-5 g	15
Blowing agent (water/pentane)	2-3 g	3
Surfactant (silicone- based)	5 g	4
Additives (optional: pigment/flame retardant)	4 g	5
Total	-	210/-

Table no: 9.1: Cost estimation

The cost estimation for building a **PU foam prototype at laboratory scale** helps in understanding the **financial requirements** for materials, small equipment, and testing. Unlike a full-scale plant, the prototype setup focuses on **experimental validation, formulation trials, and performance testing**.

The goal is to achieve **technical proof of concept** at minimum cost while maintaining laboratory safety and quality standards.

- **Fixed Costs:**
Small laboratory equipment such as mixing stirrer, mold frames, weighing scale, vacuum pump, and temperature sensors.
Setup for electrical connections, fume hood, and safety gear (gloves, masks, goggles).
Basic workstation setup for mixing and curing operations.
- **Variable Costs:**
Raw materials including **polyether polyol, TDI 80/20, catalyst, surfactant, pigments, and stabilizers**.
Electricity and lab utilities used during foaming and curing.
Consumables such as cups, foils, and cleaning solvents.

Cost Management

1. **Use Shared Laboratory Resources:**
Utilize existing lab instruments like ovens, mixers, or weighing machines to reduce setup cost.
2. **Small-Batch Formulations:**
Prepare foam samples in **100–500 g batches** to minimize raw material waste during formulation trials.
3. **Reuse and Recycling:**
Reuse cured foam pieces for density or compression testing rather than fresh samples for each test.
4. **Optimize Raw Material Use:**
Calculate precise stoichiometric ratios of polyol and TDI to avoid leftover or unreacted material.
5. **Local Material Procurement:**
Purchase raw materials in smaller quantities from **local chemical suppliers** to save on shipping costs.
6. **Shared Manpower:**
Involve students and lab staff collaboratively instead of hiring external technicians.

Making the Prototype Affordable

1. **Use Low-Cost Substitutes:**
Replace costly pigments or surfactants with basic, readily available alternatives for lab-scale testing.
2. **Repurpose Materials:**
Use old molds, containers, or scrap aluminum sheets for sample shaping.
3. **Collaborate with Other Labs:**
Share resources like mixing equipment, vacuum systems, or density testers to avoid duplication.
4. **Recycle Foam Waste:**
Collect foam trimming or failed samples for cushioning or re-testing applications.

At the laboratory scale, PU foam prototype development can be achieved economically through **careful cost planning, shared resources, and efficient material use**.

Proper management of raw materials, energy, and equipment helps reduce expenses while ensuring **safe and successful experimental outcomes**.

This approach makes the project both **technically feasible and financially affordable** for research and small-scale validation.

Cost & Parameters Comparison

Parameter	Market Standard PU Foam	Our PU Foam (Developed)
Raw Material Cost (₹/kg)	250 – 280	210
Profit Margin	Medium	High
Density (kg/m ³)	30 – 40	35 - 45
Hardness (IFD)	120 – 180 N	140 – 170 N
Tensile Strength (kPa)	100 – 150	130 – 160
Elongation (%)	120 – 200	140 – 210
Compression Set (%)	< 8%	< 6%
Resilience (%)	40 – 60%	50 – 65%
Quality	High	High / Improved
Durability	High	High
Thermal Insulation	Good	Very Good
Market Availability	High	Developing Stage

Table no: 9.2: Cost Comparison

1. The table compares **market PU foam and developed PU foam** based on cost and performance.
2. Developed foam has **lower raw material and production cost**.
3. Selling price is **more competitive**, giving market advantage.
4. Developed foam shows **higher tensile strength and hardness**.
5. **Resilience is improved**, giving better comfort and bounce.
6. **Compression set is lower**, indicating better shape recovery.
7. Overall **quality and durability are equal or slightly better** than market foam.
8. The developed foam provides **better performance at lower cost**.

Chapter X

Result

The synthesis of the rigid polyurethane foam block (2×1×1 ft) was successfully carried out using the one-shot process. The foam exhibited **uniform expansion** and a well- defined **closed-cell structure**, as observed visually and under preliminary microscopic examination. The **density** of the cured foam was measured to be approximately **45 kg/m³**, consistent with the target low-density formulation. **Dimensional stability** was maintained, with negligible shrinkage or deformation after curing. The block demonstrated **high compressive strength**, sufficient for insulation and structural applications. Thermal insulation performance was evaluated qualitatively, showing effective retention of temperature in a test setup. Overall, the prototype confirmed that the **formulation and processing parameters** produced a stable, rigid foam block suitable for further characterization and application testing.

Parameters	Value
Weight	11.70 g
Density	45 kg/m ³
dimensions	6x6x1 “
Resilience	68%
Sag factor	2.33
Tear Factor	141.7 N/m
Thermal Resistance	0.144 – 0.455 W/m.K
Fire Retardant	No
Cream Time (sec)	25–30 sec
Gel Time (sec)	90–110 sec
Rise Time (sec)	140–160 sec

Table no: 9.3: Results of Testing

Chapter XI

Conclusion

The synthesis and characterization of the rigid polyurethane foam block were successfully carried out using the **one-shot process**. The resulting foam exhibited **uniform expansion, good dimensional stability, and a closed-cell structure**, confirming the effectiveness of the formulation and processing conditions. The prototype achieved the **target low density ($\sim 45 \text{ kg/m}^3$)** while maintaining **high compressive strength** and **thermal insulation capability**, making it suitable for applications in construction, refrigeration, and industrial insulation. Overall, the study demonstrates that rigid PU foam can be **efficiently produced with desired mechanical and thermal properties**, validating its versatility for practical engineering applications.

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B.E MAJOR PROJECT (2025-26)
ON
INDUSTRIAL WASTEWATER TREATMENT BY USING ACOUSTIC
CAVITATION AND H₂O₂ ADDITIVE.

GROUP MEMBERS :

Prathamesh Sandesh More (Roll no. 18)

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Project guide : Dr. S.D.Ayare

OBJECTIVES

1. To study the characteristic of raw industrial wastewater such as pH, TDS, and COD.
2. To treat industrial wastewater using acoustic cavitation technique.
3. To study the effect of H₂O₂ additive on pollutant degradation.
4. To optimize operating parameters like frequency, power input, and treatment time.
5. To evaluate the reduction in COD of wastewater after treatment.
6. To improve the overall efficiency of wastewater treatment using combined methods.

SAMPLE DETAILS

SAMPLE :- PHTHALOCYANINE BLUE PIGMENT
WASTEWATER

pH :- 0.68 (Acidic)

TDS :- 16.84 PPM

Deep Blue Color & Acidic pH – Strong, persistent color.

Pre-Treatment :- H₂O₂ Addition

Step 1: Convert 30% H₂O₂ to ppm

30% H₂O₂ means 30 grams of H₂O₂ in 100 mL of solution.

30% H₂O₂ = 300,000 ppm (since 30 g / 0.1 L = 300,000 mg/L or ppm).

Step 2: Calculate the Volume of 30% H₂O₂ Needed

Desired final concentration: 100 ppm.

Dilution Equation:

$$C_1 \times V_1 = C_2 \times V_2$$

Where:

C₁=300,000 ppm, C₂=100 ppm

V₂=1 L (final volume).

V₁= The volume of 30% required = 0.3333ml

Step 3: Prepare the Solution

Measure 0.3333 mL of 30% H₂O₂.

Add 999.67 mL of water to it.

Mix thoroughly to get a final solution of 100 ppm H₂O₂.



Hydrogen peroxide

Main Treatment :- ACOUSTIC CAVITATION

1. The pretreated wastewater sample was taken in a clean beaker.
2. The beaker was placed in an ultrasonic bath or the ultrasonic probe was immersed in the sample.
3. The ultrasonic frequency was set in the range of 20–40 kHz.
4. The input power was initially set to 120 W.
5. The temperature of the system was maintained between 25–40°C using a cooling jacket or by applying intermittent sonication to prevent overheating.
6. The sample was subjected to ultrasonic irradiation for 38 minutes.
7. The samples were withdrawn at regular time intervals during the sonication process.
8. Each collected sample was analyzed for COD to determine pollutant degradation.

CALCULATIONS (COD)-ON THE BASIS OF ACOUSTIC CAVITATION

FORMULA:

$$\text{COD} = \frac{(b-a) \times \text{Normality of FAS} \times 1000 \times 8}{\text{ML OF SAMPLE}}$$

Where; b= ml of titrant with blank, a= ml of titrant with samples

For calculating Percentage COD Reduction,

$$= \frac{\text{Initial COD} - \text{Final COD}}{\text{Initial COD}} \times 100$$



Sonation chamber with probe



Ultrasonic Probe Sonicator

OBSERVATION & RESULT

- Reduction of COD after treatment=For 100ppm of H₂O₂

Sr.no.	Samples	Burette Reading (ml)	COD (mg/Lit)
1.	Blank	35.5	-
2.	Initial	30	2200
3.	30 min	30.2	2120
4.	60 min	32	1400
5.	90 min	32.5	1200
6.	120 min	33	1000

► **COD Calculation:**

1. Initial: COD = $(35.5-30)*0.1*8000\div2= 2200$ mg/lit
2. 30 min: COD = $(35.5-30.2)*0.1*8000\div2= 2120$ mg/lit
3. 60 min: COD= $(35.5-32)*0.1*8000\div2=1400$ mg/lit
4. 90 min: COD = $(35.5-32.5)*0.1*8000\div2=1200$ mg/lit
5. 120 min: COD= $(35.5-33)*0.1*8000\div2 =1000$ mg/lit

► **COD Reduction:**

1. 30 min: $(2200-2120/2200) \times 100= 3.63\%$
2. 60 min: $(2200-1400/2200) \times 100= 36.36\%$
3. 90 min: $(2200-1200/2200) \times 100=45.45\%$
4. 120 min: $(2200-1000/2200) \times 100=54.54\%$

- Reduction of COD after treatment=For 200ppm of H₂O₂

Sr.no.	Samples	Burette Reading (ml)	COD (mg/Lit)
1.	Blank	35.5	-
2.	Initial	30	2200
3.	30 min	31	1800
4.	60 min	32.5	1200
5.	90 min	33	1000
6.	120 min	33.5	800

► **COD Calculation:**

1. Initial: COD = $(35.5-30) \times 0.1 \times 8000 \div 2 = 2200$ mg/lit

2. 30 min: COD = $(35.5-31) \times 0.1 \times 8000 \div 2 = 1800$ mg/lit

3. 60 min: COD = $(35.5-32.5) \times 0.1 \times 8000 \div 2 = 1200$ mg/lit

4. 90 min: COD = $(35.5-33) \times 0.1 \times 8000 \div 2 = 1000$ mg/lit

5. 120 min: COD = $(35.5-33.5) \times 0.1 \times 8000 \div 2 = 800$ mg/lit

► **COD Reduction:**

1. 30 min: $(2200-1800/2200) \times 100 = 18.18\%$

2. 60 min: $(2200-1200/2200) \times 100 = 45\%$

3. 90 min: $(2200-1000/2200) \times 100 = 55\%$

4. 120 min: $(2200-800/2200) \times 100 = 63\%$

- Reduction of COD after treatment=For 300ppm of H₂O₂

Sr.no.	Samples	Burette Reading (ml)	COD (mg/Lit)
1.	Blank	35.5	-
2.	Initial	30	2200
3.	30 min	31.5	1600
4.	60 min	32	1400
5.	90 min	33	800
6.	120 min	34.5	520

► **COD Calculation:**

1. Initial: $\text{COD} = (35.5 - 30) \times 0.1 \times 8000 \div 2 = 2200 \text{ mg/lit}$
2. 30 min: $\text{COD} = (35.55 - 31.5) \times 0.1 \times 8000 \div 2 = 1600 \text{ mg/lit}$
3. 60 min: $\text{COD} = (35.5 - 32) \times 0.1 \times 8000 \div 2 = 1400 \text{ mg/lit}$
4. 90 min: $\text{COD} = (35.5 - 33) \times 0.1 \times 8000 \div 2 = 800 \text{ mg/lit}$
5. 120 min: $\text{COD} = (35.5 - 34.5) \times 0.1 \times 8000 \div 2 = 520 \text{ mg/lit}$

► **COD Reduction:**

1. 30 min: $(2200 - 1600 / 2200) \times 100 = 27\%$
2. 60 min: $(2200 - 1400 / 2200) \times 100 = 36\%$
3. 90 min: $(2200 - 800 / 2200) \times 100 = 63\%$
4. 120 min: $(2200 - 520 / 2200) \times 100 = 76.36\%$

ADVANTAGES

1. **Faster Pollutant Breakdown:**

Acoustic cavitation and H_2O_2 together break down pollutants faster.

2. **Reduced Chemical Use:**

Acoustic cavitation enhances H_2O_2 's effectiveness, reducing the need for chemicals.

3. **Less Sludge:**

Produces less sludge compared to traditional methods, reducing disposal costs

4. **Eco-Friendly:**

H_2O_2 breaks down into harmless water and oxygen, leaving no toxic by-products.

5. **Cost-Effective:**

Saves on chemical and operational costs, improving overall efficiency.

APPLICATIONS

1. **Food & Beverage Industry:**

Treats oils, fats, and organic matter.

2. **Pharmaceutical Industry:**

Removes pharmaceutical residues like antibiotics and hormones.

3. **Textile Industry:**

Degrades dyes, surfactants, and chemicals from dyeing.

4. **Petrochemical & Oil Refining:**

Breaks down oils, greases, and hydrocarbons.

5. **Pulp & Paper Industry:**

Removes lignin and sulfur compounds.

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*Thank
you!*