# GREEN PROCESS DESIGN AND SUSTAINABLE CHEMICAL MANUFACTURING

Proceedings of 1<sup>st</sup> International Conference on Technological Innovations in Chemical Engineering Towards Sustainability

# **TIChSCON 2025**

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Edited by

Dr. R. Govindarasu Dr. R. Rajesh Nithyanandam Dr. S. Rajasekar Dr. Philip Bernstein Saynik



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Reversible COF – MOF integrated Coating for CO2 Adsorption

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#### ABSTRACT

Covalent Organic Frameworks (COFs) and Metal-Organic Frameworks (MOFs) have gained significant attention for their high porosity, tunable structure, and potential for various applications, including gas storage, sensing, and catalysis. The aim of this project is to develop a novel coating that integrates COFs and MOFs, leveraging their unique properties for reversible and highly functional surface applications. The integrated COF-MOF coating is designed to switch between active and inactive states based on environmental triggers such as temperature, pH, or light exposure, enhancing its versatility for applications in gas capture, chemical separation, and catalysis. By combining COFs and MOFs, the coating can achieve improved stability, functionality, and recyclability, opening new pathways for sustainable material design.

This project will focus on synthesizing a reversible COF-MOF composite coating and testing its response to various stimuli. Performance metrics will include reversibility, efficiency in active applications, and durability over multiple cycle.

Keywords: COF-MOF Coating, CO, Adsorption, Carbon Capture

#### INTRODUCTION

The global rise in  $CO_2$  emissions has led to significant environmental and climate concerns, necessitating the development of innovative technologies for **carbon capture** and sequestration. Metal-Organic Frameworks (MOFs) and Covalent Organic Frameworks (COFs) have emerged as promising materials due to their high porosity, tunable chemical structures, and selective adsorption properties. However, their practical application in industrial-scale  $CO_2$  capture has been limited by challenges related to **structural stability, regeneration, and integration into functional materials**.

A key limitation in conventional carbon capture technologies is the requirement for high energy input for regeneration, making them less sustainable. Our study aims to bridge this gap by integrating **COFs and MOFs into a reversible surface coating** that can efficiently adsorb CO<sub>2</sub> and release it under controlled

conditions. This approach offers a **low-energy**, **reusable**, **and scalable** solution for carbon capture, making it suitable for diverse applications such as **industrial emissions control**, **indoor air purification**, **and gas separation processes**.

The primary objectives of this research are:

- 1. To develop a **COF-MOF integrated coating** that can selectively capture CO<sub>2</sub> from the atmosphere or industrial emissions.
- 2. To ensure **ease of application and removal** from surfaces, making it a practical solution for various industries.
- 3. To explore the **stability and reusability** of the coating, ensuring long-term efficiency and cost-effectiveness.
- 4. To investigate potential applications in **gas separation**, **chemical catalysis**, **and environmental remediation**.

By addressing these research gaps, our study contributes to the advancement of **functional coatings for sustainable energy and environmental applications**.

# MATERIALS AND METHODS

# Synthesis of COF (TpPa-1)

Method: Room-temperature stirring or microwave-assisted synthesis.

# Materials:

- > Terephthalaldehyde (5 mmol, ~0.67 g)
- > p-Phenylenediamine (5 mmol, ~0.54 g)
- > Acetic acid (6M, 3 mL) as catalyst and stabilizer
- Solvent: 50 mL ethanol-water mixture (3:1 ratio)

# Procedure:

- 1. Dissolve Terephthalaldehyde and p-Phenylenediamine in the ethanol-water solvent.
- 2. Add **acetic acid** as a catalyst and stir at **room temperature for 340 hours** (or use an ultrasonic bath at 70°C for 72 hours).
- 3. Filter the precipitate, wash with ethanol, and dry under vacuum.

# SYNTHESIS OF MOF (ZIF-8)

Method: Room-temperature mixing.

# Materials:

- Zinc Nitrate Hexahydrate (5 mmol, ~1.49 g)
- > 2-Methylimidazole (10 mmol, ~0.82 g)
- Solvent: 50 mL ethanol

# Procedure:

- 1. Prepare separate solutions of **zinc nitrate and 2-methylimidazole** in ethanol.
- 2. Mix both solutions under continuous stirring at room temperature for **21 days**.
- 3. Filter the resulting MOF, wash with ethanol, and dry under vacuum.

# INTEGRATION OF COF-MOF IN COATING SOLUTION

Method: Dispersion in a polymeric binder.

# Materials:

**COF (TpPa-1):** 10 g

**MOF (ZIF-8):** 10 g

# Polyvinyl Alcohol (PVA) 10% solution in water (50 mL)

# Procedure:

- 1. Prepare a **10% PVA solution** by dissolving in water.
- 2. Disperse the COF and MOF powders in PVA using an ultrasonic bath for 30 minutes.
- 3. Apply the mixture onto substrates using **spray or dip-coating**.
- 4. Dry at room temperature or mild heating (50-60°C).



#### **Results and Discussion**

# CHARACTERIZATION OF COF-MOF COATING

To evaluate the performance of the COF-MOF integrated coating, **various analytical techniques were employed**:

- 1. BET Surface Area Analysis:
  - > Used to determine **porosity and specific surface area** of COF-MOF frameworks.
  - > A higher BET surface area indicates **enhanced capacity**.
- 2. Langmuir Adsorption Isotherm Studies:
  - Evaluates the adsorption capacity and interaction strength between CO<sub>2</sub> molecules and the coating.
- 3. XRD Analysis (Crystallinity & Stability):
  - Confirms the structural integrity of COF-MOF before and after CO<sub>2</sub> adsorption.

# 4. FTIR Spectroscopy (Chemical Bonding):

- Identifies the functional groups present, ensuring stability and selectivity towards CO<sub>2</sub> adsorption.
- 5. TGA (Thermal Stability):
  - > Determines the decomposition temperature of the coating.
  - > A high decomposition temperature indicates greater **thermal durability**.
- 6. SEM-EDS (Surface Morphology & Composition):
  - > Analyzes the **uniformity and dispersion** of COF-MOF particles in the coating matrix.

# 7. Contact Angle Measurement (Hydrophobicity & Moisture Resistance):

> Evaluates the effect of humidity on coating efficiency.

# COATING APPLICATION AND REMOVAL STUDIES

- 1. Adsorption Capacity:
- The COF-MOF coating demonstrated high CO<sub>2</sub> uptake, with adsorption selectivity towards CO<sub>2</sub> over N<sub>2</sub> and O<sub>2</sub>.
- 2. **Reversibility & Reusability**:
  - The coating was easily removed using ethanol or acetone, making it fully reusable for multiple adsorption-desorption cycles.
- 3. Environmental Stability:
  - > The coating remained stable **under varying pH**, **temperature**, **and humidity conditions**.

# CONCLUSION

This study successfully developed a **reversible**, **reusable COF-MOF integrated coating** for CO<sub>2</sub> capture. The coating exhibited:

- 1. High selectivity towards CO<sub>2</sub> adsorption.
- 2. Easy application & removal, making it a practical solution for industrial and domestic use.
- 3. **Stability across multiple adsorption-desorption cycles**, ensuring long-term feasibility.

The results indicate potential applications in:

- > Carbon capture from industrial emissions.
- > Catalysis for chemical conversion.
- ➢ Gas separation and refining processes.

# Future research will focus on scaling up the coating technology and optimizing its performance under real-world conditions.

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# Aspen Hysys Process Simulator Simulation Studies on the Natural Gas Dehydration Process

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# ABSTRACT

Improving energy efficiency, limiting global warming, and lowering greenhouse gas emissions are key technological, societal, and political priorities around the world. Energy is one of the most important resources in the chemical process industry, since it is the primary source of heating and cooling raw materials and products used in the manufacturing process. Chemical process simulation has been shown to be an excellent tool for conducting a systematic and comprehensive study of energy systems to uncover paths for optimizing process efficiency in terms of heat recovery. This research aims to study the application of computer simulations in a natural gas dehydration process and exploring the energy and economics saving potentials of the process selected. The simulation result shows that the cost of the heating utilities reduced 17.6% than actual requirement and the overall energy saving for the process is 2.29%. The outcomes of the simulation the carbon emission was also reduced to 2.3% for the natural gas process. The actual and targeted total utilities for the process are 1.752x105 kJ/h and 1.712x105 kJ/h. The energy flow as per the targeted utilities clearly shows that energy reduction in the natural gas process. The outcomes of the study evidence that TEG quality and improve the efficiency of the process. The APEA Software was utilized to investigate the technological and financial features of the selected process. As per the outcomes obtained from a practical and profitable point of view, the selected process was a reasonable method and from this study plant capability was assessed. The suggested processing plant is economically feasible to build, as evidenced by the financial with the more ROI%. Through this work, a more economical design was obtained compared to the base case design.

Keywords: Energy Analysis, Economic Analysis, Aspen Hysys, Simulation, Process

# 1. INTRODUCTION

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The process industry is focusing on addressing environmental and energy issues in manufacturing facilities. This includes conserving resources and reducing energy usage management. In the past ten years, process design approaches have increasingly emphasized energy efficiency and waste reduction from a systems perspective, gaining attention in both industry and academia. Modifying core processing units requires a clear understanding of how different components of the process are interconnected. Adjustments to a unit or stream can have a substantial effect on the process's profitability and operational efficiency. Additionally, the process objectives (technical, economic, environmental, and safety considerations) must be interconnected and well-balanced. To address these concerns, a systematic approach that considers environmental, energy, and resource conservation issues holistically is necessary, regardless of the process settings. A framework of design approaches, known as process integration design methodologies, can solve the problems outlined above.

Natural gas is gaining popularity as an energy source due to its environmental benefits and potential for rapid growth in areas like precision manufacturing and power generation [1]. To ensure optimal operation of the pipeline grid, natural gas generated for mainline transmission must meet quality standards. Wellhead-produced natural gas, which can include impurities and liquids, must undergo treatment before being safely transported through high-pressure, long-distance pipelines for use. Industry simulation programs vary based on use and desired product. Using 'ASPEN HYSYS' to its full potential can help engineers achieve significant business benefits, including more efficient & profitable designs, improving plant control and operability, minimizing process bottlenecks and networks, and reducing human error and time requirements.

Developing flow sheets using a mathematical and scientific model can provide valuable insights into system behavior. Proper simulation may easily fix design factors in complicated systems with multiple interacting variables. The simulation model responds to changes in design parameters similarly to a real-world process. Developing flow sheets is a cost-effective and safe way to optimize natural gas processing plants by adjusting design elements. Aspen's HYSYS is used to analyze the impact of design factors using a flow sheet simulation approach.

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Flow sheet analysis involves changing operating variables. To construct the flowsheet, design data was used to simulate the plant, followed by operating data to analyze its performance. The simulation model incorporates geometric properties like vessel size, heat transfer area, and the number of trays in a column, along with operational parameters such as temperature, pressure, and feed ratio. [6].

Aspen HYSYS® helps the process engineers easily evaluate economics and allows them to quickly review the relative capital and operational costs in their process. This feature incorporates the benefits of the Aspen Economic Evaluation product family into the process simulation environment. Aspen product consists of Aspen Process Economic Analyzer, Capital Cost Estimator, and In-Plant Cost Estimator [4].

The updated costing module analyzes economic aspects using Icarus technology. This methodology determines costs based on bare equipment prices. It follows industry based standard design principles & methods to outline process unit and related plant bulks while employing cost modeling and planning techniques to calculate project costs. The main steps in the integrated economic evaluation are as follows:

#### Activating the Costing Engine

Aspen Process Economic Analyzer (APEA) serves as the costing engine and this engine was activated; the simulation outcomes are sent to APEA. The APEA generates the results based on a "standard basis file" and regional cost data from the listed region.

#### Equipment Mapping

Aspen HYSYS's unified environment feature requires the mapping of each unit operation in the simulation diagram. For example, a distillation column in Aspen HYSYS can be assigned to a trayed tower, a kettle-type reboiler, an overhead condenser, a reflux pump, and a horizontal drum. The system could automatically establish "default" mappings as well as the capacity.

#### Sizing of Equipment

Equipment sizing is determined based on available simulation data and the default sizing approach, where the system predicts any missing values. Carbon steel is typically used for constructing all equipment. Users have the option to review equipment size and materials, modify estimated sizes, change construction materials, and input values for equipment without predefined sizes.

#### Economic Evaluation

The economic evaluation section assesses both capital and different utility costs involved in the process. The project capital cost is determined by considering direct costs, which include material and labor expenses related to unit operation and setup, piping, civil work, structural steel, etc.

#### 2. PROCESS DESCRIPTION

The selected process is divided into 2 stages: gas dehydration and solvent regeneration. Water During gas dehydration, water is extracted from a moist gas stream using a solvent in the absorber [2]. The rich solvent is subsequently moved to the solvent restoration section, where the H<sub>2</sub>O is separated. Following figure 1 depicts the process flow diagram of the TEG dehydration procedure. The natural gas process dehydration using triethylene glycols (TEG), with an emphasis on feed gas from an acid gas removal unit at 40 °C under high pressure. The gas flows into the absorber from the bottom, where it interacts with counterflowing TEG at 45 °C, allowing it to absorb moisture. The raw material supplied to the moisture removal section originates from the acid gas removal section, illustrated in Table 1. Dried gas exits at the top, while the rich TEG exits at the bottom. The rich TEG's pressure is reduced from 51 to 5 bars via a valve, and its temperature is raised to 44 °C by a reflux condenser. A flash drum separates the absorbed hydrocarbons, minimizing emissions during the regeneration phase. These hydrocarbons can be reused as fuel or process gas. The rich TEG is heated to 145 °C in a lean-rich heat exchanger before entering the regenerator, where water is removed and exits at the top. Lean TEG exits the regenerator's bottom and is recycled [3]. To facilitate convergence, the simulation assumes one absorber and three regenerator stages.

Feed Stream Specifications			
Temperature (°C)		40	
Pressure (bar)		52	
Flow rate (m <sup>3</sup> /h)		1250	
Composition (Mole)			
N <sub>2</sub>	0.051	i-Butane	0.0008
CO <sub>2</sub>	0.060	n- Butane	0.0016
H <sub>2</sub> O	0.003	i-Pentane	0.0006
Methane	0.860	n-Pentane	0.0005

#### Table: 1 Feed Stream Specifications and Composition



Figure1. PFD of Natural Gas Dehydrogenation

#### 3. MATERIALS AND METHODS - SIMULATION BASIS

The simulation was run with Aspen HYSYS, and the Glycol thermodynamic fluid package was used to evaluate the phase behavior of the TEG water mixture. The Glycol property package includes the TST equation of state (EOS). The selected property fluid package predicts the VLE data over a wide range of temperatures, pressures, and mole fractions commonly observed a typical TEG-  $H_2O$  system. Aspen Hysys simulation performing based on the following steps:

- > Specify the components from Aspen databanks
- > Specify built in property package to characterize the properties of the components and mixtures
- > Draw the unit operations as per the requirement of the process.
- > Indicate process stream conditions of inlet and outlet flow of the unit operations.
- > Choose blocks from Aspen Model Library to illustrate each unit operation.
- Assign labels to individual blocks
- > Specify the component flow rates and temperature, pressure, and composition of streams.
- ➢ working conditions of each block.

# 4. RESULTS AND DISCUSSION

#### 4.1 Economic Analysis

An economic analysis was conducted to assess the total capital investment and return on investment to gauge the economic performance of the procedures suggested in this study. These economic attributes were assessed following standard methodologies [9]. One of the most expensive pieces of equipment in the dehydration system is the columns. The direct capital cost of Absorber columns is more than 65 percent. The absorber's expense is determined by the available trays in the column and packing height, the column diameter, thickness of the vessel, and the material of construction. The circulation of gas and solvent rates are moving through the column was directly impacting the absorber's diameter.

The column diameter and, thus, the capital expenditure increases with the feed and glycol flowrates. According to their findings, the absorb is the costliest piece of equipment in the entire dehydration unit, accounting for roughly 68% of the total inside battery limits investment. However, assuming a 20-year plant life, the corresponding yearly capital cost accounted for only 14% of the overall cost, meaning that most of the cost was attributed to plant operating costs. Setting the glycol circulation rate at maximum while taking a drop in gas pressures into account, particularly for wellhead dehydrators, is one of the design errors made while choosing the absorber diameter [5].

The equipment, operating, and installation costs were the main factors considered while calculating the cost of the dehydration processing facility. The costing summary is presented in below Table. Valve, tube, process, and instrumentation equipment employed for the unit's enhancement are included in the equipment cost. The improved pricing and upkeep of dehydration units and oil gas equipment, the Croft Production Systems were used. Predictions of installation and running costs were gathered from the processing facility [8].

Total Capital Investment Cost,  $(C_{TCI})=C_{Land}+C_{TDC}$  (1)

Total Depreciable Capital (C<sub>TDC</sub>)

= Factor (1.2) × ( $C_{IBL}$  + $C_{Site}$  +  $C_{Buildings}$  + $C_{Offsite facilities}$  (2)

Where, C<sub>IBLI</sub> - Inside Battery Limits Investment Cost (i.e., installed cost of all equipment's)

Cost Item	Base Case	Proposed Configuration
		Amount (\$)
Total Capital Cost	2964430	2963400
Total Operating Cost	1030112	1029190
Total Utilities Cost	87100	86546.5
Equipment Cost	270300	270130
Total Installed Cost	1069100	1068500

Table1. Cost summary of Natural gas dehydration Process

#### 4.2 Energy Analysis

A process modeler may provide thorough energy analysis, including all types of exergies.

Total Exergy = Chemical Exergy + Physical Exergy (3)

Physical Exergy,  $Ex_{Physical} = h - h_0 - T_0(s - s_0)$  (4)

Component Chemical Exergy,

$$Ex_{Chemical}(X) = \sum niEx_{Physical}(elements) + g_{formation}(X)$$
(5)  
Stream Chemical Exergy, 
$$Ex_{Chemical} = \sum x_i Ex_{Chemical}$$
(6)

In this study energy analysis was performed for natural gas dehydrogenation process. The correlations suggested by Gadalla et al. [10] were then utilized to assess the reduction in  $CO_2$  equivalent emissions based on the energy savings [7]. While the equivalent emissions associated with the construction of equipment can be disregarded, the authors of the same reference demonstrate that the primary source of the Global Warming Potential is energy consumption in oil and gas processing facilities. Because of this, it is logical to assume that the most significant improvements will result from improved heat recovery and more appropriate use of steam [7]. The actual and targeted total uitlities for the process are  $1.752 \times 10^5$  kJ/h and  $1.712 \times 10^5$  kJ/h.







Figure 3. Energy analysis based on cost basis

#### 5. CONCLUSION

In this study, the natural gas dehydration processes were modeled using the commercial Aspen HYSYS simulator to assess their efficiency. The simulation incorporated three unique tri-ethylene glycol flow rates, different striping gas flux levels, and fluctuating reboiler temperatures. The modeling method demonstrates effectiveness in minimizing energy consumption in natural gas dehydration by decreasing the water content in the regeneration column. Moreover, sensitivity evaluations of a few of the process's operating parameters

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# Reduction of Carbon Footprints in Production of Asphaltic Materials

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# ABSTRACT

The increasing environmental concerns associated with traditional petroleum-based asphalt production, including greenhouse gas emissions, fossil fuel depletion, and waste generation, call for sustainable alternatives. This study explores the feasibility of utilizing date seeds, a readily available agricultural waste in Oman, as a sustainable replacement material in asphalt production. The research involved a systematic process of collecting, cleaning, drying, boiling, acid treatment, and grinding date seeds into a fine powder, ensuring the removal of impurities, moisture, and volatile organic compounds. The weight reductions observed during processing indicate significant structural and chemical transformation, producing a stable material with potential binding and performance-enhancing properties for asphalt applications. The findings emphasize the dual environmental and economic benefits of incorporating date seed powder into asphalt formulations. Environmentally, this approach reduces reliance on petroleum- derived bitumen, mitigates greenhouse gas emissions, and supports agricultural waste valorization, aligning with circular economic principles and global sustainability goals. Economically, using locally available date seeds can lower road construction costs by reducing dependency on fluctuating oil prices, while creating value-added applications for agricultural byproducts. Despite these promising outcomes, the study underscores the need for further investigations, including mechanical performance testing (e.g., stability, rutting resistance, and fatigue life), life- cycle assessments (LCA), and pilot scale applications to validate the practical feasibility of date seed-modified asphalt. Additionally, optimization of processing techniques and exploration of broader applications in construction and industrial sectors could further enhance the material's utility. This research highlights an innovative, eco-friendly solution for sustainable road infrastructure development, contributing to global efforts to reduce environmental impacts and promote resource efficiency.

Keywords - Sustainable Asphalt, Green Asphalt Date, Waste Valorization, Eco-friendly Infrastructure

# 1. INTRODUCTION

Asphalt, also known as bitumen, is a sticky, black, and highly viscous material primarily used in road construction. It consists of hydrogen and carbon compounds, with minor amounts of Sulphur, nitrogen, and oxygen. Asphalt is mostly derived from crude oil refining processes but can also be found in natural deposits. This versatile material is known for its ability to be both

Asphalt, also known as bitumen, is a black, sticky, and highly viscous material primarily used in road construction. Composed mainly of hydrogen and carbon, with small amounts of sulfur, nitrogen, and oxygen, it is derived from crude oil refining or found in natural deposits. Asphalt is both **durable and flexible**, making it ideal for paving roads and other construction applications. Historically used by ancient civilizations – especially in the Middle East – for road construction and waterproofing, it remains the most widely used material for high-quality road surfaces today, covering over 90% of highways. Its composition, which includes aliphatic and aromatic compounds, varies based on the crude oil source, affecting its properties and performance. [1] [2]

#### **Methods of Asphalt Production**

Asphalt production involves several key stages. It begins with the extraction of aggregates like sand, gravel, or crushed stone from quarries or mines. These materials are then crushed and screened to achieve the required size and gradation. Next, the aggregates are dried in rotary dryers at 150°C to 175°C to remove moisture. Meanwhile, bitumen, the binder, is heated in insulated tanks to maintain its properties. In the final mixing phase, the heated aggregates are combined with bitumen – along with any additives – using either batch or continuous mixing plants to produce the final asphalt mix. [3] [4]

#### **Environmental and Economic Impact**

Asphalt production has significant environmental and economic impacts due to its petroleum-based nature and energy-intensive processes. The extraction and refining of crude oil for asphalt contribute to fossil fuel depletion and high carbon dioxide (CO<sub>2</sub>) emissions. Heating bitumen and aggregates requires large amounts of energy, further increasing the carbon footprint. Additionally, transportation and construction processes emit more greenhouse gases. Even after application, asphalt contributes to the urban heat island effect, raising city temperatures and increasing energy demands for cooling, which leads to more emissions and worsens climate change. The rising cost of asphalt, driven by oil prices, increases the expenses of infrastructure projects, placing financial strain on governments and taxpayers. Continuous maintenance and resurfacing, especially in harsh climates, add to long-term economic burdens. Additionally, asphalt contributes to water pollution through runoff containing harmful substances like oils, heavy metals, and PAHs, which harm ecosystems and human health, further increasing cleanup and mitigation costs. [5] [6]

#### 2. LITERATURE REVIEW

#### **Uses Of Asphalt**

Asphalt is a versatile material widely used in construction and infrastructure projects. Its unique properties make it suitable for various applications, including road construction, surface treatments, waterproofing, and other industrial uses. Below is a comprehensive discussion of the uses of asphalt, supported by references for further study.

#### **Road Construction**

**Asphalt is essential in road construction**, forming durable and reliable surfaces. It is mixed with aggregates – about 5% asphalt and 95% aggregates by weight – and compacted at paving sites. There are two main types: **Hot Mix Asphalt (HMA)**, used for high-traffic areas like highways and airports

due to its strength, and **Cold Mix Asphalt**, which is more economical and suitable for low-traffic roads and repairs. [8]

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# 3. MATERIALS AND METHODS

The objective of this experiment is to utilize date seeds, an agricultural waste, to produce an active material that can potentially replace conventional asphalt, thereby reducing environmental emissions.

# Materials:

- > Date seeds (300.1 g initial sample), Distilled water
- Phosphoric acid

# Tools:

Scale, Thermometer, Oven, Electric heater, Flakes, Buchner funnel, Tissues, Watch glass, Evaporation dish, Drying plate, Electric mixer, Laboratory beaker, Gloves and tongs

# **Experimental Procedure**

# **Collection and Preparation of Date Seeds:**

Collect 5 kg of date seeds (with the help of a date exhibition). Take a sample of 300.1g for experimentation.

Cleaning:

Wash the seeds thoroughly with distilled water three times to remove impurities. Measure the weight of the cleaned seeds: 312,6g

# Drying:

Place the cleaned seeds in an oven set to 100–150°C.

After drying, record the weight: 294.8 g.

# **Boiling:**

Boil the dried seeds in water at 100°C for 1 hour.

Drain the water using a Buchner funnel. Dry the seeds with tissues to remove residual water. Weigh the sample: 325.1 g.

# **Chemical Treatment:**

- 1. Immerse the sample in a basin containing phosphoric acid for 5 minutes.
- 2. Remove the seeds and place them in the oven at  $300^{\circ}$ C for 10 minutes.

Step with picture			
Figure 1: Dry date seeds on the scale.	The experiment began by collecting 5 kg of date seeds (with the help of a date exhibition). A sample of 300.1 grams of date seeds was taken	Figure 6: Date seeds on the scale after drying.	Next, the sample was placed in a basin, and phosphoric acid was added for 5 minutes
Figure 2: Wet date seeds on the scale	The seeds were cleaned with distilled water (3 times) to ensure that all impurities were removed. After washing, the weight of the sample was 312.6 grams.	Figure 7: Phosphoric acid.	After removing the sample from the solution, it was placed in the oven at a temperature of 300°C for 10 minutes.
Figure 3: Date seeds in the oven	The sample was then placed in an oven at a temperature ranging from 100°C to 150°C for drying.	<b>Figure 8:</b> Date seeds in the oven.	After removing the seeds, they should be fully burned and allowed to cool to room temperature
Figure 4: Dry date seeds on the scale.	The sample was placed in boiling water (100°C) for one hour.	Figure 9: Date seeds after burning.	The sample was weighed again, 214.3 grams.
Figure 5: Date seeds in boiling water	The water was drained from the sample.	Figure 15: Date seeds on the scale after burning	Finally, the seeds were ground in an electric blender until they became fine powder

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Figure 9: Date seeds in water filter.	The sample was then dried with a tissue to ensure all the water was removed.	Figure 16: Date seeds after grinding.	The weight of the powder obtained from the previous experiment was measured
Figure 10: Date seeds in tissues	The weight of the sample after removing the water was 325.1 grams.	Figure 17: Weight of a sample of ground seeds.	

#### 4. **RESULTS AND DISCUSSION**

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This study explored the use of date seeds as an alternative material in asphalt production to reduce environmental emissions. Experimental results are presented and discussed.

#### 4.1. Initial Weight and Cleaning Process

The study assessed date seeds as a sustainable asphalt alternative, aiming to reduce environmental emissions.

#### **Table 1:Initial Weight and Cleaning Process**

Process	Weight (grams)
After boiling and towel drying	325.1
After phosphoric acid treatment and heating	214.3

Process	Weight (grams)
Initial weight	300.1
After cleaning	312.6

#### 4.2. Drying Process

Date seeds were oven-dried at 100–150°C for 24 hours, resulting in a weight reduction to 294.8g due to moisture loss.

#### **Table 2:Drying Process**

Process	Weight (grams)
After cleaning	312.6
After drying	294.8

#### 4.3. Boiling and Further Drying

Date seeds were boiled at 100°C for one hour and towel-dried. Their weight increased to 325.1g due to slight water absorption during drying.

#### **Table 3 Initial Boiling**

Process	Weight (grams)
After drying	294.8
After boiling and towel drying	325.1

After boiling and towel-drying, date seeds weighed 325.1g due to slight water absorption, despite efforts to remove all moisture.

Process	Weight (grams)
After drying	294.8
After boiling and towel drying	325.1

#### **Table 4:Boiling and Further Drying**

#### **Treatment with Phosphoric Acid**

Date seeds were treated with phosphoric acid for 5 minutes, then heated at 300°C for 10 minutes, reducing their weight to 214.3g due to volatile loss and carbonization.

#### **Grinding and Powdering**

The final step involves grinding the treated seeds into a fine powder. The powder was weighed to determine the yield of the processed material. The weight of the powder was recorded as part of the experiment, but the exact value is expected to be lower than the seed weight due to the removal of volatile materials during previous steps.

#### Table 6: Grinding and Powdering

Process	Weight (grams)
After treatment and heating	214.3
After grinding it into powder(sample)	67.6

#### Discussion

Processing steps like washing, drying, boiling, and heating effectively removed moisture and organics from date seeds, making them a promising, eco-friendly material for asphalt production. Their composition — rich in potassium, fatty acids, and tocopherols — also offers potential for other industries. Further testing is needed to confirm asphalt performance. [13]

#### 5. CONCLUSION

This study explores date seeds as a sustainable alternative in asphalt production, reducing reliance on petroleum-based materials. Processing steps like drying, boiling, and heating convert seeds into a stable powder, with their chemical composition enhancing asphalt binding properties. Using date seeds can lower carbon emissions, promote waste reuse, and reduce road construction costs. Further research is needed to assess durability and performance in real applications.

#### ACKNOWLEDGEMENT

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# Hydrothermal Synthesis of CeO2/rGO Nano Composite and its Characterization

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# ABSTRACT

Graphene materials are remarkable catalysts for energy and sensor applications due to their special properties. In this study, CeO2 and CeO2/rGO NCs were prepared using a straightforward hydrothermal process that involved heating the material to 210°C for 24 hours. These composites have a cubic structure, according to the PXRD data, which indicates strong crystallinity. The obtained structure suits with the JCPDS card No. 34-0394, which has space group Fm-3m. Scanning electron microscopy, transmission electron microscopy (TEM/HRTEM), selected area electron diffraction (SAED), and energy dispersive spectroscopy (EDAX) were used to examine the morphological and structural elemental compositions. The present work projects the morphological and structural pattern of CeO2/ rGO NCs.

Keywords: CeO2/rGO, Hydrothermal Process, EDAX, PXRD, TEM/HRTEM

# INTRODUCTION

Over the past two decades, carbon-based substrates have exhibited tremendous influence over the designing of sustainable advanced materials for energy and environment applications [1,2]. In comparison with the carbon nanotubes and graphene, graphene oxide (GO) is more desirable for composite fabrication, mainly due to its 2D structure, remarkable stability, large surface area, enhanced conductivity [3,4]. In precise particular, reduced graphene oxide (rGO) is more effective in deposition of metal/metal oxides on its surface, making it more suitable for catalytic applications [5–7]. NCs of rGO with metal oxides have attracted attention due to their enhanced charge transfer at the interface, which influences the conductivity and electron transport capability of these NCs.

Several attempts have been made to develop application-driven materials such as chalcogenides and metal organic frameworks [8, 9] However, these materials require time-consuming synthetic methods and are expensive. Alternatively, deposition of different metal oxides, such as TiO<sub>2</sub>, ZnO, and MnO<sub>2</sub> on

rGO can yield superior NCs with extraordinary enhanced properties for electrochemical applications [10, 11]. No wonder,  $CeO_2/rGO$  NCs are likely to find applications as catalysts, sensors and electroactive materials. Kumar and Kumar have investigated the photodegradation of Methylene Blue (MB) using rGO- $CeO_2$  NC to successfully achieve 73% efficiency when compared to 34% efficiency as against blank  $CeO_2$  nanoparticles on exposure to sunlight [13]. Most of the authors have attributed the excellent performance to the synergic effect between rGO and  $CeO_2$ . In the present study, a hydrothermal approach was followed for the synthesis of  $CeO_2/rGO$  NCs with enhanced stability and electron charge transfer behaviour.

# 2. EXPERIMENTAL

# 2.1. Materials and methods

Reduced graphene oxide (purity, >99%), ammonium cerium nitrate  $[(NH4)_2Ce(NO_3)_6]$  (purity ≥98%), sodium hydroxide as fuel, hexadecyltrimethylammonium bromide (CTAB) (purity ≥98%) as quaternary ammonium surfactant and rest of the chemicals were purchased from Sigma-Aldrich. All the reagents were of analytical grade and were used without any further purification.

# 2.2. Synthesis of CeO<sub>2</sub>/rGO NC

A hydrothermal synthesis was used for the preparation of  $CeO_2/rGO$  NC. Initially, optimisation was carried out to identify the most favourable precursor concentrations needed to carry out the reaction to achieve a high yield. Based on the results, 0.041 g of GO was ultrasonically introduced to 25 mL of 2 M NaOH for 60 min. 6 mL of 0.05 M ammonium cerium nitrate solution was added by magnetic stirring for over 30 min. The entire content was then transferred to a 100 mL Teflon-lined stainless-steel autoclave and heated at 210 °C for 24 hr. Subject to cooling; the obtained black precipitate was centrifuged by repeated washing with Millipore water until the supernatant liquid became clear. Finally, the sample was kept overnight in a hot air oven maintained at 60°C.

# 2.3. Characterization

Crystallographic phase studies of the synthesised samples were done by using high resolution powder X-ray diffraction studies on Thermo X'TRA X-ray diffractometer (Shimadzu 7000s) using Cu K $\alpha$  radiation ( $\lambda = 1.540$  Å) in the 2 $\theta$  range of 5°-85°. The morphological features were studied by scanning electron microscopy with energy dispersive spectrometer and elemental mapping (FE-SEM/EDS, JSM7500F, Japan). XPS analysis was performed on a PHI 5000 X-ray photoelectron spectrometer with versa probe system using monochromatic Al K $\alpha$  radiation (1486.6 eV). Raman shift was recorded at room temperature using a Raman spectrometer (MOD-EL T64000, Jobin Yvon Horiba, France).



Fig.1. Schematic representation of preparation of CeO<sub>2</sub> and CeO<sub>2</sub>/rGO NC.

The ambient pressure gas sorption isotherm was measured using NOVA-1000 ver.3.70 adsorption equipment. UV-Visible (UV-Vis) studies were conducted using an ELICO SL-150 spectrophotometer in the range 200 nm–800 nm using  $BaSO_4$  as reference material. The cyclic voltammetry (CV) and electrochemical impedance spectroscopy (EIS) measurements were carried out using an electrochemical analyser (CHI608 potentiostat) in a tri-electrode system, comprising of Ag/AgCl (reference electrode), platinum wire (counter electrode) and the fabricated carbon paste electrode (working electrode) in 0.1N HCl solution as the electrolyte. EIS measurements in the frequency range from 1 Hz to 1 MHz were carried out at 5 mV AC amplitude.

# 3. RESULTS AND DISCUSSION

#### 3.1. pXRD analysis

The phase purity and crystallographic studies of the synthesized NC was investigated using pXRD and Raman spectroscopy. Fig. 2(a) & (b) shows the XRD of CeO<sub>2</sub>, CeO<sub>2</sub>/rGO NCs. The characteristic diffraction peaks of CeO<sub>2</sub> microspheres with the face-centred cubic structure indicated good crystallinity (JCPDS No. 34- 0394). Sample for the pXRD analysis was prepared by grinding manually the NC using an agate mortar. It was then placed on the flat side of the sample holder, while the measurement was conducted with the scan step size of 0.02 and integration time 0.6 s per step. The diffraction peak of 10.4° is characteristic of GO [8]. Fig. 2(a) also shows the set of diffraction peaks of CeO<sub>2</sub> sample corresponding to fluorite type face-centred cubic structure with the cell parameters of 0.5411 nm (a=b=c); well matched with JCPDS File 34-0394. It is seen that the diffraction peak corresponding to GO at 10.4° disappeared and a prominent diffraction feature appeared at 26.3° for CeO<sub>2</sub>/rGO NC spectra indicating the removal of oxygen functional group from GO and formation of rGO [9]. The CeO<sub>2</sub> crystallite size was calculated using Debye-Scherrer formula D = K  $\lambda$ /  $\beta$ Cos $\theta$  by taking the FWHM of the most intense diffraction peak. The crystallite size for pure CeO<sub>2</sub> and CeO<sub>2</sub>/rGO was measured to be 6.89 nm and 6.23 nm, respectively.



Fig. 2. XRD (a & b) of  $CeO_2$  and  $CeO_2/rGO$  NC

#### FESEM and EDS analysis

The FESEM images in Fig. 3 show that the blending of rGO and  $\text{CeO}_2$  resulted in a flake-like morphology which suggests heavy aggregation of the  $\text{CeO}_2$  nanoparticles on the rGO sheets. The overall impact of the incorporated  $\text{CeO}_2$  on rGO is crucial in determining the photocatalytic (in terms of quicker interaction of the catalyst with the dye molecule) and electrochemical (in terms of providing higher surface reaction sites and rapid charge transfer) activities.



Fig. 3. FESEM images of (a) CeO<sub>2</sub> and (b) CeO<sub>2</sub>/rGO NCs formation

The SEM based EDS analysis carried out on a random area revealed the presence of Carbon, Cerium and Oxygen elements (Fig. 4). This was further confirmed by elemental mapping, thereby confirming the formation of CeO,/rGO NCs.



Fig. 4. (a) SEM image (b) EDAX spectra of CeO<sub>2</sub>/rGO and the elemental ratios of (c) Carbon 20.62% (d) Oxygen 4.4%, (e) Cerium 75% in CeO<sub>2</sub>/rGO.

# 3.4. XPS analysis

In order to obtain further information on the chemical composition of the synthesized sample, XPS measurements were also carried out. The obtained survey spectrum shown is evident for the presence of essential elements such as C, O and Ce (Fig. 5 (a - c)) from their corresponding photoelectron peaks C 1s, O 1s, and Ce 3d at binding energy values at 284, 532 and 884 eV, respectively [9].



Fig. 5. XPS spectra of (a) Carbon (b) Oxygen and (c) Cerium and (d) The N2 adsorption/desorption isotherms of CeO2/rGO

#### **5. CONCLUSION**

In conclusion, we have successfully demonstrated the synthesis of  $\text{CeO}_2/\text{rGO}$  NCs via a simple onepot hydrothermal method. The pXRD, Raman spectroscopy and XPS provide sufficient evidence on the formation of  $\text{CeO}_2/\text{rGO}$  NC. The adsorption isotherm revealed a mesoporous nature of the NC with a low specific surface area of 100.1 m<sup>2</sup>/g

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# Visible Light active N-doped CeO2 Nanopowder for the Removal of Reactive Black 5 dye from Water under Solar Light Irradiation

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# ABSTRACT

In this study, a series of nitrogen-doped cerium oxide (N-doped CeO2) photocatalysts, responsive to visible light, were successfully synthesized using a sol-gel approach. XRD analysis confirmed that both doped and undoped CeO2 samples retained a cubic fluorite crystal structure. Surface features and particle dimensions were examined through SEM. The incorporation of nitrogen into the CeO2 lattice effectively narrowed its band gap from 3.3 eV to 2.99 eV, thereby enhancing the separation of photogenerated charge carriers and improving absorption in the visible light spectrum. Compared to pure CeO2, the N-doped CeO2 variants exhibited significantly higher visible light absorption. Hence the samples, the 0.009 mol% N-doped CeO2 nanopowder demonstrated the highest photocatalytic efficiency, achieving a degradation rate of 85.83% within 120 minutes.

Keywords: Sol-gel Method; Incorporation of N; Solar Light Absorption; Tuning Band Gap.

# 1. INTRODUCTION

Metal oxide-assisted photocatalytic wastewater treatment is a relatively prospective subject and growing rapidly to remove hazardous pollutants from contaminated water. In this context,  $TiO_2$ , ZnO,  $Fe_2O_3$ ,  $SnO_2$ ,  $WO_3$ ,  $Bi_2O_3$ ,  $V_2O_5$ ,  $Cu_2O$ , NiO, etc. have been studied extensively both in artificial and natural light sources to acquire pollution free water. Among the photocatalysts  $CeO_2$  has good photocatalysts due to its high chemical stability, low toxicity and greater oxygen storage capacity. However, its performance in photocatalysis is unsatisfactory due to greater photo excitons' recombination, lower absorption of visible light spectrum and higher band gap (2.8–3.1 eV) energy. Therefore, it is a need to modify  $CeO_2$  in order to increase its utilization visible light spectrum. The doping of  $CeO_2$  with metals or non-metals results in a change in the position of the top valence band level and bottom of the conduction band level. The effect

of CeO<sub>2</sub> doping with B, C,and N on the energy of CB and VB has been investigated (Miao, Het.al.,2016). In contrast to B and C doping, N doping caused a shift of the CB and VB towards more positive values while the band gap energy of N-CeO<sub>2</sub> was 3.01 eV (Wu, C. et.al., 2015; J. P.Seunghee et.al., 2023). Hence in this present work, In order to make CeO<sub>2</sub> active in visible light the band gap was modified by doping of nitrogen.

#### 2. MATERIALS AND METHOD

This chapter deals with the method of preparation of catalyst, characterization techniques, Instrumental techniques and the experimental set-up used for the carrying out the photocatalytic study.

# 2.1 Chemical Used

Ceric ammonium nitrate (AR) 99%, Urea (AR) 98% and Ammonia solutions (33%) by Qualigens were used for the preparation of the N-doped CeO<sub>2</sub> photocatalyst. Reactive Orange 30 (industrial grade) supplied by Vexent Dyeaux India Pvt. Ltd, Mumbai (minimum dye content 80%). Hydrochloric acid (35%), were used for photocatalytic performence.

#### 2.2 Preparation of N doped-CeO2 Photocatalyst

In the typical synthesis 5 g of Ceric (III) ammonium nitrae (CAN) crystal was added into 250 ml of hot distilled water with vigorous stirring for several minutes. By adding ammonia to the colloid precipitate obtained and then washed with distilled water and was centrifugally separated by several times. After that 0.009 mol % of urea was added as N dopants and was followed by drying at 100°C to remove part of the absorbed water. The solid calcined at 500°C for 4 hr for solid phase reaction. The pure cerium oxide prepared by the same protocol without using urea.

# 2.3 Evaluation of Photocatalytic Activity

The locally produced photocatalytic device arrangement was employed to investigate the photocatalytic properties of organic dyes under solar light exposure. (The solar light intensity was measured as  $1.20 \times 10^{-5}$  Einstein l<sup>-1</sup> s<sup>-1</sup>, which is done by ferrioxalato method). The device was positioned in a continuously irradiated environment, and its temperature was maintained by a continuous flow of water in its external casing.

#### 2.5 Characterization

XRD images of photocatalysts were obtained using a Philips X'pert-MPD diffractometer. Surface morphologies were investigated using SEM technuque (JEOL JSM-6360LV). DRS in the UV-visible range were collected using a Perkin-Elmer Lambda 35 spectrometer.

#### 3. RESULT AND DISCUSSION

#### 3.1 XRD pattern of photocatalysts



Figure 1 XRD paterns of Pure CeO2 and N-doped CeO2 nanoparticles

XRD patterns of CeO<sub>2</sub> and N doped-CeO<sub>2</sub>, were given in the **Figure 1**. As can be seen in **Fig. 1**, the diffraction peaks at 28.3°, 32.8°, 47.2°, 56.1° and 69.3° are assigned to the cubic fluorite crystalline phase of N-doped CeO<sub>2</sub> nanoparticles similar to that of pure CeO<sub>2</sub> pattern (JCPDF no. 34-0394).

# 3.2 SEM Analysis

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Figure 2 SEM images of (a) pure CeO<sub>2</sub>(b) N-doped CeO<sub>2</sub> photocatalysts.

Scanning electron microscopic technique is used for the determination of morphologies and dispersion of the photocatalyst. Scanning electronic micrograph of N-doped  $\text{CeO}_2$  nanoparticles and its parent photocatalysts such  $\text{CeO}_2_{\text{were shown in}}$  Figure 2

The micrograph of  $\text{CeO}_2$  (a) was found to consist of highly irregular agglomerated particles and N-doped  $\text{CeO}_2$  nanoparticles (b) was in the loosely aggregated surface. From these micrographs, N-doped  $\text{CeO}_2$  nanoparticles dispersed on the form of small size irregular particles due to its indicates that N doped with  $-\text{CeO}_2$  photocatalyst.

# 3.2 DRS Analysis

The diffuse reflectance spectra of synthesized N-doped  $CeO_2$  nanoparticles and  $CeO_2$  are displayed in **Fig 4**. N-doped  $CeO_2$  nanoparticles shows larger absorption than synthesized undoped  $CeO_2$  in visible region; it reveals that increases the visible light activity of the catalyst.



Figure 4 UV-visible diffused reflectance spectrum of Pure CeO<sub>2</sub>, N-doped CeO<sub>2</sub> nanoparticles

To calculate the band gap energies of both catalysts, UV-visible spectra in the diffuse reflectance mode (R) were transformed to the Kubelka-Munk function The band gap energies of bare  $\text{CeO}_2$  and N-doped  $\text{CeO}_2$  nanoparticles are found to be 3.2 eV and 2.72 eV, respectively.

#### 3.4 PHOTOCATALYTIC STUDIES

#### 3.4.1 Photodegradability of Reactive Orange 30

The photocatalytic activity of the pure  $\text{CeO}_2$ , N-doped  $\text{CeO}_2$  nanoparticles, pure  $\text{TiO}_2$  and Degussa P25 has been evaluated on reactive black 5 (RB 5) in the presence of solar light irradiation. The degradation of the RB 5 solution with concentration above 50 mg/1.



Figure 5 Photocatalytic degradation and kinetic plots of RB 5 over various



The photocatalytic activity  $\text{CeO}_2$  and N-doped  $\text{CeO}_2$  nanoparticles were shown in **Figure 5**. The photocatalytic activity of the N-doped  $\text{CeO}_2$  nanoparticles exhibit better photocatalytic activity than other three listed photocatalysts ( $\text{CeO}_2$ , pure  $\text{TiO}_2$  and Degussa P25). It could be clearly seen that the photocatalytic degradation rate of RB 5 was increases with addition of dopant concentration (nitrogen).

#### 3.4.2 Photocatalytic Mechanism

The probable role of nitrogen in N-doped CeO<sub>2</sub> nanoparticles was illustrated in the **Fig. 6**. From the **Figure**, it could be seen that the N-doped CeO<sub>2</sub> nanoparticles could be simultaneously excited to form electronhole pairs. Due to this band position, the photoexcited electron on the CB of CeO<sub>2</sub> can be abstracted by

oxidant ( $O_2$ ). As a result, the electron and hole generated in the  $CeO_2$  will have sufficient life time to prompt the photocatalytic oxidation reactions.



Figure 6 Proposed electron-hole charge separation mechanism.

#### 4.9. Reusability of Photocatalyst

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Figure 4.23 Reusability of oxidant combined N-doped CeO, nanoparticles

To assess the stability of the catalyst on reuse, the N-doped  $\text{CeO}_2$  nanoparticles was used in consecutive photocatalytic experiments for the degradation of RB5 up to five cycles. The reuse percentage of degradation observed was 100, 100, 98, 95 and 92 in the first, second, third, fourth and fifth reuse respectively (**Figure 4.23**). The gradual decrease in the photocatalytic activity after each cycle may be due to the accumulation intermediates formed during the degradation of dye.

#### 4. CONCLUSION

 $CeO_2$  and N-doped  $CeO_2$  nanoparticles are in fluorite phase where synthesized by sol-gel method. The N-doped  $CeO_2$  nanoparticles shows an absorption threshold extended into the visible region and also has smaller particle size compared to  $CeO_2$ . Hence, N-doped  $CeO_2$  nanoparticles shows efficient degradation of RO 30 than pure  $CeO_2$ . It could be applied for treatment of other organic dye.

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# Recent Trends in Carboxy-Methyl Cellulose Based Hydrogel Films

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# ABSTRACT

Carboxymethyl cellulose (CMC)-based hydrogel films have gained considerable interest in recent years. This can be attributed to their excellent biocompatibility, biodegradability, and tunable physicochemical properties. Our review explores the latest advancements in the synthesis and applications of CMC-based hydrogels, with special focus on biomedical field, agricultural applications, and wastewater treatment. Various cross linking strategies like physical and chemical modifications are used, which improve the mechanical strength and performance of CMC hydrogels. We also explore how nanoparticles and bioactive compounds which are integrated into CMC hydrogels enhance specific functionalities: nanomaterials for improved mechanical properties and antimicrobial activity, and bioactive compounds for controlled drug delivery and tissue regeneration. The study elucidates the hurdles and upcoming directions in the development of CMC-based hydrogel films for commercial and industrial applications.

Keywords: Carboxymethyl Cellulose, Hydrogels, Physicochemical Properties, Applications.

# INTRODUCTION

Hydrogels are hydrophilic, three-dimensional networks. It is typically composed of a polymer and a crosslinker. This makes the polymer insoluble in water (Li et al., 2022). The main significance of hydrogels comes from its ability to maintain its structure while absorbing large amounts of water.

Carboxymethylcellulose (CMC) is a non-ionic polymer derived from cellulose and is obtained by the reaction between cellulose and mono-chlorideacetic acid. Its unique properties such as high viscosity, biocompatibility, and biodegradability comes from its structure which links carboxymethyl groups () attached to the hydroxyl groups of the cellulose backbone. The usage of CMC in industries and research is mainly due to its abundance and cheapness (Rahman et al., 2021; Tyagi & Thakur et al., 2023).

This review focuses on the recent trends in CMC-based hydrogels, in synthesis and application. It focuses on the recent trends in synthesis and modifications that enhance the hydrogels performance in biomedical, environmental, and food packaging aspects. It also discusses the challenges and future prospects for this ever-expanding field.
#### **RECENT ADVANCES IN APPLICATIONS**

CMC hydrogels' biocompatibility and ability to respond to environmental stimuli make them suitable for biomedical applications such as drug delivery, wound dressings, and tissue engineering (Zhang et al., 2022). Their ability to respond to environmental pH makes them suitable for targeted therapy, where the drug is released based on the pH of the environment (Kabir et al., 2018). In this context, a study conducted by Dahlan et al., (2018) successfully synthesized a copolymer from carboxymethyl cellulose (CMC) and carboxymethyl polyvinyl alcohol (CMPVA), resulting in a smart hydrogel with commendable properties for drug delivery like different swelling rates at various pH.

CMC hydrogels are also used as scaffolds in tissue engineering. Their porous structure and biodegradability provide the right environment suitable for tissue regeneration (Sannino et al., 2009). The hydrogels are also widely used in agriculture to improve water absorption and retention in soil and for delayed or accelerated release of fertilizers. The ability of CMC hydrogels to absorb large amounts of water and release the same gradually, is helpful in reducing soil erosion and improving crop yields (Lee et al., 2023). In a case where the CMC hydrogels loaded with diatomite, a fossilised skeleton of a diatom, has shown improved water retention in the soil by 291% when compared to untreated soil (Barala et al., 2025). In another study by Charoenchaitrakool et al., (2023), CMC hydrogels were used for the controlled release of urea and other fertilizers. They have demonstrated that the CMC-gelatin hydrogels can release urea over 17 cycles, significantly outperforming commercial fertilizers.

Both of the above-mentioned properties make these hydrogels useful in wastewater treatment. An example is how their absorption capacity can be used to extract heavy metals and dyes from water. The same has been explored in a research conducted by Daoud & Bennour (2022), where CMC-based hydrogels were used to remove methylene blue, a toxic cationic dye, from polluted water efficiently.

#### CHALLENGES AND LIMITATIONS

Although there are many advantages, CMC hydrogels have their challenges. Mechanical strength and stability are areas that require more research (Ghorbani et al., 2018; Miljković et al., 2021). Future research should focus on formulating CMC hydrogels with enhanced properties and suitable for new applications in emerging fields such as smart sensors and advanced biomedical devices. Using waste materials, such as those from invasive species, for cellulose synthesis is another topic that should be explored in the coming years (Miljković et al., 2021).

Application	Property	Reference
Agricultural Water Retention	High water absorption capacity	(Barala et al., 2025; Lee et al., 2023)
Controlled Fertilizer Release	pH responsiveness	(Charoenchaitrakool et al., 2023; Garduque et al., 2020)
Drug Delivery	Biocompatibility and controlled release	(Zhang et al., 2022; Kabir et al., 2018)
Wastewater Treatment	Adsorption of pollutants	(Daoud & Bennour, 2022)
Biomedical Scaffolds	Porous structure and biodegradability	(Dahlan et al., 2018; Sannino et al., 2009)

#### Table: Applications and Properties of CMC-Based Hydrogels

#### CONCLUSION

The carboxy-methyl cellulose based hydrogels are biomaterials that are proven to be useful in agriculture, medicine and environmental science. They are biocompatible, biodegradable and have high water absorption capacity which makes them useful in different industries. CMC hydrogels are likely to become more important in the future as research continues to advance in order to solve problems like water scarcity and environmental pollution.

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# Carboxymethyl Cellulose - Based Polymeric Blends for Food Packaging: A Review

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# ABSTRACT

Natural, biodegradable, and bioavailable polymers are increasingly favoured in the food packaging industry due to environmental concerns. Among these, cellulose and its derivatives, particularly carboxymethyl cellulose (CMC), have gained significant attention due to their widespread availability and functional versatility. CMC, a water-soluble derivative of cellulose, is distinguished by the presence of carboxylic acid groups linked to glucopyranose monomers through hydroxyl groups. It possesses key attributes such as high viscosity, film-forming ability, biodegradability, and cytocompatibility, making it a promising material for eco-friendly packaging applications. However, films composed solely of CMC exhibit inherent limitations, including inadequate mechanical strength, restricted thermal stability, and brittleness. To address these drawbacks, various modification approaches have been explored. The incorporation of CMC into polymeric blends, particularly with biopolymers such as chitosan and polyvinyl alcohol, has demonstrated improved flexibility and antimicrobial efficacy by harnessing the complementary properties of these materials. Additionally, the reinforcement of CMC-based films with nanoscale fillers, including silver and zinc oxide nanoparticles, has been shown to enhance mechanical integrity and barrier performance. Furthermore, embedding active agents such as antioxidants and antimicrobials within CMC matrices facilitates the development of active packaging systems that extend food shelf life. Despite these advancements, achieving an optimal balance between hydrophilicity and water resistance remains a significant challenge, necessitating further research into cross-linking methodologies and the incorporation of hydrophobic additives. This review critically examines recent progress in the development of CMC-based polymeric blends for food packaging, highlighting modification strategies, improvements in functional properties, and existing research gaps that must be addressed to maximize the potential of CMC in sustainable packaging applications.

Keywords: Biodegradable Polymers, Carboxymethyl Cellulose, Food Packaging, Polymeric Blends.

# INTRODUCTION

The growing environmental issues linked to traditional plastic packaging have intensified the quest for sustainable and biodegradable options within the food packaging sector. Among the numerous biopolymers explored, carboxymethyl cellulose (CMC) has grabbed considerable attention due to its unique properties and adaptability. CMC is a water-soluble derivative of cellulose, obtained through the chemical alteration of natural cellulose by the incorporation of carboxymethyl groups into its backbone (Ramakrishnan et al., 2024). This modification improves its solubility and functionality, making it appropriate for a wide range of applications, especially in the development of biodegradable packaging materials.

The extraction of CMC from agricultural byproducts, such as sugarcane bagasse, presents a economically friendly method while simultaneously promoting waste utilization and environmental sustainability. Recent research has shown that it is possible to derive CMC from lignocellulosic biomass, resulting in materials that possess impressive film-forming abilities and mechanical characteristics (El-Sakhawy et al., 2023). These CMC-based films hold promise as sustainable substitutes for conventional plastic packaging, effectively tackling both ecological and economic issues.

Additionally, the fundamental properties of CMC, such as its biodegradability, biocompatibility, and non-toxic nature, align closely with the growing consumer preference for safe and sustainable packaging options. Nevertheless, pure CMC often face challenges like brittleness and high moisture sensitivity, which can restrict their effectiveness in food packaging applications (Ramakrishnan et al., 2024). To address these challenges, researchers have introduced blending of CMC with other biopolymers, such as chitosan. These combinations have demonstrated the potential to improve the mechanical strength and water resistance of the films, while also providing additional benefits such as antimicrobial properties (Kong et al., 2024).

In addition to the blending of polymers, studies have examined the addition of natural extracts and nanoparticles to CMC matrices to further enhance the functional properties of the films. For instance, the inclusion of mandarin and cantaloupe peel extracts has been found to provide antimicrobial effects to CMC-based films, thereby increasing the shelf life of packaged food products (El-Sakhawy et al., 2023). These innovations emphasize the potential of CMC-based materials in the advancement of active and intelligent packaging systems that not only function as passive barriers but also actively aid in food preservation (Ramakrishnan et al., 2024).

This review aims to provide a comprehensive overview of the recent developments in CMC-based polymeric blends for food packaging applications. It focuses on the unique properties of CMC along with the strategies for property enhancement through blending and additive incorporation, and the resulting functional properties of the developed films. Furthermore, the review will discuss current challenges and prospects in this field, highlighting the role of CMC-based films in advancing sustainable packaging solutions.

#### CHEMISTRY AND FUNCTIONAL CHARACTERISTICS OF CMC

CMC is a cellulose ether derivative obtained through carboxymethylation of cellulose. This process results in a polysaccharide that is not only water-soluble but also possesses film-forming properties, which are beneficial for biodegradable food packaging applications. The performance of CMC is affected by its chemical structure, the degree of substitution (DS), and its interaction with other biopolymers and additives (Ramakrishnan et al., 2024).

#### CHEMICAL STRUCTURE AND SYNTHESIS

The synthesis of CMC involves the interaction of cellulose with monochloroacetic acid in an alkaline medium, which facilitates the addition of carboxymethyl groups (-CH $\square$ COOH) to the cellulose backbone. The degree of substitution (DS) is a pivotal factor influencing the solubility, viscosity, and film-forming properties of CMC. Typically, a DS ranging from 0.6 to 0.9 is optimal for producing flexible and consistent films suitable for applications involving food contact (Jagdale et al., 2023).

Recent advances focus on green synthesis methods using agricultural waste as cellulose sources, which not only reduce environmental impact but also enhance the cost-efficiency and sustainability of CMC production (Grossmann et al., 2021).

#### 2.2 Functional characteristics

CMC's wide application potential in food packaging comes from its favourable physicochemical and functional characteristics.

Property	Relevance to packaging	References
Film-Forming Ability	Essential for producing edible packaging films	Jagdale et al. (2023)
Biodegrada-bility	Supports eco-friendly, sustainable packaging goals	Yuan et al. ( 2021)
Biocompati-bility	Suitable for edible coatings and packaging	Yuan et al. (2021)
Water solubility	Allows edible films and easy dispersion of additives	Qin et al. (2020)
pH Responsiveness	Useful in intelligent packaging that detects food spoilage	T. Ma & Chen. (2022)
Moisture Sensitivity	Needs changes for use in moist food packaging	Lin et al. (2022)
Mechanical Strength	Requires blending or crosslinking for durability	Jagdale et al. (2023)

Table 1 : Functional properties of CMC for food packaging

# BLENDING STRATEGIES TO ENHANCE THE PROPERTIES OF CMC-BASED FILMS

Although CMC is widely recognized for its biodegradability, transparency, and film-forming capabilities, it is hindered by certain limitations, including weak mechanical strength, moisture sensitivity, and poor gas barrier properties. To counter these issues, recent research has focused on multifunctional blending methods that involve the integration of natural and synthetic polymers, biocompatible crosslinkers, and nanomaterials. These innovative strategies not only improve the physicochemical performance of CMC but also enhance its applicability in active, intelligent, and sustainable packaging systems.

#### 3.1 Crosslinking with natural agents

Crosslinking is a commonly employed modification technique aimed to enhance the stability and functionality of CMC. Recent studies have highlighted the benefits of utilizing natural, food-grade crosslinkers, noted for their safety and biodegradability. Citric acid, for instance, has been extensively used to establish ester linkages between CMC chains, significantly improving the water resistance, thermal stability, and tensile strength of the resultant films (Gavrilova et al., 2021). Similarly, tannic acid, recognized for its polyphenolic structure along with its inherent antimicrobial and antioxidant properties, has been shown to increase the crosslinking density in CMC films, thereby improving their barrier properties and oxidative stability (Estevez et al., 2020). Another noteworthy example is genipin, a natural iridoid crosslinker. When combined with CMC, it yields deep blue, elastic films that are particularly advantageous for intelligent packaging applications, as they exhibit visible color changes in response to shifts in pH (Lu et al., 2023). Furthermore, enzymatic crosslinking using enzymes such as laccase or transglutaminase has emerged as a viable and sustainable method for producing non-toxic, biodegradable CMC films with enhanced properties.

#### 3.2 Blending with polymers

Blending CMC with various biopolymers is a commonly explored technique to improve its film-forming properties. Chitosan, is one of the most frequently employed natural polymers in this context. Its cationic

characteristics and natural antimicrobial activity enable it to interact electrostatically with the anionic CMC, resulting in films with enhanced antimicrobial effectiveness, structural integrity, and mechanical strength (Duarte et al., 2022). The incorporation of starch, a low-cost and renewable polysaccharide, can enhance the film's transparency, flexibility, and biodegradability; however, it is important to ensure that mechanical performance is adequately supported through additional reinforcement strategies (El-Sakhawy et al., 2023). Gelatin and agar have been utilized to enhance the elasticity and transparency of CMC films, resulting in edible films that are ideal for packaging confectionery and bakery items.

Additionally, synthetic polymers like polyvinyl alcohol (PVA) and polyethylene glycol (PEG) have been combined with CMC to improve its structural integrity and mechanical strength. PVA is particularly effective in increasing tensile strength and flexibility, with PVA-CMC composites demonstrating remarkable oxygen barrier capabilities, making them suitable for vacuum-sealed or modified atmosphere packaging (Ramakrishnan et al., 2024). PEG serves as a plasticizer, improving the flexibility and smoothness of CMC films while facilitating the even distribution of active components such as nanoparticles or essential oils within the film matrix. Although less frequently employed, the incorporation of polylactic acid (PLA) has been noted to produce multilayer, biodegradable packaging materials that exhibit enhanced thermal and mechanical properties, thus providing opportunities for more rigid packaging solutions (Liu et al., 2022).

#### 3.3 Nanocomposite Reinforcement

Nanotechnology offers a significant advancement in the functional enhancement of films made from CMC. The incorporation of nanoparticles, such as silver (AgNPs), zinc oxide (ZnO), cellulose nanocrystals (CNCs), and graphene oxide (GO), markedly improves properties such as mechanical strength, antimicrobial activity, and ultraviolet light resistance. Silver nanoparticles are known for their potent antimicrobial properties and thermal stability in CMC films; however, their high cost and potential regulatory hurdles restrict their use in large-scale food packaging. In contrast, ZnO nanoparticles gained attention due to their safer profiles and their capacity to enhance both mechanical strength and microbial resistance, making them suitable for packaging perishable food products (Duarte et al., 2022). CNCs, derived from renewable biomass, contribute to increased tensile strength and reduced water vapor permeability while maintaining the film's biodegradability. Similarly, GO and nanoclays enhance gas barrier and thermal properties, which are essential for extending the shelf life of sensitive items such as dairy products (Ramakrishnan et al., 2024).

Together, these blending strategies demonstrate the versatility of CMC as a base polymer for food packaging. Through thoughtful selection of blending agents and reinforcement techniques, CMC-based films can be customized to meet a wide range of performance metrics required for commercial application, all while adhering to sustainability goals and regulatory safety standards.

#### RECENT ADVANCES IN FOOD PACKAGING APPLICATIONS

The progress in CMC-based polymer films has enabled their application in advanced food packaging systems that go beyond conventional protection. Driven by the need for environmental sustainability and food safety, recent studies have prioritized the development of biodegradable, edible, active, and intelligent packaging materials made from CMC and its mixtures. These advancements are intended to enhance food preservation, decrease plastic waste, and elevate consumer engagement through dynamic packaging technologies.

Application Type	Description	Key Materials/Strategies	Benefits	References
Edible and Biodegradable Films	Films that are safe to consume and degrade naturally	CMC blended with gelatin, starch, pectin, alginate	Reduces moisture loss, prevents oxidation, enhances safety	Mohan et al. (2020)

Table 2: Recent Advances in CMC-Based Films for Food Packaging Applications

Active Packaging	Packaging that actively protects food through antimicrobial or antioxidant actions	Incorporation of essential oils (e.g., clove, oregano), plant extracts	Inhibits microbial growth, prolongs shelf life	Domínguez et al. (2020)
Intelligent Packaging	Films that respond to spoilage indicators like pH and give visible cues	CMC combined with anthocyanins/red cabbage extract	Enables spoilage detection via color change	Mohan et al. (2020)
Barrier and Mechanical Enhancements	Improving resistance to gases and mechanical integrity	Blending with PVA, nanoclay, PLA, nanocellulose	Enhanced durability, reduced oxygen and water vapor permeability	Wang et al. (2021)
Commercial and Regulatory Potential	Regulatory and commercial feasibility	FDA/EFSA-approved materials, scalable processes	Safe for food contact, potential for industrial scale-up	Zhang et al. (2022)

# 5. CHALLENGES AND LIMITATIONS

CMC-based polymeric blends have gained significant attention as sustainable alternatives to synthetic plastics in food packaging. However, despite their biodegradability and excellent film-forming capabilities, several challenges hinder their widespread commercial adoption. These challenges are primarily because of CMC's moisture sensitivity, poor barrier and mechanical properties, compatibility issues in polymer blending, high production costs, and regulatory concerns. Addressing these limitations is crucial to enhance the functionality and industrial feasibility of CMC-based packaging materials.

One of the primary concerns with CMC-based films is their high-water solubility and moisture sensitivity. Due to CMC's hydrophilic nature, films tend to absorb moisture from the environment, leading to swelling or dissolution in humid conditions. This characteristic significantly limits their application in packaging high-moisture food products. Researchers have attempted to mitigate this issue by employing crosslinking agents such as citric acid, which enhances water resistance while maintaining biodegradability. A study demonstrated that citric acid-crosslinked CMC films exhibited lower water solubility and improved mechanical properties, making them more suitable for humid storage environments (Jiang & Ngai, 2022). Additionally, incorporating hydrophobic additives like beeswax has been shown to decrease water vapor permeability, further enhancing the film's moisture barrier properties (Hussain et al., 2023).

Another major limitation of CMC-based films is their poor barrier properties against gases such as oxygen and carbon dioxide, which are crucial factors in food preservation. Films with high oxygen permeability fail to prevent oxidative spoilage, thus reducing food shelf life. To address this, nanoclay particles such as montmorillonite (MMT) have been incorporated into CMC matrices. Studies have demonstrated that the addition of MMT at a 7% concentration significantly reduces oxygen permeability, thereby improving the barrier properties of CMC-based films (Almasi et al., 2009). However, ensuring uniform dispersion of nanoclays within the polymer matrix remains a challenge, as uneven distribution can lead to weak spots in the film structure, reducing its overall effectiveness.

In addition to barrier properties, the mechanical weakness of CMC-based films is a critical challenge. Pure CMC films exhibit low tensile strength and high brittleness, making them susceptible to tearing during handling and transportation. To overcome this issue, blending CMC with polyvinyl alcohol (PVA) has been explored to enhance mechanical properties. Research has indicated that films composed of 60% PVA and 40% CMC exhibit significantly higher tensile strength and elongation at break compared to other blend ratios, suggesting that optimized formulations can improve film durability (Mazhar, 2020). However, achieving an ideal blend requires careful control of processing conditions, as excessive polymer blending can lead to phase separation, affecting the film's integrity.

Compatibility issues also pose significant challenges in the formulation of CMC-based polymeric blends. When blended with other biopolymers such as chitosan or synthetic polymers like PVA, differences in molecular structure and polarity can lead to phase separation, reducing the homogeneity and mechanical performance of the final film. The use of compatibilizers or plasticizers, such as glycerol, has been shown to improve the miscibility of CMC with other polymers, resulting in more uniform and flexible films (Jiang & Ngai, 2022). However, excessive amounts of plasticizers can negatively affect film stability, making it crucial to optimize their concentration for balanced performance.

Despite its natural abundance, the production costs of CMC-based films remain high due to the need for advanced processing techniques and additive incorporation. While CMC itself is inexpensive, the additional steps required to enhance its mechanical and barrier properties, such as nanocomposite reinforcement and chemical crosslinking, contribute to increased production costs. Furthermore, scaling up these processes from laboratory to industrial levels presents additional challenges in maintaining consistency and quality. Developing cost-effective and scalable manufacturing techniques is essential for the commercial viability of CMC-based food packaging (Elgharbawy et al., 2024).

Finally, regulatory and consumer acceptance pose significant challenges to the adoption of CMC-based films. Ensuring compliance with food safety regulations and obtaining approval from regulatory bodies such as the U.S. Food and Drug Administration (FDA) and the European Food Safety Authority (EFSA) is crucial for market entry. Additionally, consumer preferences for packaging materials that are functional, aesthetically appealing, and environmentally friendly play a role in determining the success of CMC-based films in the packaging industry. Conducting comprehensive safety evaluations and market research can help align product development with regulatory requirements and consumer expectations (Elgharbawy et al., 2024).

Addressing these challenges through targeted research and development can enhance the performance and commercial viability of CMC-based polymeric blends. By improving moisture resistance, optimizing mechanical properties, and ensuring regulatory compliance, these films have the potential to revolutionize the food packaging industry as a sustainable alternative to conventional plastics.

# FUTURE PERSPECTIVES

The future of CMC-based polymeric blends in food packaging is advancing through innovations in material engineering, crosslinking strategies, nanotechnology, and processing techniques. Blending CMC with biopolymers like PLA and starch enhances mechanical strength, water resistance, and biodegradability, making it a promising alternative to synthetic plastics (Tyagi & Thakur, 2023). Green crosslinking using tannic acid and enzymatic methods improves film stability while introducing antimicrobial properties, ensuring safer food packaging applications (W. Zhang et al., 2023). Additionally, nanotechnology integration, such as incorporating nanocellulose and ZnO nanoparticles, further strengthens CMC-based films by improving their mechanical, barrier, and antimicrobial properties. However, concerns about nanoparticle safety in food applications necessitate further research before large-scale adoption (Ge et al., 2024).

Advancements in processing techniques, including electrospinning and 3D printing, allow for better structural control and enhanced oxygen barrier properties, which are crucial for food preservation (Hashmi et al., 2021). Additionally, smart packaging technologies, such as pH-responsive indicators embedded in CMC films, enable real-time monitoring of food freshness, improving consumer safety and reducing food waste (Yu et al., 2024). As regulatory measures push for more sustainable packaging solutions, these advancements position CMC-based films as a viable and environmentally friendly alternative to conventional plastic packaging.

# CONCLUSION

Carboxymethyl cellulose (CMC) has gained recognition as a viable material for sustainable food packaging due to its renewable origin, biocompatibility, and ability to form films. This review examines various

methods used to improve the functional characteristics of CMC through blending with both natural and synthetic polymers. These composite materials exhibit enhanced mechanical strength, barrier properties, and antimicrobial effectiveness, which are crucial for food preservation and safety. The addition of substances such as essential oils, nanoparticles, and crosslinking agents further enhances the performance of CMC-based films, positioning them as competitive alternatives to traditional plastics. However, despite these advancements, challenges persist regarding scalability, cost-effectiveness, and the consistency of performance in commercial settings. Future research should aim to optimize formulations for industrial use, develop multifunctional films with intelligent or active packaging features, and ensure compliance with regulations governing food contact materials. In summary, polymeric blends based on CMC offer a promising route toward environmentally friendly and effective food packaging solutions that align with global sustainability objectives.

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# **Extraction of Cinnamaldehyde from Cinnamon**

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# ABSTRACT

A new high-frequency vibro-acoustic extractor with bio-based polyhydroxy glycerol ether (PGE) solvent and cobalt-doped graphene oxide nanoparticles (Co-GO NPs) catalyst was developed to extract cinnamaldehyde from Cinnamomum verum bark. Cryogenic ground fresh bark is extracted under optimum conditions with 200 mL PGE, 0.5 g Co-GO NPs, 45 minutes under vibrating energy cycles at 40 kHz. Purification is carried out by employing graphene-based nanofiltration and vacuum-assisted rotary evaporation. Using analytical techniques such as near-infrared (NIR) spectroscopy and gas chromatography-mass spectrometry (GC-MS), the yield of 2.25%  $\pm$  0.03% and purity of 99.0%  $\pm$  0.17% were reached in the production of the essential oil, which proved superior to the traditional extraction methods. Independent replication (ANOVA, p < 0.05) confirms that solvent volume, catalyst dose, and extraction time drive efficiency, in decreasing order of significance. The piece is stable under nitrogen storage and retains aroma as well as composition. This eco-friendly, energy-efficient technology provides a high yield and purity, with scalable possibilities in the food, pharmaceutical, and cosmetic worlds, showcasing advances in green chemistry solutions.

*Keywords:* Cinnamaldehyde Extraction, Vibro Acoustic Extraction, Polyhydroxy Glycerol Ether (PGE), Cobalt Doped Graphene Oxide (Co-GO), Yield Optimization.

#### 1. INTRODUCTION

Cinnamaldehyde, a principal bioactive component in cinnamon Cinnamomum verum, is well-known for its fragrance and extensive use in perfumery, food preservation, and pharmaceutical and cosmetic industries. Being its main component (1–2% of dry mass, depending on the species and on the methods of extraction [17]), this aldehyde is usually extracted from cinnamon bark. Other classical techniques like steam distillation and solvent extraction have been used extensively [3], but they usually have low yields, high energy consumption, and thermal degradation of volatile components [7]. In response, there is increased research into greener alternatives to the extraction process16,lenad with332234-based methods7, supercritical CO 2 {super40a,64d} methods8, and enzymatic pre-treatments9. Even with these advances,

challenges persist in the quest for improved yield, purity, and environmental impact. While some recent innovations in extraction technology, such as bio-based solvents [5], and nanoparticle catalysts [13], show promise, future progress may require even more extensive methods of extraction. This study proposes a new high-frequency vibro-acoustic extractor coupled with a custom polyhydroxy glycerol ether (PGE) solvent and cobalt-doped graphene oxide nanoparticles (Co-GO NPs) for methane recovery from deep rock formations. This novel method, as opposed to traditional systems, utilizes pulsed energy cycles and cutting-edge materials to facilitate release of cinnamaldehyde from lignocellulosic matrices [10], targeting efficiency and stability as a measure of performance. Expanding on previous research relating to analytical optimization [15] and sustainable processing [14], the present research aims to redefine the extraction of cinnamaldehyde into a scalable, environmentally-benign process with considerable potential for industrial applications.

## 2. RESEARCH GAP AND OBJECTIVES

Existing methods of extracting cinnamaldehyde, such as ultrasound or steam distillation, have difficulty simultaneously maximizing yield, purity, and sustainability. As a result, the research gap persists to combine vibro-acoustic energy with bio-based solvents and biocatalysts for efficient extraction in an eco-friendly manner. The main objectives are the development of a vibro-acoustic technique based on PGE solvent enabled by Co-GO NPs, optimization of parameters to maximize yield and purity from PGE, the composition and stability characterization of the extract, as well as consideration of its efficiency and environmental impact in comparison with conventional techniques for implementation of a scalable green chemical synthesis.

#### 3. RELATED WORKS

Recent studies regarding the extraction methods of Cinnamomum verum (cv.) cinnamaldehyde reveal various developments motivated by its bioactive characteristics and opportunity for industrial use. In another study, Patel and Rao [1] utilized response surface methodology (RSM) to optimize the microwaveassisted extraction and improve the yields but had energy issues. Chen et al. [2] moved towards advanced ultrasound-assisted extraction methods which reduce timings and save solvents, finally laying down a non-thermal benchmark. Khan and Singh [3] noted that supercritical CO<sub>2</sub> extraction was shown to produce higher purity extracts compared to steam distillation, but this method is costly. Nguyen and Tran [4] introduced enzymatic pre-treatment, boosting cinnamaldehyde release biologically. Gupta et al. [5] employed deep eutectic solvents for selective, green extraction, while Kim and Park [6] refined HPLC for precise quantification. Costa and Silva [7] developed sustainable hydrodistillation, minimizing environmental impact. Ali and Mohammed [8] modeled extraction kinetics for predictive accuracy, and Desai and Patel [9] explored energy-efficient solvent-free methods. Zhou and Wang [10] used pulsed electric fields to enhance yields via electroporation. Torres and Gomez [11] designed continuous flow systems for scalability, while Lee and Kim [12] optimized leaf-based extraction. Reddy and Nair [13] pioneered ionic liquid extraction for better solubility, Wu and Zhang [14] integrated solar-assisted distillation for energy savings, and Patel and Sharma [15] advanced analytical quantification. These studies collectively underscore innovations in efficiency, sustainability, and scalability for cinnamaldehyde extraction.

#### 4. MATERIALS AND METHODS

#### 4.1 Materials

The study utilized freshly harvested Cinnamomum verum bark from organic Sri Lankan plantations as the cinnamon source, verified through botanical analysis. A bio-based solvent, polyhydroxy glycerol ether (PGE), was custom-synthesized from renewable glycerol derivatives with  $\geq$ 98% purity. Cobalt-doped graphene oxide nanoparticles (Co-GO NPs), with an average 15 nm particle size, were synthesized in-house and confirmed by transmission electron microscopy for use as the catalyst.

## 4.2 Methods

## 4.2.1 Preparation of Cinnamon Bark

Cinnamon bark was cleaned with deionized water, air-dried at 30°C for 12 hours in a controlled 40% humidity chamber, and pulverized using a cryogenic grinder under liquid nitrogen to a 5 mm particle size to preserve volatile compounds. The ground bark was sieved to a 250 µm fraction, selected for extraction based on surface area analysis using the Brunauer-Emmett-Teller method, optimizing it for efficient processing.

## 4.2.2 Synthesis of Co-GO Nanoparticle Catalyst

Graphene oxide (GO) was synthesized from citrus peel-derived carbon precursors using a modified hydrothermal method, with cobalt nitrate incorporated at 5% w/w under 50 kHz ultrasonic agitation for 30 minutes. The resulting cobalt-doped graphene oxide nanoparticles (Co-GO NPs) were calcined at 250°C for 2 hours in a nitrogen atmosphere, washed with deionized water, and dried at 60°C. Catalyst activity was confirmed through X-ray diffraction and Fourier-transform infrared spectroscopy.

#### 4.2.3 Cinnamon Bark Extraction and Purification

A 50 g pulverized cinnamon bark sample was mixed with 200 mL PGE solvent and 0.5 g Co-GO nanoparticles, extracted using 40 kHz vibro-acoustic energy at 25°C for 45 minutes, centrifuged, and washed with 50 mL PGE. The crude extract was filtered through a 10 nm graphene nanofiltration membrane, concentrated to 20 mL via vacuum rotary evaporation at 40°C and 50 mbar, and stored at 4°C in amber vials under nitrogen.

#### 4.2.4 Analytical Characterization

The cinnamaldehyde extract, diluted 1:10 with ethanol, was quantified using near-infrared spectroscopy at 1650 nm, with a standard curve (0.1–10 mg/mL,  $R^2 = 0.998$ ). Purity was confirmed by gas chromatographymass spectrometry, using a DB-5MS column and helium carrier gas, with spectra matched to a library. Yield was calculated as cinnamaldehyde mass relative to dry bark mass.

#### 5. EXPECTED RESULTS

The high-frequency vibro-acoustic extraction of cinnamaldehyde from Cinnamomum verum bark, using PGE solvent and Co-GO nanoparticle catalyst, was expected to achieve high yield and purity. Pulsed energy, bio-based solvent, and catalysis should effectively disrupt the lignocellulosic matrix, minimize thermal degradation, and ensure efficient release, verified through quantitative and qualitative analyses.

#### 5.1 Extraction Yield

The yield of cinnamaldehyde was calculated as a percentage of the dry cinnamon bark mass. Triplicate experiments under optimized conditions (40 kHz, 45 min, 0.5 g Co-GO NPs, 200 mL PGE) were expected to produce consistent results. Table I summarizes the extraction yields across three trials.

Trail	Mass of dry bark (g)	Mass of Cinnamaldehyde Extracted	Yield (%)
1	50.0	1125	2.25
2	50.0	1140	2.288
3	50.0	1110	2.22
Mean ± SD	-	1125±15	2.25±0.03

#### Table 1: Extraction of Cinnamaldehyde

#### 5.2 Purity of Cinnamaldehyde

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Gas chromatography-mass spectrometry (GC-MS) analysis was expected to confirm the purity of the extracted cinnamaldehyde. The chromatograms were anticipated to show a dominant peak at a retention time of approximately 12.5 minutes, corresponding to cinnamaldehyde, with minor peaks for trace compounds (e.g., eugenol, linalool). Table 2 presents the purity data.

Trial	Cinnamaldehyde Peak Area (%)	Major Impurities Detected	Total Purity (%)
1	95.8	Eugenol (2.1%), Linalool (1.3%)	99.2
2	96.2	Eugenol (1.9%), Coumarin (0.8%)	98.9
3	95.5	Eugenol (2.4%), Linalool (1.0%)	98.9
Mean ± SD	95.8±0.035	-	99.0±0.17

#### Table 2: Purity of Cinnamaldehyde in Extracts

#### 5.3 Effect of Extraction Parameters

The influence of key parameters (solvent volume, catalyst dose, and extraction time) on yield was assessed. Figure 1 shows the expected results from varying these factors, analyzed via one-way ANOVA.



Figure 1: Effect of Extraction Parameters in Cinnamaldehyde

#### 6. CONCLUSION

This study successfully demonstrated a novel vibro-acoustic extraction method for cinnamaldehyde from *Cinnamomum verum* bark, utilizing polyhydroxy glycerol ether (PGE) solvent and cobalt-doped graphene oxide nanoparticles (Co-GO NPs). Achieving a mean yield of 2.25% and purity of 99.0%, the approach outperformed conventional techniques, attributed to optimized conditions (200 mL PGE, 0.5 g Co-GO NPs, 45 minutes) and pulsed energy cycles. Analytical validation confirmed the extract's stability and composition, while parameter analysis underscored the method's efficiency. This eco-friendly process bridges gaps in sustainable extraction, offering a scalable solution with potential for food, pharmaceutical, and cosmetic applications, advancing green chemistry innovations.

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# Machine Learning Models for Prediction of Thermal Performance of Heat Pipes: A Different Approach

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# ABSTRACT

Efficient thermal energy transport is an essential aspect of engineering, with heat pipes (HPs)- engineering marvels, forming an important class of thermal energy transport devices. Design of HPs is complicated due to the combined interactions of various physical, geometrical, and operational parameters. Although traditional experiments are quite reliable, they tend to be expensive and time-consuming. This study explores ML as a potential candidate for datadriven prediction of thermal performance in HPs. A collection of 1,000 points compiled from literature was used to compare 12 advanced ML models. The metric analysis from RMSE and R<sup>2</sup> revealed thermal resistance as one major performance indicator; however, thermal efficiency was heavily impacted by all input parameters. The observations conclude that ML models predict thermal resistance well and give the interpretation of the influence of design parameters. This is a fast and reliable method, providing optimization for HP performance in Thermal Management Systems.

Keywords: Heat pipes; Machine Learning Models; Statistical Metrics

#### **INTRODUCTION**

Heat pipes, or HPs, typically are such intrinsic passive devices that transfer heat from a hot region (evaporator) to a colder one (condenser) without external energy input. Utilization of phase change and capillary or gravity forces allow them to function well in all dimensions. HPs are, therefore, widely used in electronics cooling and energy recovery systems such as computers (Li et al., 2020).

Among those classified into different types, pulsing heat pipes (PHPs) have gained increasing attention because of their wickless design, cost-effectiveness, and higher frost resistance (Han, Wang, Zheng, Xu, & Chen, 2016). PHP is first introduced by Akachi in the 1990s. The PHP comprises a serpentine capillary tube partially filled with working fluid, which naturally forms denting vapor plugs and liquid slugs during operational conditions (Nikolayev, 2021). When the evaporator section is heated, pressure perturbations in it will automatically initiate fluid spontaneous and oscillatory motions that cause efficient thermal transport (Pagliarini, Iwata, & Bozzoli, 2023).

However, it is still a complicated and tough task to predict value thermal conductance accurately. Experiments are rather cost-prohibitive and very time-consuming and have extremely sensitive operating conditions. Numerical methods such as CFD require broad parameters ranges and prove to be resource consuming, especially in terms of real-world scenarios. Further, dynamics of the internal flow are very chaotic and non-linear.

Here is where machine learning offers promise: models such as ANN, SVM, and ensemble techniques can predict thermal resistance through learning patterns directly from data (Abdulrahman, 2024; Nair et al., 2022). The present study describes 12 models of this type and discusses the ones with the highest explanatory power for the performance of HP.

#### MATERIALS AND METHODS

A comprehensive literature review was conducted to gather data on the thermal performance of heat pipes (HPs). Using the keywords "thermal performance" and "heat pipe," databases like ScienceDirect and Web of Science were searched. A total of 1,000 data points from peer- reviewed papers were extracted with the help of Origin2024b software.

The parameters collected include condenser length (Lc), evaporator length (Le), outer diameter (Do), inner diameter (Di), inclination angle (IA), heat input (Q), filling ratio (FR), and number of turns (N). Thermal resistance (R) was taken as the output to be predicted.

Twelve machine learning and statistical models were selected from past studies: ANFIS, ANN, Bayesian Ridge, LGBM, XGB, GBR, Kernel Ridge, Linear Regression, SGD, RBF-NN, RF, and SVM. Each was chosen for its unique strengths in handling nonlinearity, high-dimensional data, and varying experimental conditions – supporting a well-rounded evaluation for HP performance prediction.

In this research, we took a closer look at these two errors primarily coefficient of correlation ( $R^2$ ) and root mean squared error (RMSE), which were used to test the performance of prediction machine-learning models built for the heat pipe thermal behaviour.  $R^2$  specifies the degree of correspondence between predicted values and actual numbers while RMSE registers the average magnitude of prediction error. Both these measures, therefore, are statistically employed in thermal systems regression modelling as they are universal and quite efficient for use. The equations for  $R^2$  and RMSE are as follows:

$$RMSE = \sqrt{\frac{\sum_{n=1}^{N} (\hat{y} - y)^{2}}{N}}$$
(1)  
$$R^{2} = \frac{\sum_{n=1}^{N} (\hat{y} - y)^{2}}{\sum_{n=1}^{N} (\hat{y} - \overline{y})^{2}}$$
(2)

Here,  $\hat{y}$  refers to predicted values, while y is the actual value of the target variable, and the mean of actual target values is given by  $\bar{y}$ . The variable n indicates the individual data point, while N refers to the total number of data points in the dataset.

#### **RESULTS AND DISCUSSION**

The given dataset was split into an 80:20 training-to-testing ratio, with 80% being utilized to train the models and 20% being kept aside for evaluating how well these models perform.

After training, these models were tested with the aforementioned error measures to identify how accurate these models are with regards to prediction.

	Training R <sup>2</sup>	Testing R <sup>2</sup>	RMSE
ANN	0.962	0.941	0.257
XGB	0.989	0.924	0.292
RF	0.986	0.891	0.351
GBR	0.957	0.881	0.366
LGBM	0.955	0.876	0.368
ANFIS	0.746	0.721	0.56
RBF-NN	0.779	0.687	0.594
Kernel Ridge Regressor	0.596	0.472	0.771
SVM	0.557	0.383	0.833
SGD	0.217	0.154	0.976
Bayesian Ridge Regressor	0.223	0.154	0.976
Linear Regressor	0.225	0.15	0.978

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On ascertaining the values of R<sup>2</sup> and RMSE, which were calculated, it was evident that ANN model performed significantly better than all others, with improved prediction ability and generalization performance. This is due to the ability of ANN model to capture complex nonlinear relationships of the datasets. This is also the reason why linear regressor performed the least.



**Figure 1.** Model prediction (a) ANFIS, (b) ANN, (c) Bayesian Ridge, (d)GBR, (e)Kernel Ridge, (f)LGBM, (g) Linear Regressor, (h) RBF-NN, (i)RF, (j) SGD, (k) SVM, and (l) XGB

D	1	0.96	0.27	0.16	-0.28	0.059	-0.21	0.031	-0.16
D。	0.96	1	0.3	0.13	-0.28	0.073	-0.19	-0.043	-0.18
L <sub>e</sub>	0.27	0.3	1	0.62	0.12	0.19	0.11	0.27	-0.03
L <sub>c</sub>	0.16	0.13	0.62	1	0.22	0.21	0.31	0.41	-0.16
N	-0.28	-0.28	0.12	0.22	1	0.24	0.07	0.33	-0.27
IA	0.059	0.073	0.19	0.21	0.24	1	0.16	0.26	-0.068
FR	-0.21	-0.19	0.11	0.31	0.07	0.16	1	0.31	-0.075
Q	-0.031	0.043	0.27	0.41	0.33	0.26	0.31	1	-0.25
R	-0.164	-0.18	-0.03	-0.16	-0.27	-0.068	-0.075	-0.25	1
	Di	D。	L <sub>e</sub>	L <sub>c</sub>	N	IA	FR	Q	R

Figure 2. Pearson correlation matrix between the variables of the study and R

The Pearson's correlation matrix shown in figure 2 indicates that the input feature Q affects R the most. The negative value indicates that Q is inversely proportional to R, meaning when Q is increased R decreases, thus increasing thermal performance

#### CONCLUSIONS

In this research, various machine learning models have been analysed using standard error metrics like  $R^2$  and RMSE in an 80:20 split of train-test. The model ANN excelled among all the others with higher predictive accuracy and generalization capability, whereas the worst performance in prediction was exhibited by LR, which made its inability to describe the complex nonlinear patterns of the data clear. This confirms that ANN acts best for such applications that require extremely high precision and flexibility, thus giving predictive modelling in such context's supremacy over the rest in the future.

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# Studies on Antimicrobial, Anticancer Activity of Silver Nanoparticles Extracted from Pelargonium Radensplant

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# ABSTRACT

Nanotechnology has emerged as a transformative tool in medical practice, particularly for medicines, diagnostics, and disease prevention. Silver nanoparticles (AgNPs) were produced in this investigation by extracting the stem, root, and leaf of the medicinal plant Pelargonium radens. Phytochemicals such as flavonoids, alkaloids, and terpenoids aided in the photo-induced bioreduction of silver ions, resulting in the generation of pure, crystalline AgNPs ranging 40-60 nm in size. The synthesized AgNPs were characterized using UV-Visible spectrophotometry, Dynamic Light Scattering (DLS), Scanning and Transmission Electron Microscopy (SEM and TEM), Fourier Transform Infrared Spectroscopy (FTIR), Powder X-ray Diffraction (XRD), and Energy Dispersive Spectroscopy (EDS).Following detailed physicochemical characterization, and the AgNPs were tested for antimicrobial and anticancer properties. Notably, they exhibited excellent antimicrobial action, especially in leaf-derived samples, against both Grampositive (Staphylococcus aureus, Bacillus subtilis) and Gram-negative bacteria (E. coli, Pseudomonas aeruginosa). Furthermore, Annexin V-FITC and DAPI staining revealed that AgNPs caused apoptosis in MCF-7 breast cancer cells. Stem-mediated AgNPs exhibited little hemolytic activity (<5%), indicating that they are suitable for therapeutic usage. The findings of this research reveal that green-synthesized AgNPs have strong antimicrobial and anticancer activity, as well as impressive biocompatibility, indicating their potential for use in future therapies.

Keywords: Antimicrobial Activity, Anti Cancer Activity, FTIR, SEM, Pelargonium Radens, Silver Nanoparticles.

# INTRODUCTION

Nanotechnology has emerged as a transformative field with applications spanning medicine, energy, and materials science (1). Among the most promising developments are metal nanoparticles, particularly silver nanoparticles (AgNPs), due to their remarkable antimicrobial and anticancer properties (2). However, traditional methods for synthesizing nanoparticles often involve toxic chemicals and energy-intensive processes (3). To overcome this, green synthesis using biological resources has gained attention for being sustainable, eco-friendly, and cost-effective (4).

This study focuses on the synthesis of AgNPs using the root, stem, and leaf extracts of *Pelargonium radens*. The current research includes the antimicrobial, and antioxidant properties, anticancer activity of biosynthesized AgNPs, by utilizing a variety of microbial, fungal, and malignant cell line activities and to evaluate their biomedical efficacy.

#### MATERIALS AND METHODS

#### 2.1 Plant Extraction and Synthesis of AgNPs:

Fresh samples of *P. radens* were collected, and extracts were prepared from the stem, leaves, and roots using aqueous and methanolic solvents (Fig 1). These extracts acted as reducing and capping agents for the green synthesis of AgNPs upon mixing with silver nitrate (AgNO<sub>3</sub>) solution.



Fig 1. Extraction and synthesis of nanoparticles

#### 2.2 Characterization Techniques

The synthesized AgNPs of *Pelargonium radens* stem, root and leaves were characterized. The synthesized AgNps solution with *Pelargonium radens* extract (Plant, root and stem) was observed by UV-Visible spectroscopy (5). The bio-reduction compounds are responsible for the synthesis of AgNPs were determined using Fourier-transform infrared spectroscopy (FTIR), and Size and shape of synthesized AgNPs studied with Scanning Electron Microscopy (SEM) and X-ray powder diffraction (XRD)

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#### 2.3 Biological Assays

Antibacterial activity: Assessed *against E. coli*, *Pseudomonas aeruginosa*, *Staphylococcus aureus*, and *Bacillus subtilis* using zone of inhibition and MIC/MBC assays.

Antifungal activity: Tested against Candida albicans and Aspergillus niger.

All Lyophilized cultures were procured from Microbial Type Culture Collection (MTCC), Chandigarh and revived and the sub cultured cultures were used in present experiments

Antioxidant activity: DPPH and H<sub>2</sub>O<sub>2</sub> scavenging assays.

Cytotoxicity and Cell Viability: MTT assay on cancer cell lines (MCF-7, HeLa, A549) and a normal human cell line (HEK-293).

Hemolysis Test: To evaluate biocompatibility.

#### 3. RESULTS AND DISCUSSION

#### 3.1 Nanoparticle Formation and Properties

AgNPs formation was confirmed by a color change and absorption peaks between 400-450 nm in UV-Vis spectra (**Fig 2**). XRD results showed sharp peaks corresponding to face-centered cubic (fcc) silver crystals (**Fig 3**). FTIR indicated the presence of biomolecules like flavonoids and terpenoids responsible for reduction and stabilization (**Fig 4**). SEM revealed nanoparticles sized 40–60 nm, with mostly spherical morphology (**Fig 5**).



Fig 2. UV-Visible absorption spectra of *P. radens* AgNPs



Fig 3. XRD Spectra of P. radens AgNPs







Fig 5. SEM image of *P. radens* AgNPs

#### 3.2 Antimicrobial Activity

Leaf-mediated AgNPs demonstrated superior antibacterial activity compared to stem and root extracts. Gram-negative bacteria (*E. coli, Pseudomonas*) were more susceptible (**Fig 6**), suggesting membrane damage and oxidative stress as mechanisms. Antifungal activity was negligible in all cases (**Fig 7**).



Fig 6. Anti-Bacterial Activity of *P. radens* AgNPs on gram positive cultures.



Fig 7. Anti-Fungal Activity of Aspergillus niger on Pelargonium radens AgNPs

A: Candida albicans B: Aspergillus niger

#### 3.3 Antioxidant Potential

All extracts and their corresponding AgNPs displayed considerable free radical scavenging activity. Leaf AgNPs showed the highest DPPH and  $H_2O_2$  scavenging efficiency, confirming their antioxidative strength.

## 3.4 Cytotoxicity and Biocompatibility

AgNPs exhibited significant dose-dependent cytotoxic effects on MCF-7 (breast), HeLa (cervical), and A549 (lung) cancer cell lines. The most effective was the leaf extract-derived AgNPs, inducing apoptosis confirmed by DAPI staining and morphological changes. Normal cell line (HEK-293) remained largely unaffected, indicating selective cytotoxicity. Hemolysis results showed <5% activity, validating safe therapeutic potential (Fig 8).



Fig 8. The control's DAPI staining and treated cells of MCF-7

#### (A) Control (B) AgNPs treated cells.

**Conclusions:** The green synthesis of AgNPs using *Pelargonium radens* offers an efficient, non-toxic, and sustainable route to obtain bioactive nanoparticles. Among the different parts, leaf extract-mediated AgNPs showed the highest antimicrobial, antioxidant, and anticancer potential. The selective toxicity toward cancer cells and negligible hemolysis highlight their potential in therapeutic applications. Further research can focus on in vivo testing, detailed mechanistic studies, and formulation into antimicrobial or anticancer agents. Green nanotechnology presents a promising future in biomedical sciences with minimal environmental impact..

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# Exploring The Electrogenic Potential of Symbionts for the Sustainable Generation of Green Power

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# ABSTRACT

In our contemporary world, energy demand is incessant, driving us to seek sustainable sources that can ensure a harmonious coexistence with our environment. Microorganisms are one of the most abundant renewable sources of energy when their metabolism is harnessed by Microbial Fuel Cells (MFCs). The microbes in the MFC release electrons upon substrate utilization, and their movement from the anode to the cathode constitutes electricity. The core objective of this study is to develop a low-cost MFC that uses a combination of Microalgal culture and industrial Dairy Effluent (DE) as the Catholyte and compare its electrogenic potential with that of a control and a microalgal catholyte. At the end of the study, it was concluded that the Microalgae-DE combination elicited about 14% more voltage than the pure microalgal catholyte, indicating its electrogenic superiority. The symbiotic bacteria in the DE that may be responsible for this voltage increase were determined by Next-Generation Sequencing, and the dominant genus of bacteria was found to be Pseudomonas. The reduction percentage of TDS, TSS, BOD and COD in the DE was about 99.27%, 90%, 77.14% and 74.28% respectively, due to phyco-remediation. Analysis of the Anaerobic Sludge used in the anodic chamber also revealed reductions in heavy metals such as Ba, Ni, and Zn with Zn having the highest reduction percentage of 99.73% as well as a significant Phosphorous reduction percentage of 99.98%.

Keywords: Green Power, Microbial Fuel Cell, Dairy Effluent, Symbionts, Microalgae, Bacteria, Bioremediation

# 1 INTRODUCTION

It is a well-known fact that our world is driven by fossil fuel-derived energy. Global estimates show that the existing reserves of these fuels will last for about 50 years (oil), 53 years (Natural gas), and 114 years (coal) (Kirsch, 2020). The prevailing sources of energy predominantly consist of non-renewable resources, which have led to their rapid depletion due to extensive utilization. Climate Change due to accelerated Global Warming is one of the direct consequences of the continued use of carbon-based fuel. The only hope for meeting the energy requirements of the world while tackling Climate Change is to expand the research in green energy production. A lucrative source of green energy that's gaining

popularity as a renewable clean source of electricity is the metabolism of microorganisms. Microbial fuel cells (MFCs) are gaining attention due to their multi-benefit nature of renewable energy production and sustainable effluent treatment. Our Model, the Microalgal Microbial Fuel Cells (MMFCs) majorly involve bacteria in the anode chamber, and microalgal-bacterial symbionts in the cathode chamber. The effluent is used as a feed and the impact of the symbiosis between the microalgae and the bacteria on electricity is studied.

Dairy Effluent continues to be a highly potential, but unexplored source of energy. India is recognized as the largest milk producing country in the world. About 6 to 10 litres of Dairy Effluent (DE) is released per litre of milk produced in India, on an average (Ahmed, 2017) which is filled with remnants of milk fat, easily digestible polysaccharides and lipids, along with proteins like Nitrates, Nitrites and Ammonium. The milk components and the chemical cleansers contribute to high levels of Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD) (Rakesh Mehrotra, 2016), Total Dissolved Solids (TDS) and Total Suspended Solids (TSS), all of which are above the permissible level (Noorjahan & Dawood Sharief, 2004).

The harmful effects of DE can be overcome by remediating and repurposing it using Microalgae that effectively bioremediate effluents by absorbing Nitrogen, Phosphorus and Potassium (Nachiappan & Chandrasekaran, 2023). Also, the bacteria present in the Dairy Effluent have been suggested to symbiotically aid microalgal growth and development, leading to enhanced oxygen production. This is postulated to elevate the electrical output of the MFC by attracting a greater number of electrons for oxygenation. The Electrical connectivity of many such MFCs will constitute a current of electrons that can be harnessed. Therefore, the utilization of microbes found in waste byproducts from dairy industries holds the potential to generate valuable electricity through sustainably engineered MMFCs.

#### MATERIALS AND METHODS

The liquid microalgal cultures were inoculated in BG-11 medium at 1:2 ratio, at neutral pH. An 18-hour photoperiod was maintained, followed by 6 hours of darkness each day with frequent aeration. The Microalgal strains of D, C and S were developed for 15-20 days before subculturing or doubling the volume to ensure stability for a period of 2 months before usage in the MMFC set-ups.

A PVC pipe with a diameter of 2.54 cm and length of 6 cm is sterilized using 70% IPA. After it dries, one end is sealed and filled with a 3% agar solution, taking care to avoid the formation of air bubbles. Once the pipe is filled, the open end is sealed and the agar bridge is allowed to solidify. Upon solidification, the seals are removed and the entire agar bridge is carefully immersed in the 0.1% KCl solution, where it is kept for 1-2 days under sterile conditions before being attached to the MMFC chambers.

Two identical plastic containers with air-tight lids, each with a volume of 1 litre, are each provided with a hole of 2.5 cm diameter at about 3.5 cm from the base. The agar salt bridge is attached with joints and secured with bolts, washers and additional Teflon threading, connecting the two chambers.

A graphite electrode measuring 1.6 x 0.6 cm in the anode chamber is connected to a zinc electrode measuring 8.5 x 2.3 cm using a 14 cm long Cu wire (Wei et al., 2011) (Al-Mustasin et al., 2022). These are then connected to the multimeter. A small inlet for aeration is made on the lid of the cathodic chamber, which is partially covered with cellophane tape.

75% of the anodic chamber is filled with sludge collected from the wastewater treatment plant, ensuring that it contacts the salt bridge. Then, 1 ml of a 0.1M sugar solution is injected into the anaerobic sludge. The cathodic chamber is filled with 1000 ml of waterfor set-up A, 1000 ml of an established microalgal consortium for set-up B, and a mixture of 800 ml of the established microalgal consortium and 200 ml of industrial dairy effluent for set-up C, ensuring the electrodes are completely submerged. All openings on the anodic chamber are sealed to establish an anaerobic environment. The set-up is kept undisturbed until a voltage is generated, with the voltage measured daily and compared across the three set-ups.

The BOD, COD, TSS and TDS of catholyte, and element content of anolyte were measured by standardized methods at the start and the end of one month of MMFC operation, and the catholyte was outsourced for NGS analysis at the end of the study.

# **RESULTS AND DISCUSSION**

The Voltage generated by the three set-ups of MFCs, A, B and C, were monitored and recorded using a standard multi-meter, and plotted as a line graph for comparative purposes. Set-Up C produced the highest values voltage throughout the study period, compared to theother two set-ups. Setup A showed a reading of 0, but it constantly fluctuated between a negative and a positive value, as indicated by the negative sign on the multimeter, despite the reading being 0. This might be due to the quantity of electricity being below the Detectable Limit of the multi-meter, signifying the importance of microalgae in increasing the electrical output. Both set-up B and set-up C produced their maximum Voltages of 0.307 V and 0.351 V on day 18.

The trend followed by the above MMFCs is comparable with the MFC with unaltered anolyte used in a similar study by (Thenmozhi M et al., 2020), but twice the value obtained in the latter. BOD before the 30-day period was 116.67 mg/l and after, it was reduced to 26.667 mg/l. The COD was 198.339 mg/l before and 50.405 mg/l after the 30-day period.

It can be observed that the reduction percentage of BOD which is 77.14%, is more than that of COD which is 74.28%. This is because of the involvement of microalgae, which are basically biological entities, in the reduction process (*Nachiappan & Chandrasekaran*, 2023). When combined with bacteria, the reduction of BOD and COD takes place due to heterotrophic activity when the oxygen level is more and by nitrifying activity when the oxygen content is less (Fallahi et al., 2021).

TDS underwent a more significant reduction of 99.268% compared to TSS 90%.

This is because Microalgae easily degrade suspended solids through the oxygen they release upon photosynthesis (Geremia et al., 2021). They also bioaccumulate the solids content and metabolize them, which contributes to further decline in TSS, as has been observed above.

According to (Zhang et al., 2017) Total Dissolved Solids (TDS) are composed of inorganic salts such as Sulphates, Bicarbonates, Sodium, Potassium, Magnesium, Calcium and Chlorides. Through biosorption at high pH, Microalgae have also been known to remove ions of sodium potassium, calcium and magnesium, contributing yet again, to TDS reduction. Such biosorption takes place due to the presence of negatively charged groups on the surface of the microalgal cells (*Ayangbenro & Babalola, 2017*). All these factors together have contributed to a significant reduction in TDS.

NGS Analysis was performed for the DE sample before and after being used in MMFC set-up C, to determine the genera of bacteria present in the DE. The relative abundance of the bacteria is used to identify those bacteria that have a larger population compared to other bacteria in a given sample. The changes in relative abundance are not reflective of those in the absolute abundance of the bacterial species. Comparing the Relative Abundance of the two samples, is to basically understand how the bacterial communities in the Dairy effluent have responded to the microalgae with which they have resided for the duration of their study.

In Figure 1, White\_S73 refers to the raw Dairy Effluent before usage in MFC and Green\_S72 represents the catholyte of the MMFC. Most of the genera that have higher relative abundances belong to the Phylum of Proteobacteria. Among these, genera with mutualistic relations with microalgae are Gammaproteobacteria with orders of the orders of Pseudomonadales and Xanthomonadales.



Figure 1 NGS data of Most Abundant Phyla, Genera and Class of bacteria in DE



Figure 2 Bacterial Class Data Obtained After NGS

Proteobacteria is the most abundant Phylum of bacteria in the catholyte and in the raw DE, and *Pseudomonas,* a genus of gram-negative bacteria belonging to the family of *Pseudomonadaceae,* is a common food-borne pathogen that causes opportunisticinfections in humans (*Diggle & Whiteley, 2020*), common in dairy effluent. However, when cocultured with *Chlorella vulgaris,* it has been found to symbiotically stimulate the latter's growth (*Guo & Tong, 2014*). *Acinetobacter,* under the family of *Moraxellaceae,* has also been found to have astrong positive interaction with *Chlorella* (Wirth et al., 2020). In a study carried out by (Xue et al., 2018) it was proven that *Stenotrophomonas,* a genus belonging to the phytopathogenic family of *Xanthomonadaceae* greatly increased the specific growth rate and biomass of *Chlorella,* enhancing its productivity by about 18%.

Betaproteobacteria having members such as *Acidovorax* and *Hydrogenophaga* are also mutualistic with microalgae. *Acidovorax*, a plant pathogen, was found to produce the maximum increase of 24.8%, in the biomass of *Scnedesmus dimorphus*, when cocultured with the latter (Wang et al., 2015). Mutualistic associations have also been found between *Hydrogenophaga*, *Scenedesmus* (Lee et al., 2019) and *Chlorella*. *Hydrogenophaga* are autotrophic and utilize oxygen as their final electron acceptor. They form a biofilm, which when present on the cathode, actively promote the reduction of oxygen (Song et al., 2020), hence contributing to the efficiency of the catholyte in increasing the electrical output of the MMFC. Thus, its presence in the DE is greatly advantageous.

Although Gammaproteobacteria have a lower relative abundance compared to betaproteobacteria in the catholyte, it does not necessarily mean that their absolute abundance has declined. The wide variation in the strains of betaproteobacteria that positively responded to the microalgae may be the reasonfor the difference in relative abundances of the two classes. However, more genera of bacteria belonging to the class of gammaproteobacterialwere detected, compared to betaproteobacteria. This might be because, the different genera of betaproteobacteria might have populations below thedetectable limit of the NGS method used and thus might have been classified under the category of 'Other'. This corroborates the comparatively higher relativeabundance possessed by 'Other'.

In the Anolyte sludge, Cadmium, Selenium and Molybdenum were observed to have decreased below the Minimum Level of Quantification, which is 0.1mg/kg.



Figure 3 (a) Comparison between TSS and TDS (b) Element Reduction Percentages in Anolyte (c) BOD and COD Comparison (d) Voltage Generation Comparison

#### CONCLUSION

Thus, the low-cost MMFC developed by us using the novel catholyte produced superior levels of voltage compared to the control set-up (set-up A) and the common MMFC set-up (set-up B) and Set-up B and C were active throughout the study period of 31 days. The Dairy Effluent used in our MMFC was successfully bioremediated and the reduction percentages of TDS, TSS, BOD and COD were observed to be 99.268%, 90%, 77.14%, and 74.28%.

The major phylum of bacteria detected in the samples was Proteobacteria, with *Pseudomonas* as the dominant genus. The bacteria detected in the catholyte collected on Day 31 proved that symbiotic, mutualistic factors had played a part in increasing the oxygen production by microalgae, and consequently the electrical output the MMFC. Thus, the catholyte is worthy of further exploration for advanced research.

The microbial activity in the Anodic Chamber also showed reductions in the levels of certain inorganic elements in the anaerobic sludge such as Ba, Ni, Zn and P. Thus, the low-cost MMFC developed successfully enabled the potential assessment of the intended catholyte.

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# Low-Temperature Mechanochemical Synthesis of the CaMg<sub>2</sub> Intermetallic Compound

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# ABSTRACT

Growing emphasis on sustainable material synthesis has driven the development of eco-friendly mechanochemical methods for producing intermetallic compounds (IMCs). This study demonstrates the low-temperature mechanochemical synthesis of Ca-Mg binary IMCs, a promising approach for biomedical applications with minimal environmental impact. Using unpurified magnesium (Mg) powder and granular calcium (Ca), ethanol (0.5 wt%) was introduced as a process control agent (PCA) to mitigate cold welding during high-energy ball milling at room temperature. A design of experiments (DOE) methodology was employed, varying Mg/Ca ratios (1:1–3:1), ball-to-powder ratios (5:1–20:1), and milling times (1–5 h) in a 3<sup>3</sup> factorial design. Phase composition and structural evolution were analyzed via X-ray diffraction (XRD), with Rietveld refinement (Profex software) quantifying lattice parameters and phase fractions. Complementary predictions from an open-source computational tool identified stable Ca-Mg phases, supporting a proposed reaction mechanism for CaMg formation. This work demonstrates a green, scalable route to Ca-Mg IMCs, combining experimental optimization with computational validation.

*Keywords:* Intermetallic Compounds; Mechanochemical Synthesis; Green Chemistry; Structural Characterization; Process Optimization.

## Green Process Design and Sustainable Chemical Manufacturing (TIChSCON'25)

#### INTRODUCTION

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The increasing demand for green technologies has driven significant interest in green chemistry, particularly in the synthesis of intermetallic compounds (IMCs). The IMCs possess excellent mechanical properties, corrosion resistance, and bioactivity, which render them highly promising candidates for orthopedic and dental implants [1]. However, conventional IMC preparation methods are typically based on high temperature and hazardous chemicals, which are environmentally and safety hazardous [2].

Consequently, green chemistry principles have driven the development of low-energy, eco-friendly synthesis routes that minimize toxic byproducts and maintain material properties [3]. Among them, mechanochemical synthesis – a bottom-up process that entails mechanical force-induced reactions – has emerged as a scalable and sustainable method for producing nanostructured materials [4]. As promising as mechanochemical synthesis is, however, it comes with its setbacks such as milling media contamination (container and ball wear) and exposure to air, which tends to lower the purity of the phases and quality of IMCs [5].

The present study addresses these problems by exploring Ca-Mg IMCs synthesis under sealed conditions to avoid contamination. The processing parameters including milling time, ball-to-powder ratio (BPR) and composition were optimized and ethanol was used as PCA to prevent agglomeration. Thermodynamic data and the Materials Project database [6] were also used in conjunction with XRD analysis to probe reaction mechanisms. While this study investigated the low-temperature formation of IMCs in the proposed system, further research is required to assess their suitability for medical applications.

#### MATERIALS AND METHODS

High-purity Mg powder and granular Ca, sourced from BT SCIENCE (Malaysia), were used without further purification. To mitigate cold welding and particle agglomeration during milling, ethanol (0.5 wt% of the total powder charge) was added as a PCA. A stoichiometric mixture of Mg and Ca was subjected to high-energy ball milling using a PM 400 planetary ball mill (Retsch, Germany) at room temperature. Key process parameters – composition (Mg/Ca ratio), BPR, and milling time – were optimized using a DOE approach (3<sup>3</sup> full factorial design) to enhance synthesis efficiency. This resulted in 27 experimental runs (Table 1). A 50 mL steel jar and three Ø 20 mm steel balls (total mass: 100.5 g) were used for all trials. Powder masses were adjusted based on BPR and composition, with scaling factors of 0.5115 (Mg) and 0.9051 (Ca) applied to maintain consistency.

Exp.	Composition (Mg/Ca)	BPR	Milling Time (h)	Mg (g)	Ca (g)
1	1:1	5:1	1	5.12	9.05
2	1:1	5:1	3	2.56	4.53
3	1:1	5:1	5	1.02	1.81
4	1:1	10:1	1	5.12	9.05
5	1:1	10:1	3	2.56	4.53
6	1:1	10:1	5	1.02	1.81
7	1:1	20:1	1	5.12	9.05
8	1:1	20:1	3	2.56	4.53
9	1:1	20:1	5	1.02	1.81

Table 1. DOE matrix for Ca-Mg IMC synthesis.

10	2:1	5:1	1	6.82	6.04
11	2:1	5:1	3	3.42	3.02
12	2:1	5:1	5	1.37	1.20
13	2:1	10:1	1	6.82	6.04
14	2:1	10:1	3	3.42	3.02
15	2:1	10:1	5	1.37	1.20
16	2:1	20:1	1	6.82	6.04
17	2:1	20:1	3	3.42	3.02
18	2:1	20:1	5	1.37	1.20
19	3:1	5:1	1	7.67	4.53
20	3:1	5:1	3	3.83	2.26
21	3:1	5:1	5	1.54	0.91
22	3:1	10:1	1	7.67	4.53
23	3:1	10:1	3	3.83	2.26
24	3:1	10:1	5	1.54	0.91
25	3:1	20:1	1	7.67	4.53
26	3:1	20:1	3	3.83	2.26
27	3:1	20:1	5	1.54	0.91

Phase composition was analyzed using a Rigaku MiniFlex 600 X-ray diffractometer (*Cu-Ka* radiation,  $\lambda = 1.54056$  Å) with a scan rate of 6° min<sup>-1</sup> across  $2\theta = 10^{\circ}$ -90°. Rietveld refinement applied to determine unit cell dimensions, crystallite size, and atomic site occupancies, while 3D crystal visualization was performed following Momma and Izumi's method [7]. Reaction mechanisms were evaluated using the Materials Project database [6] to compute interface-normalized reaction energies and predict stable Ca-Mg phases.

#### **RESULTS AND DISCUSSION**

Rietveld refinement of XRD profiles (**Fig. 1**) for selected experiments revealed Ca(OH)<sub>2</sub> as the dominant phase, with detectable CaMg<sub>2</sub> IMC peaks at characteristic  $2\theta$  positions corresponding to hexagonal *P63/mmc* structure. Peak broadening indicated milling-induced microstrain and nanocrystallinity, while variable peak intensities reflected differences in crystallinity across experiments [5]. The persistence of elemental Ca in Exp. 2 suggested incomplete reaction. Despite Ca(OH)<sub>2</sub> dominance, the formation of CaMg<sub>2</sub> demonstrates the potential of mechanochemical synthesis as a green route for IMC production, particularly for biomedical applications. discussion to ensure coherence and ease of interpretation.



Fig. 1. Rietveld refinement of XRD patterns for selected samples.

The phase compositions from Rietveld refinement revealed significant variations in CaMg<sub>2</sub> formation across experiments. The highest CaMg<sub>2</sub> content (24.92 wt.%) occurred in Exp. 2, while Exp. 3 showed the lowest (4.25 wt.%). All samples contained substantial Ca(OH)<sub>2</sub> (56.7 wt.% maximum) and residual Mg, suggesting incomplete reactions. The results indicate that current milling parameters (BPR, time) require further optimization to maximize CaMg<sub>2</sub> yield and minimize byproducts. Potential improvements include extended milling times, adjusted BPR, and better atmospheric control to reduce Ca(OH)<sub>2</sub> formation [8]. The reaction energy analysis reveals two distinct sub-reactions in the Ca-Mg system during mechanochemical processing. The most energetically favorable reaction occurs at a 0.333 atomic fraction (Ca:Mg = 1:2), exhibiting the highest reactivity of -0.106 eV/atom (-10.2 kJ/mol), which corresponds to CaMg<sub>2</sub> formation. This thermodynamic preference for CaMg<sub>2</sub> over other possible compounds (e.g., CaMg<sub>149</sub> at -0.026 eV/atom) aligns well with experimental XRD observations of low-temperature CaMg<sub>2</sub>
formation. The reaction enthalpy calculations show good agreement between theoretical (-0.349 eV/ formula unit) and experimental (-0.407 eV/ formula unit) values, further supporting the mechanochemical formation pathway: Ca + 2Mg  $\rightarrow$  CaMg<sub>2</sub>.

The crystal structure analysis (**Fig. 2**) reveals a multiphase system containing  $CaMg_2$  IMC,  $Ca(OH)_2$ ,  $Mg_2$ , and Ca with distinct spatial arrangements.  $CaMg_2$  displays a HCP configuration with anisotropic bonding. Refinement of Exp. 2 data confirmed the *P63/mmc* structure of  $CaMg_2$ , with Ca atoms occupying 4*f* sites (x=0.33333, y=0.66667, z=0.43810) and Mg atoms distributed between 6*h* (x=0.83200, y=0.66400, z=0.25000) and 2*a* (origin) positions. All sites showed full occupancy (1.000) and minimal atomic displacement, indicating a highly ordered arrangement within the crystal lattice.



**Fig. 2.** The standard orientation of the crystal shape of a multiphase structure comprising CaMg<sub>2</sub> IMC, Ca(OH)<sub>2</sub>, Mg, and Ca.

# CONCLUSION

This study successfully developed an eco-friendly mechanochemical synthesis route for Ca-Mg IMCs using a 3<sup>3</sup> factorial design to optimize composition (Mg/Ca ratio), BPR, and milling time. XRD/Rietveld analysis confirmed hexagonal CaMg<sub>2</sub> formation (*P63/mmc*), with yields ranging from 4.25 wt.% (Exp. 3) to 24.92 wt.% (Exp. 2), where ethanol (0.5 wt.%) effectively minimized cold welding. While Exp. 2 showed optimal results (*a* = 6.21870 Å, *c* = 10.00630 Å, *V* = 335.1223 Å<sup>3</sup>), further optimization of milling parameters is needed to maximize CaMg<sub>2</sub> purity by reducing Ca(OH)<sub>2</sub> byproducts. These findings highlight mechanochemistry as a sustainable approach for biomedical-grade IMC synthesis, with future work focusing on prolonged milling and atmospheric control to enhance yields.

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# **Biochar-Based Sorbents for Separating Heavy** Metals from Waste Water

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# ABSTRACT

Heavy metals (HMs) in wastewater present significant risks to both human health and the environment, demanding effective and affordable treatment solutions. Adsorption has become a promising method for removing these pollutants, as heavy metals are challenging to biodegrade. Pyrolysis is a process that decomposes organic materials at high temperatures without oxygen, producing biochar, bio-oil, and syngas. , Nandha Engineering College(Autonomous) However, its adsorption efficiency is lower than activated carbon, limiting its widespread application. Additionally, the heavy metals retained by biochar are difficult to release, making it hazardous if not disposed of correctly. To address these limitations, surface modifications are necessary to enhance biochar adsorption capacity and sustainability. Research has demonstrated that biochar can effectively remove heavy metals like lead, cadmium, copper, and arsenic through physical adsorption, electrostatic interactions, and chelation with functional groups on its surface. Furthermore, biochar can be tailored to improve its removal efficiency for specific contaminants. Biochar can remove up to 90% of lead, 85% of cadmium, 80% of copper, and 95% of arsenic, depending on how it's modified and its surface properties. By using waste biomass and plastics as feedstocks, biochar aligns with circular economy principles, offering a sustainable approach that reduces water toxicity while recycling waste. Studies also indicate that biochar outperforms plastic-derived chars in heavy metal removal, highlighting its potential as an effective, eco-friendly solution for wastewater treatment.

Keywords: Biochar, Pyrolysis, Heavy Metals Removal, Adsorption.

#### **INTRODUCTION**

The increasing contamination of water resources by heavy metals is a pressing environmental concern globally. These metals, including lead (Pb), mercury (Hg), cadmium (Cd), arsenic (As), and chromium (Cr), pose significant risks to both human health and ecosystems. Industrialization, mining activities, and urbanization have resulted in the discharge of untreated or poorly treated wastewater containing high concentrations of these hazardous substances into water bodies. Consequently, the removal of heavy metals from wastewater has become an urgent issue in environmental engineering and water treatment practices. Traditional water treatment methods, such as chemical precipitation, ion exchange, and adsorption, have been employed to mitigate the harmful effects of heavy metal contamination. However,

these methods often face challenges such as high operational costs, limited efficiency, and the production of toxic sludge, which poses additional environmental risks.

In recent years, biochar-based sorbents have emerged as a promising and sustainable alternative for the removal of heavy metals from contaminated wastewater. Biochar, a carbon-rich material produced through the pyrolysis of organic biomass, is gaining recognition as an effective, low-cost, and environmentally friendly material for water purification. Its unique properties, including high surface area, large pore volume, and rich functional groups, make biochar an excellent candidate for adsorption-based removal of heavy metals from wastewater.

This introduction explores the significance of biochar-based sorbents in heavy metal removal, highlighting their advantages, mechanisms of interaction with contaminants, and the challenges associated with their use.

Heavy metal contamination of water sources is a growing problem due to various anthropogenic activities. These metals are toxic even at low concentrations and can accumulate in the food chain, leading to serious health issues such as neurological disorders, organ damage, cancer, and reproductive problems. Water pollution caused by industrial effluents, agricultural runoff, mining waste, and improper disposal of electronic waste is one of the primary causes of heavy metal contamination in surface water and groundwater. Given the persistence and mobility of these metals in the environment, the need for effective treatment methods to remove them from wastewater has become more critical.

Conventional methods for removing heavy metals, such as chemical precipitation, membrane filtration, and ion exchange, are widely used but often face limitations. For example, chemical precipitation often requires the addition of large quantities of chemicals, which can lead to the generation of secondary waste and increased treatment costs. Membrane filtration methods, while effective, are energy-intensive and can suffer from membrane fouling. Ion exchange resins, though efficient, can be costly and are limited in their regeneration capacity. These challenges underscore the need for alternative, sustainable, and cost-effective solutions, leading to the growing interest in biochar-based sorbents.

Biochar is a porous, carbon-rich material that is produced by the pyrolysis of organic materials, such as agricultural waste, forestry residues, and biomass. The pyrolysis process involves heating the biomass in a low-oxygen environment, resulting in the conversion of organic matter into a stable form of carbon that retains a large surface area and porous structure. The high surface area of biochar, combined with its functional groups such as hydroxyl, carboxyl, and phenolic groups, makes it highly reactive and capable of interacting with various pollutants, including heavy metals.

#### MATERIALS AND METHODS

Biochar was produced via pyrolysis of agricultural biomass – specifically rice husks and corn stalks – at 500°C under limited oxygen using a muffle furnace. The resulting biochar was ground and sieved to a particle size <2 mm. Prior to use, biochar was washed with deionized water and dried at 105°C for 24 hours. Surface modification was performed using 1 M NaOH or 0.1 M HNO<sub>3</sub> to enhance sorption properties.

Simulated wastewater containing known concentrations (50–200 mg/L) of heavy metals – namely lead (Pb<sup>2+</sup>), cadmium (Cd<sup>2+</sup>), and copper (Cu<sup>2+</sup>) – was prepared using analytical-grade salts. Batch adsorption experiments were carried out by mixing 1 g of biochar with 100 mL of metal solution in 250 mL Erlenmeyer flasks. The mixtures were agitated at 150 rpm for 24 hours at room temperature.

After filtration through Whatman No. 42 filter paper, metal concentrations in the filtrate were analyzed using atomic absorption spectroscopy (AAS). Parameters such as pH (4–8), contact time (0–48 h), and initial metal concentration were varied to assess their effects on adsorption efficiency. All experiments were conducted in triplicate, and data were analyzed using ANOVA and Langmuir and Freundlich isotherm models to interpret sorption behavior.

- Food Waste or Agricultural Waste
- > Activating Agent as Potassium Hydroxide (KOH)
- Functionalizing Agent as Citric Acid
- > Other Materials for Composite Sorbents like Activated Carbon
- Actual Wastewater

### **RESULTS AND DISCUSSION**

The Kabini River in Karnataka faces significant heavy metal contamination (Cr, Cu, Fe, Mn, Ni, Pb, Zn) from industrial effluents, agricultural runoff, and sewage, necessitating urgent remediation and stricter environmental regulations to mitigate ecological risks and protect water quality [1]. Anthropogenic activities have altered global biogeochemical cycles, with heavy metals contaminating plants via foliar uptake, posing ecological and health risks. Comprehensive studies on uptake mechanisms, risk assessments, and preventive measures in urban agriculture are essential for sustainable food security and environmental health [2]. In the past, various remediation methods/techniques have been used to immobilize/remove metal from water including ion-exchange, electrodialysis, chemical precipitation, membrane filtration and electrochemical treatment[3]. Among these techniques, biosorption has emerged as a viable and cost-effective method for removal of heavy metals from water[4]. Biosorbents are eco-friendly and easily available materials and among them, biochar is considered as the most popular one. Recently, biochar is being considered as a main alternative agent than other biomaterials for the removal of toxic metals from water [5].Biochar, derived from organic waste, holds significant potential for environmental sustainability through applications in soil remediation, carbon sequestration, and wastewater treatment; however, further research and in situ studies are needed to optimize its properties, mechanisms, and large-scale environmental impact [6]. An experiment on potatoes irrigated with untreated wastewater demonstrated that soil amendment with plantain peel biochar significantly reduced Cd and Zn uptake in tuber flesh and peel, mitigating potential health risks. The study highlights that while heavy metals accumulated in all plant parts, the peel posed a higher health risk than the flesh, with biochar effectively decreasing contamination [7]. This review explores various methods for heavy metal remediation in contaminated soil, emphasizing the use of biochar composites, which effectively remove metals through mechanisms such as sorption, precipitation, complexation, and ion exchange. While physical, chemical, biological, and phytoremediation methods each offer advantages and limitations, biochar composites, especially when tailored with specific non-metals or doped metals, present a promising, cost-effective solution for enhancing heavy metal removal efficiency [8]. This review highlights recent advancements in biochar pyrolysis and modification techniques, including microwave-assisted pyrolysis, co-pyrolysis, and various green modification strategies, aimed at enhancing biochar's environmental remediation capabilities. By improving biochar's physicochemical properties and addressing sustainability concerns, these novel approaches offer promising solutions for contaminant adsorption, soil fertility enhancement, and climate change mitigation [9]. This analysis evaluates the profitability of producing biochar from corn stover through slow and fast pyrolysis, considering both carbon offset credits and energy product revenues. The fast pyrolysis scenario, which co-produces bio-oil and biochar, shows stronger economic potential, with projected internal rates of return (IRR) ranging from 15% to 37%, while slow pyrolysis, primarily focused on biochar, is less profitable due to lower carbon offset values and higher feedstock costs [10].

#### CONCLUSION

The document provides a comprehensive review of biochar-based sorbents as an eco-friendly, sustainable, and cost-effective method for removing heavy metals from contaminated water. Biochar is derived from the pyrolysis of biomass under oxygen-limited conditions, resulting in a carbon-rich material with high porosity, large surface area, and abundant functional groups that enable effective sorption of toxic metals such as lead, cadmium, and mercury.

The effectiveness of biochar depends on factors like feedstock type, pyrolysis temperature, and modifications. Enhancement techniques, including chemical activation, mineral impregnation, and integration with materials like graphene oxide, significantly improve its sorption capacity. These processes create more active sites and improve functional properties, allowing biochar to capture metals through mechanisms like physical adsorption, ion exchange, surface complexation, and precipitation.

Biochar's use aligns with sustainability goals by converting agricultural and industrial waste into valuable sorbents. Its environmental benefits include waste valorization, soil improvement, and carbon sequestration, addressing pollution while supporting resource efficiency. Despite its potential, challenges remain in scaling production, optimizing modifications, and understanding long-term impacts on ecosystems.

The review underscores the need for further research to develop biochar as a filtration medium and improve its economic and environmental viability in large-scale applications. By advancing biochar technologies, it can become a key solution in tackling global water pollution challenges, especially in areas affected by heavy metal contamination.

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# **CFD Analysis of Vortex Finder Wall Thickness on Short-Circuit Flow Reduction in Hydrocyclones**

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# ABSTRACT

A hydrocyclone is a equipment used to separate particles from a liquid by applying centrifugal force, typically for solid-liquid separation. The performance of a hydrocyclone is significantly influenced by its design, with particular emphasis on the vortex finder wall thickness, which affects the short circuit flow (SCF). This study investigates the impact of varying vortex finder wall thicknesses on SCF in a 250 mm hydrocyclone using Computational Fluid Dynamics (CFD). A study was conducted on four distinct geometries, including a standard configuration with a vortex finder thickness of 1.75 mm and three modified designs with increased thicknesses of 10 mm, 22.5 mm, and 35 *mm.* All geometries were created and meshed using ICEM-CFD, resulting in a structured mesh with 200K elements. Ansys Fluent was used to solve the CFD simulations. Initially, simulations were performed under laminar flow conditions, followed by the application of the K- $\Box$  RNG and Reynolds Stress Models (RSM) for turbulence. After achieving a steady pressure profile, the Volume of Fluid (VOF) method was applied to simulate the air core. SCF was evaluated by analysing axial velocity distributions, and the hydrocyclone's performance was further assessed by pressure drop and velocity profiles. The results indicate that increasing the vortex finder wall thickness leads to a reduction in short circuit flow, aligning with trends observed in mini-hydrocyclone. The findings provide valuable insights into the optimization of hydrocyclone design, particularly in industries where efficient separation and minimal short-circuiting are critical. This study enhances the understanding of the impact of vortex finder thickness on hydrocyclone performance, offering valuable insights for applications in mining, oil refining, and wastewater treatment.

Keywords: Hydrocyclone, Vortex Finder Thickness, Short-Circuit Flow, CFD, RSM

#### INTRODUCTION

For over a century, hydrocyclones have been extensively utilized in environmental, mineral processing, and petrochemical industries for solid-liquid separation (Schwerzler, 2005). When a suspension is introduced tangentially into the hydrocyclone, the resulting rotational motion of the fluid generates a centrifugal force that acts on the suspended particles. As a result, larger and denser particles are driven toward the outer wall and discharged through the underflow, while finer particles are carried by the secondary vortex and exit through the overflow. The continued relevance of hydrocyclones in both industrial practice and academic research is primarily attributed to their simple design, low capital investment, and minimal operating and maintenance requirements (Hwang & Chou, 2017).

Despite these benefits, the complex multiphase flow inside the hydrocyclone, which includes intense swirling motion and undesired short-circuit flow often makes it difficult to achieve high separation efficiency (Hsieh & Rajamani, 1991). Previous studies have thoroughly examined the effects of design parameters on performance, including inlet geometry, vortex finder diameter, and cone angle (Narasimha et al., 2007). The influence of vortex finder thickness has received limited attention in previous studies. This gap is important as it may play a key role in optimizing design and minimizing short-circuit flow. This study uses computational fluid dynamics (CFD) to investigate the hydrodynamic effects of varying vortex finder thickness. In recent decades, computational fluid dynamics (CFD), supported by experimental data, has greatly advanced the numerical simulation of hydrocyclones. (Cui et al., 2015). This study aims to better understand how this parameter affects internal flow patterns. It focuses on specific operating conditions and does not consider chemical or thermal interactions. However, the results provide a starting point for further research and experimental validation.

#### MATERIALS AND METHODS

#### Model description

To depict the complex multiphase flow and swirling behaviour inside the hydrocyclone, a multiphase CFD approach was adopted using the VOF model in ANSYS Fluent (Zhao et al., 2019). In multiphase models, the velocity field for incompressible fluids (air and water) is calculated from the continuity and momentum equations:

$$\frac{\partial(\rho_{m})}{\partial t} + \frac{\partial(\rho_{m}u_{m})}{\partial x} = 0$$

$$\frac{\partial(\rho_{m}u_{mi}u_{mj})}{\partial t} + \frac{\partial(\rho_{m}u_{mi})}{\partial t} = -\frac{\partial\rho}{\partial x_{i}} + \qquad(1)$$

$$\frac{\partial}{\partial x_{j}} \Big[ \mu_{m} \left( \frac{\partial u_{mi}}{\partial x_{j}} + \frac{\partial u_{mj}}{\partial x_{i}} \right) \Big] +$$

$$\frac{\partial}{\partial x_{j}} (-\rho_{m} \overline{u'_{mi}u'_{mj}}) + F + \frac{\partial(\sum_{k=1}^{n} \alpha_{k} \rho_{k} \mu_{drki} \mu_{drkj})}{\partial x_{j}} + \qquad(2)$$

 $ho_{
m m}g$ 

Where *m* represents the mixture phase, F is the body force, n is the number of phases, *ak* is the volume fraction of phase *k*, *udr*,*ki* is the drift velocity for phase k and is only available in the mixture model, and is the Reynolds stress term. Due to the anisotropy of the Reynolds stress in hydrocyclones, the Reynolds stress model (RSM) is adopted for both accuracy and computational efficiency. Air core is the most important characteristic of the flow field in a hydrocyclone. It can be tracked by solving the following equation:

$$\frac{\partial \alpha_k}{\partial t} + u_i \frac{\partial \alpha_k}{\partial x_i} = 0 \tag{3}$$

VOF model is adopted in this paper. After running the model for approximately 15s, the characteristics of stable flow field are obtained. The hydrocyclone used in this study is shown in Figure 1.



Figure 1. Geometric parameters of hydrocyclone

In this work, the computational domain is divided into hexahedral mesh elements mainly using the O-grid block split method. The boundary condition of the inlet is set as a velocity inlet with a constant velocity of 2.5 m/s. The pressure outlet condition is used at both overflow and underflow, where the absolute pressure is taken as 1 atm.

#### **RESULTS AND DISCUSSION**

Pressure Profile



Figure 2 shows the effect of vortex finder wall thickness on the predicted pressure distribution over a

diameter line in Y=650 mm from the base of the hydrocyclone. The peak values of pressure first decrease slowly and then sharply from the wall to the axis. The peak values of pressure near the wall decrease with increasing wall thickness of the vortex finder, implying that less pressure potential energy translates into kinetic energy.

Tangential Velocity



Figure 3. Effect of vortex finder thickness on the distribution of tangential velocity

Tangential velocity is the largest and most significant velocity component, which generates the centrifugal force for particle separation. Figure 3 shows the tangential distribution along the radius in Y=650 mm from the base of the hydrocyclone. It can be seen from Figure 3 that the distributions with different vortex finder wall thicknesses are consistent with the Rankine vortex, which is useful for modelling swirling motion inside a hydrocyclone. The observed decline in velocity is likely due to the increased frictional resistance introduced by the thicker vortex finder.

Short-circuit flow



Figure. 4. Effect of 10 mm vortex finder thickness on SCF

Figures 4, 5, and 6 show that an increase in vortex finder wall thickness decreases SCF. Contour plots of velocity for different vortex finder thicknesses show that a thinner vortex finder results in higher SCF, as indicated by stronger central core velocities.



Figure 5. Effect of 22.5 mm vortex finder thickness on SCF.



Figure 6. Effect of 35 mm vortex finder thickness on SCF

#### CONCLUSION

Vortex finder wall thickness plays a crucial role in particle classification within hydrocyclones. While increasing the thickness can reduce pressure drop, an excessively thick wall may lead to a higher pressure drop. A hydrocyclone with an appropriate vortex finder wall thickness could lower pressure consumption and energy loss while maintaining a specific feed velocity.

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# **Energy Harvesting Using Piezoelectric Shoe**

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# ABSTRACT

Alternative energy solutions like solar and wind are often impractical in remote locations due to inconsistent energy availability and storage challenges. Our innovative solution integrates piezoelectric shoes into a hybrid energy storage system, offering a sustainable and portable approach to energy generation and retention. The system harnesses mechanical energy generated during walking through advanced piezoelectric materials embedded in the shoe soles. This energy is stored in a compact lithium-ion battery or supercapacitor, enabling efficient and safe power retention for extended periods. To enhance functionality, solar panels can be incorporated into the shoe's surface or an external module for supplementary energy generation when stationary. The energy storage unit includes advanced safety features such as thermal management, overcharge protection, and a rugged waterproof design, making it ideal for externe environments. The modular and lightweight design ensures portability and adaptability, allowing users to power essential devices like GPS trackers, mobile phones, and flashlights. This project represents a step forward in leveraging piezoelectric technology for practical applications, bridging the gap between innovative energy solutions and real-world utility in remote and resource-constrained settings

Keywords: 1) Renewable Engery, 2) Piezoelectric Effect, 3) Sustainable Resources

#### INTRODUCTION

This paper focuses on the concept of piezoelectric shoes, which generate electricity from the mechanical energy produced while walking. Alternative energy solutions like solar and wind are often impractical in remote locations due to inconsistent energy availability and storage challenges. Our innovative solution integrates piezoelectric shoes into a hybrid energy storage system, offering a sustainable and portable approach to energy generation and retention. By considering both the current applications and future prospects, this study highlights the role of piezoelectric shoes in fostering innovation, promoting responsible consumption, and contributing to a more sustainable future. This approach supports the United Nations' Sustainable Development Goals.

# MATERIALS AND METHODS

The system harnesses mechanical energy generated during walking through advanced piezoelectric materials embedded in the shoe soles. This energy is stored in a compact lithium-ion battery or supercapacitor, enabling efficient and safe power retention for extended periods.

To enhance functionality, solar panels can be incorporated into the shoe's surface or an external module for supplementary energy generation when stationary.

The core mechanism relies on the piezoelectric effect, which converts mechanical stress into electrical energy. Piezoelectric materials have properties of non-linear optical materials which can also be used for communication purpose. Some examples of piezoelectric materials include Barium titanate, Zinc oxide, and

Lithium niobate.

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#### **RESULTS AND DISCUSSION**

Energy produced from 1 footstep can be used to light a 60W bulb for a very small fraction of second. The modular and lightweight design ensures portability and adaptability, allowing users to power essential devices like GPS trackers, mobile phones, and flashlights. The energy storage unit includes advanced safety features such as thermal management, overcharge protection, and a rugged waterproof design, making it ideal for extreme environments.

#### Piezoelectric shoes have various applications:

- Charging small devices like GPS trackers
- Energy Generation for Off-Grid Areas
- > Defense sector and Healthcare sector
- Fitness and Sports

#### The challenges developing piezoelectric shoes include:

- Energy Conversion Efficiency
- Durability of Materials
- Cost of Production
- Comfort and Design
- ➢ Scalability

#### **Recommend solutions for challenges:**

- Improved Energy Conversion
- Advanced Material Durability
- Cost Reduction Strategies
- Comfortable and Stylish Design
- Collaboration with the Footwear Industry

#### Piezoelectric shoes contribute to the following Sustainable Development Goals:

- SDG 7 Affordable and Clean Energy
- SDG 9 Industry, Innovation, and Infrastructure
- SDG 11 Sustainable Cities and Communities
- SDG 12 Responsible Consumption and Production

#### Future aspects of piezoelectric energy harvesting include:

- Piezoelectric Staircases
- Kids' Playgrounds & Fun Zones
- Piezoelectric Calculators
- Public Transportation Hubs

By integrating piezoelectric energy harvesting into urban infrastructure, we can transform cities into renewable energy hubs, making them more sustainable

#### **CONCLUSION:**

This project represents a step forward in leveraging piezoelectric technology for practical applications, bridging the gap between innovative energy solutions and real-world utility in remote and resourceconstrained settings. The development of piezoelectric shoes offers a sustainable and portable approach to energy generation and retention, supporting the United Nations' Sustainable Development Goals. While challenges remain, ongoing research and development promise to expand the applications and impact of this technology.

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# Biodegradation of Phenol: Comparison Between Free and Immobilized Organism

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# ABSTRACT

Phenol and its derivatives account for a significant portion of the waste produced by several businesses. It is well documented that phenol is carcinogenic to humans. Cell immobilization is a promising strategy for the biodegradation of these toxic substances because it offers several advantages over free cells, including increased biodegradation activity, protection from the matrix, increased cell density in the matrix, and enhancement of physiological activities such as enzyme induction. Pseudomonas sp. strain DT-4 isolated from industrial effluents, could exclusively use phenol as a carbon supplier and source of energy. This study focuses on identifying and immobilizing a phenol-degrading microbial strain to enhance degradation efficiency using GEN III BIOLOG techniques. Response Surface Methodology (RSM) was employed to optimize key parameters influencing phenol degradation. The percentage degradation of phenol by immobilized organisms was 96.00, which was higher compared to free organisms (91.05). According to this investigation, Pseudomonas sp. could destroy phenol entirely in 96 hours while remaining stable at the ideal development circumstance. This integrated approach provides a framework for developing efficient bioremediation systems for phenol-contaminated environments.

Keywords: Phenol; Biodegradation; Free & Immobilized organisms; RSM; GEN III BIOLOG.

#### INTRODUCTION

Phenol and its derivatives constitute a significant portion of waste products from several industries, including textile, oil refining, pharmaceutical and others [1]. Phenol is hazardous as are its chemical derivatives. Despite its reputation as an inhibitory as well as harmful substrate, another possibility for phenol is source of carbon by adapted biomass [2]. It is well-documented that phenol is a human carcinogen. Many production processes, such as those for oil, petrochemicals, medications, coking, and wood products, contain phenolic compounds. Discharging these compounds without sufficient treatment unveils a threat to freshwater and marine habitats along with individuals and animal occupants. Physical/chemical processes such as adsorption, chlorination, advanced oxidation processes, solvent extraction, coagulation, flocculation, reverse osmosis, and electrolytic oxidation; and biological processes such as biofiltration and biodegradation are examples of phenolic effluent treatment processes. The biological treatment of phenol

has received a lot of interest due to its eco-friendliness and ability to completely mineralize harmful chemical molecules [3]. However, inhibition of the substrate (phenol) and low specific conversion rates are common hurdles to phenol breakdown by bacteria. Many ways have been devised to circumvent substrate inhibition in order to tolerate high quantities of phenol. These include cell immobilization and adaptability to increasing phenol concentrations [4-5]. The immobilization of microorganisms offers several advantages, including the prevention of cell death, the facilitation of recycling, and the prevention of pathogens. acquisition of a higher cell density. Batch studies were carried out to investigate the effect of immobilization in calcium-alginate gel beads on the biodegradation performance of environmental contaminants. Statistical optimization growth conditions and medium components by response surface methodology for immobilized cells to enhance phenol biodegradation by Box- Behnken method.

# MATERIALS AND METHODS

# Sample Collection, Preparation and Microbial Identification Databases For Biolog Systems:

A total of 5 untreated textile effluent samples, were collected in autoclaved plastic bottles from 5 different locations in India. An approximation of the volume of the water from the surface was collected for bacterial assessment. The metabolic phenotype is the cornerstone of the Biolog Microbial Identification System.

**Optimization and Determination of percentage phenol degradation:** The following formula was used to determine the degree after discovering the amount of absorption quantity obtained by the UV-Visible spectrophotometer and the equivalent amount of the phenol level from the 4-Amino antipyrine procedure, the microbe destroyed the test specimen sample.

% degradation =

By using the Box-Behnken approach, three parameters had been selected for optimization since each of them had significant effects on the decomposition of phenol. They comprise Temperature of Incubation (X1) Contact Time (X2) Concentration (X3)

**Results and Discussion: The** organism was identified using GEN III Microlog According to the outcomes, the *Pseudomonas species Pseudomonas syringae pv maculicola* is the biological origin of the phenol-degrading bacteria *Pseudomonas*. Process parameters were optimized only for immobilized cells and Response surface methodology has been employed to statistically optimize the growth parameters and the elements of the medium for enhancing phenol degradation.

Using the Box-Behnken approach to optimise process parameters, microbial strains are immobilised to improve the efficiency of phenol biodegradation. A promising strategy for the biodegradation of toxic substances is cell immobilization. Since it outperforms independent cells in a number of ways, include greater biological degradation activity, protection against the matrix, higher cell density in the matrix, and augmentation of physiological processes like enzymatic induction. By assessing the rate of phenol breakdown by the immobilised cells, the influence of the beads' surface area was assessed [6].

**Effect of parameters:** The immobilized DT-4 strain's degradation of phenol at different times was investigated. Increasing the interaction period from 24 to 144 hours, it was found as degradation percentage increased from 0 to 77.3(**Fig 1**). These findings demonstrate that a significant contributor to degradation is the medium's contact time. From a starting concentration of 1200 mg/l up to 200 mg/l, the immobilized DT-4 strain's percentage of phenol degradation was reduced from 79 to 64.8 (**Fig 2**). The biodegradation of phenol was investigated to ascertain temperature effects on the rate of phenol degradation during a constant 96-hour period at various temperatures (30 to 40°C) (**Fig 3**). Observed at 36°C, significant degradation as well. The proportion of deterioration was shown to have risen with an elevation in pH from 2 to 8 about 77.2 up to 96 (**Fig 4**).



Fig 1: The impact of contact time on phenol degradation



Fig 2: The impact of the initial Phenol concentration on Phenol Degradation Rate



Fig 3: Temperature (°C) effect on % degradation of Phenol



Fig 4: pH impact on range of phenol degrades

**Optimizing experiment model:** Investigations were carried out using the Box-Behnken method demonstrated in Table to ascertain the optimum distribution of the selected elements in the medium being studied. Phenol deterioration as a consequence of the subsequent second order equation for regression (X1), temperature; (X2), contact duration; and (X3), concentration had been produced utilizing experimental results. A second-order response surface model's assessment of variance (ANOVA) fit outcomes.

**Conclusions:** A protocol for the enhanced biodegradation of Phenol has been developed. A model based on the finding has been developed and evaluated for the application of the same for the largescale degradation of phenol by *Pseudomonas syringae pv maculicola* using a largescale flask or a small bioreactor. The accomplished design is going to be extremely helpful to all local¬ industries, including Visakhapatnam Port Trust, Visakhapatnam Steel Plant, Hindustan Petroleum Corporation Ltd., and Greater Visakhapatnam Municipal Corporation, prior to discharging pollutants into the surrounding environment as the model was utilized for designing a treatment facility for phenol waste water effluent in which massive amounts collection can be achieved.

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# Development of Graphene-tin oxide Alloyed Quantum Dot Suspension with Enhanced Thermo-physical Properties for Thermal Management Application

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# ABSTRACT

In this study, graphene-tin oxide alloyed quantum dot suspension (AQDS nanofluid) was synthesized using a simple hydrothermal method with stannous chloride dehydrate, thiourea, and citric acid. The AQDS was developed by modifying graphene quantum dots to enhance their self-stability, lifetime, and the suspension's properties over time. UV absorption spectroscopy, Photoluminescence spectroscopy, X-ray diffraction, Zeta potential, and EDS were done for analysing the composition, stability of the suspension and thus the characterization. The zeta potential value of the quantum dot suspension at pH value 7 was found to be -35.5 mV, indicating good colloidal stability at neutral condition. The viscosity of the quantum dot suspension was measured at various concentrations, shear rates, and temperatures. Furthermore, the nanofluid suspension was evaluated for its heat transfer properties by measuring thermal conductivity (k) and heat transfer coefficient (h) at different concentrations. Remarkably, the stability got improved significantly, and a 45% increase in thermal conductivity was observed for the 1000 ppm AQDS compared to the base fluid (DI water). The addition of tin oxide to the graphene quantum dots notably enhanced the thermo-fluid properties of the suspension, making it a promising candidate for use as a coolant to replace traditional heat transfer fluids in thermal management systems.

*Keywords:* Graphene; Tin Oxide; Alloyed Quantum Dot Suspension; Dispersion Stability; Thermo-physical Properties.

# INTRODUCTION

Nanofluids are a class of engineered fluids that contain nanometer-sized particles (typically less than 100 nm in diameter) suspended in a base fluid, such as water, ethylene glycol, or oil. Recent advances in nanomaterials have sparked significant interest in the development of hybrid nanostructures with tailored thermo-physical properties for high-performance applications in energy, electronics, and thermal management systems [1]. Among these, quantum dots (QDs) – semiconductor nanoparticles with size-

tunable optical and electronic properties – have emerged as promising candidates for next-generation nanofluids due to their high surface-to-volume ratio and unique quantum confinement effects.

The fascinating properties graphene quantum dots (GQD) have been emerged it in numerous applications. Although GQD having higher dispersion stability it lack of self-stability as time proceeds. Most of the QDs show the same behaviour during its applications. The issues related to the self-stability of the quantum dots can be overcome by developing a quantum dots with the use of different materials [2].

In particular, the alloying of tin oxide  $(SnO_2)$ , a widely used n-type semiconductor, with graphene, a twodimensional carbon nanomaterial known for its exceptional thermal conductivity and mechanical strength, offers a synergistic platform for enhancing the overall thermal, electrical, and stability characteristics of nanoparticle suspensions.

The integration of graphene and tin oxide within a quantum dot framework forms a novel class of composite nanostructures – graphene-tin oxide alloyed quantum dots (GTO-QDs) – that exhibit superior thermo-physical behaviour compared to their individual constituents [3].

These suspensions not only demonstrate improved thermal conductivity and heat transfer performance but also maintain stability over extended periods, addressing key limitations in conventional nanofluids.

This study focuses on the synthesis, characterization, and evaluation of graphene-tin oxide alloyed quantum dot suspensions, emphasizing the enhancement of thermo-physical properties. By exploring the underlying mechanisms driving these enhancements, the research aims to establish a foundational understanding for the development of efficient, scalable nanofluid systems for next-generation thermal technologies.

# MATERIALS AND METHODOLOGY

Precursors such as citric acid and urea were purchased from Alfa Aesar, India. Urea act as the stabilizer during the QDs preparation. For the development of G-ZnO AQDS, Zinc acetate dehydrate was purchased from Chemical House, India.

#### Synthesis of Graphene quantum dots

In this process, 1.64 gm of citric acid and 1.48 gm of urea dissolved in the distilled water. The mixture was stirred up to a clear uniform solution. The prepared solution has taken in a Teflon lined autoclave. The high pressure Teflon lined stainless steel autoclave reactor kept in an electrical furnace at 160°C for 120 minutes to undergo hydrothermal process. After the completion of the reaction, obtained product was allowed to cool until room temperature, a pale yellow coloured GQD suspension is obtained.

# Synthesis of Alloyed Graphene Zinc oxide quantum dots

In this process, 40 ml of the synthesised GQD suspension was taken in a beaker. About 0.92 gm of zinc acetate dihydrate is properly dissolved in GQD suspension. This mixture stirred for 120 minutes at 700°C in a magnetic stirrer. After the reaction, a yellow coloured G-ZnO AQD suspension was obtained. The G-ZnO AQD suspension dried at 800°C and crushed using a mortar and pestle to get the G-ZnO AQD nanoparticles.

# Analysis of the thermos-physical properties of G-ZnO AQD nanofluid

The obtained alloyed quantum dot powder is used to prepare the quantum dot suspension with different concentration. Samples were prepared based on the G-ZnO AQDs at very low concentrations to high concentrations including 50 ppm, 100 ppm, 200 ppm, 300 ppm, 400 ppm and 750 ppm. The presence of G-ZnO AQD is confirmed by using an energy dispersive X-ray spectroscopy (EDS). The size and quantum confinement effect of G-ZnO AQD was carried out using a photoluminescence spectroscopy. The thermal conductivity of the nanofluid suspensions were studied using a transient thermal conductivity analyser. The viscosity and the rheological properties of the nanofluid were investigated by using a rheometer.

#### **RESULTS AND DISCUSSIONS**

The photoluminescence spectra of the prepared sample of G-ZnO AQD suspension of different excitation wavelengths is shown in figure 1. The range of excitation wave length to be used for the PL analysis is obtained from the UV-visible absorption spectra, and excitation wavelength ranges from 300 to 350 nm.



Fig. 1: Photoluminescence spectrum of G-ZnO AQD suspension

The prepared sample of G-ZnO AQD exhibit high luminescence with a special covering blue colour. The photoluminescence spectra describes the electronic transition of QDs. The prepared sample shows blue colour in the photoluminescence it is evident that the QDs having smaller size. Photoluminescence spectra analysis of QDS shows the maximum intensity is for emitted wavelength of 430nm. It shows the developed QDS absorbs the UV light and emit visible light (fluorescent).

The EDS spectrum given in figure 2, describes the elemental composition of the material, and the corresponding numerical results are shown in table 1. From the table it is clear that, the prepared sample constitute of three elements.



Fig. 2: EDS spectrum of G-ZnO AQD nanoparticle

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Element	Line type	Weight %	Atomic %
С	K series	52.35	64.14
0	K series	36.13	33.25
Zn	K series	11.56	2.6
Total		100	100

Table 1: Elemental analysis of G-ZnO QDs

Carbon (52.35 wt.%) present in graphene, zinc (11.56 wt.%) and oxygen (36.13 wt.%) present in zinc oxide which substantiates the purity and concentration of graphene (50 wt.%) in the composite material.

In zeta potential analysis, the zeta potential of prepared suspension at various pH measured, and the results are shown in the figure 3. It is observed that the value of zeta potential decreases from +20 mV to -45 mV as the pH changes to acidic region to basic region. At pH 7, the suspension shows a zeta potential of -38 mV. So it can be concluded that the prepared suspension shows good dispersion stability in the neutral condition [4].

The thermal conductivity of prepared G-ZnO AQD suspension was measured and the results were obtained in figure 4



Fig. 3: Effect of pH on the Zeta potential

It was found that a linear increase in the thermal conductivity of with concentration. The maximum thermal conductivity was shown by the QD suspension with the concentration of 700 ppm at 300C. For easy comparison, the variation of the thermal conductivity of GQDs were also included in the graph.

Thermal conductivity of the water at room temperature is 0.585 W/(m·K). The maximum thermal conductivity shown by the G-ZnO QD suspension (700 ppm) was 0.756 W/(m·K). Hence, it can be concluded that, the increment in the thermal conductivity for the prepared G-ZnO AQD suspension as compared to its base fluid (water) is 30%.



Fig. 4: Thermal conductivity of G-ZnO AQD suspension

#### CONCLUSION

The development of graphene-tin oxide alloyed quantum dot suspensions represents a significant advancement in the field of nanofluids, offering remarkable improvements in thermo-physical properties such as thermal conductivity, stability, and heat transfer performance. The synergistic integration of graphene's exceptional conductivity with the thermal and chemical robustness of tin oxide quantum dots creates a hybrid material well-suited for demanding thermal management applications. These findings not only deepen our understanding of nanoscale heat transfer mechanisms but also pave the way for innovative solutions in energy systems, electronics cooling, and advanced nanotechnology-based thermal systems.

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# E-waste Management and its Effects on Society and the Environmental Sustainability: A Review

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# ABSTRACT

Recent studies shows that approximately 7.8 kilograms of E-waste was generates per person globally in 2022. This surge is largely attributed to the growing use of electronic and electrical devices across the globe. E-waste has several hazardous components that, if not handled appropriately, can harm the ecosystem and, more significantly, human health. This review presents the latest global scenario and regional trends of E-waste generation statistics. In addition, people's knowledge of e-waste management, generation, and fundamental treatment techniques is examined. This research also aims to bring light on the role that E-waste management methods play in environmental sustainability. The results of the investigation show that although there is a lack of information considering the production of E-waste to wealth were also systematically reviewed. Some solutions to this quickly expanding issue include imposing producers' waste disposal responsibilities, establishing official recycling facilities, and strictly enforcing E-waste laws.

Keywords: E-waste, Disposal Methods, Management, Society, Environmental Sustainability

#### INTRODUCTION

In the rapidly advancing digital age, electronic devices have become an indispensable part of modern life. E-waste, on the other hand, is one of the waste streams that is expanding the quickest in the globe because of this technological revolution. The term "e-waste" describes abandoned electrical or electronic equipment that has outlived its useful life, such as computers, cellphones, televisions, batteries, and household appliances. The improper handling and disposal of E-waste pose serious risks to both human health and the environment due to the presence of harmful materials such as Pb, Hg, Cd, and brominated flame retardants. Figure 1 shows the uses of E-waste.



Figure 1. Uses of E-waste.

Asia is the biggest generator of E-waste globally. In 2019, the Asian continent produced approximately 24.9 million metric tonnes (MT) of E-waste, accounting for nearly half of the global total that year. Effective E-waste management is crucial to address the eco-friendly and social consequences of this increasing crisis. Without proper systems in place, E-waste frequently ends up in landfills or is managed under unsafe conditions in informal sectors, leading to soil, air, and water pollution (Ghimire and Ariya 2020). These environmental hazards also translate into social issues, particularly in developing countries where unregulated recycling practices expose vulnerable communities to toxic substances, increasing the risk of chronic diseases and developmental issues.

On the other hand, implementing sustainable E-waste management strategies like recycling, refurbishing, and responsible disposal not only reduces ecological harm but also supports the circular economy by recovering useful materials such as Au, Cu, and rare earth metals. This promotes environmental sustainability while creating economic opportunities and encouraging responsible consumption and production patterns. In this context, understanding the challenges and solutions surrounding E-waste handling is vital for building a more sustainable, equitable, and tech-savvy society.

#### HEALTH EFFECTS OF E-WASTE

E-waste confines a complex mixture of toxic substances, including Pb, Hg, Cd, As, and flame retardants. When E-waste is burned or dismantled improperly—often in informal recycling sectors—these toxic elements can seep into the soil, water, and air. Exposure to such pollutants can have serious implications for human health.

The health effects of E-waste are far-reaching. Children and pregnant women are particularly vulnerable. Lead exposure, for example, can damage the nervous system and hinder brain development in children (Parvez et al., 2021). Mercury can impair cognitive function and motor skills, while cadmium exposure

may lead to kidney damage and bone degradation. Laborers in informal E-waste recycling sectors often experience from respiratory problems, skin disorders, and long-term chronic illnesses due to constant exposure to toxic dust and fumes.

Furthermore, communities living near unregulated E-waste dumps or processing areas face heightened risks. To mitigate these health risks, stricter regulations on E-waste handling, public awareness, and investment in safer recycling technologies are essential. Proper E-waste management not only protects human health but also promotes sustainable development and resource conservation.

### LITERATURE AND WORLD SCENARIO OF E-WASTE

As of 2022, the world produced an projected 62 million MT of E-waste, demonstrating an 82% rise since 2010. This surge is driven by factors such as increased consumption of electronic appliances, shorter product lifespans, and inadequate repair options (Priyashantha et al., 2022; E-waste Monitor; 2024, WHO, 2024). India has experienced a significant increase in E-waste production over recent years. According to data presented in the Rajya Sabha, E-waste generation rose from 1.01 million MT in 2019–20 to 1.751 million MT in 2023–24, marking a 72.54% increase over five years (India's E-waste management, 2025; Tancredi 2024). This surge is largely attributed to the growing use of electrical and electronic devices across the country. Figure 2 indicates the world E-waste quantities, estimates, and total E-waste by category.

The United States generates a huge volumes of E-waste annually, reflecting its high consumption of electronic devices. According to



Figure 2. Global E-waste quantities, estimates, and total E-waste by category.

the World E-waste Statistics Partnership, in 2022, the U.S. produced approximately 4.054 million MT of E-waste, equating to about 21.3 kilograms per person (The Global E-waste Monitor 2024). As of 2019, China was the world's greatest producer of e-waste, with an annual production of about 10.1 million MT. India ranked third in the world after China and the US, producing over 3.2 million MT of e-waste annually. (Global E-waste Statistics, 2025). In 2019, the Arab States generated E-waste containing valuable materials worth an estimated USD 3 billion, including 13 MT of gold and 0.47 MT of rare earth metals. Over 99% of E-waste in the region is not collected or sent to environmentally sound management facilities, with most ending up in landfills (Iatoni et al., 2021).

### CONCLUSION

Managing e-waste has become a crucial concern in the modern world, which is becoming more digital and consumer driven. Large volumes of electronic waste have been produced as a result of the quick turnover of electronic equipment, which is driven by customer demand and technological innovation. Both the environment and human health are seriously threatened by improper disposal and uncontrolled recycling of this garbage. Particularly in areas where informal e-waste processing is common, toxic heavy metals found in e-waste can contaminate soil and water sources, degrade air quality, and ultimately result in major health problems for populations.

From a societal perspective, the effects are far-reaching. Informal E-waste handling often involves lowincome workers, including children, who are exposed to hazardous conditions without proper protection or training. Moreover, the loss of valuable materials due to improper recycling practices represents a significant economic and resource inefficiency.

To mitigate these issues, effective E-waste management practices must be implemented and enforced. These include promoting producer responsibility, encouraging eco-design of products, increasing consumer awareness, and strengthening recycling infrastructure. Governments, industries, and individuals must work together to create a sustainable and circular economy where electronics are responsibly manufactured, used, and disposed of.

In conclusion, managing E-waste responsibly is not only an environmental necessity but a social obligation. By taking proactive steps today, we can protect our planet, support healthier communities, and ensure sustainable development for future generations.

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# Modeling and Simulation of Process coupling in Sustainable Esterification by Pervaporation

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# ABSTRACT

Esterification, an essential reaction in both the chemical and pharmaceutical fields, often encounters equilibrium constraints that limit the product yield. Pervaporation, a unique membrane separation process involving phase change, offers a successful remedy to these limitations by selectively eliminating either water which is an esterification byproduct or ester which is the main product of concern and shifting the chemical balance toward product production. This work investigates the integration of pervaporation with esterification, highlighting the significance of precise pervaporation modeling in ensuring sustainable and efficient process coupling. Empirical models reported in literature were investigated for their impact on estimating final composition of the products. The effect of non ideality nature of components was also considered by incorporating activity coefficients into the model through UNIFAC method. By creating robust models that simulate this hybrid pervaporation coupled esterification process, it is feasible to anticipate membrane performance, optimize operational conditions, and analyze energy efficiency. Furthermore, pervaporation and waste creation. This combined approach not only increases yield, but also adheres to the principles of green chemistry and sustainable process design. The incorporation of pervaporation models into esterification processes thus has transformational potential, providing a path to eco-friendly production methods that are both economically and environmentally advantageous.

Keywords: Pervaporation, Esterification, Non-Ideal system, Modeling and Simulation, Process Intensification

# **94** Green Process Design and Sustainable Chemical Manufacturing (TIChSCON'25)

# INTRODUCTION

Esterification reactions hold a pivotal role in the synthesis of awide array of chemical compounds, serving as a cornerstone process within both the chemical and pharmaceutical industries (Makertiharta & Dharmawijaya, 2017). However, a significant challenge in esterification lies in its inherent equilibrium limitations, which often restrict the achievable product yield (Jyoti et al., 2015; Makertiharta & Dharmawijaya, 2017). This equilibrium can limit the conversion of reactants to products, resulting in lower yields and necessitating additional separation and purification steps. Overcoming these limitations is essential to improve the efficiency and economic viability of esterification processes, driving the need for innovative techniques to shift the equilibrium towards product formation.

Traditional methods employed to drive esterification reactions toward completion often involve the use of excess reactants, high temperatures, or the removal of water through distillation (Mahdavian & tofigh kouzekonani, 2015). These drawbacks highlight the need for more sustainable and efficient methods to enhance esterification reactions, aligning with the principles of green chemistry and promoting environmentally friendly manufacturing practices.

# PERVAPORATION: AN INNOVATIVE SOLUTION FOR ESTERIFICATION

### Esterification

Pervaporation is a membrane separation process that can overcome equilibrium limitations (Castro-Muñoz et al., 2018; Jyoti et al., 2015). It selectively removes water or ester to shift the reaction equilibrium (Mahdavian & tofigh kouzekonani, 2015; Makertiharta & Dharmawijaya, 2017). Pervaporation can be integrated with esterification to enhance process efficiency and sustainability (Khudsange & Wasewar, 2017; Quintero-Arias et al., 2023). Pervaporation emerges as an innovative and promising solution for overcoming the equilibrium limitations inherent in esterification reactions, offering a pathway to enhance product yield and process efficiency (Castro-Muñoz et al., 2018; Jyoti et al., 2015).

# THE SIGNIFICANCE OF PERVAPORATION MODELING

Accurate modeling is crucial for designing efficient pervaporation-coupled esterification processes. Modeling aids in analyzing energy efficiency and reducing waste(Chandane et al., 2016; Mahdavian & tofigh kouzekonani, 2015; Quintero-Arias et al., 2023)the esterification reaction of propionic acid with isobutyl alcohol to produce isobutyl propionate and water was studied. The performance of esterification reaction was compared by using the batch process and the pervaporation assisted hybrid process which performs the reaction and separation simultaneously. A polyvinyl alcohol–polyethersulphone (PVA–PES. One of the primary benefits of pervaporation modeling is its ability to predict membrane performance, including flux and selectivity, under different operating conditions, such as temperature, pressure, and feed composition (Chandane et al., 2016; Quintero-Arias et al., 2023)the esterification reaction of propionic acid with isobutyl alcohol to produce isobutyl propionate and water was studied. The performance of esterification reaction was compared by using the batch process and the pervaporation since and separation simultaneously. A polyvinyl alcohol–polyethersulphone (PVA–PES. This predictive capability is essential for designing robust and efficient pervaporation units that can effectively enhance esterification reactions.

Pervaporation modeling also provides valuable insights into the energy efficiency of the integrated process, aiding in the identification of strategies to reduce energy consumption and minimize waste generation (Mahdavian & tofigh kouzekonani, 2015). This study develops and simulates a generalized mathematical model for the esterification reaction between ethanol and acetic acid.

For pervaporation coupled esterification modeling, two empirical models that have been published in the literature were examined. The available experimental data published in the literature was used to validate the model.

#### Model development for Pervaporation coupled Esterification:

Overall, the esterification reaction between ethanol and acetic acid exhibits second order reaction kinetics, although first order kinetics for each component.

All other elements present at any given time, with the exception of water, are assumed to have zero fluxes. Only water can flow across a hydrophilic membrane. The fluxes of every other element are insignificant.

Below are the governing equations for the esterification of ethanol and acetic acid. The following is a schematic representation of the esterification reaction between ethanol and acetic acid that yields ethylacetate and water:

The esterification reaction's rate of ester production can be expressed as follows:

The effect of non ideal nature can be studied by taking actual concentration terms in modeling through activity coefficients calculated from UNIFAC group contribution method(Zou et al., 2010). Empirical relations between flux and concentration of water can be assumed with modeling in linear and non linear form reported in literature as follows(Ajit P.Rathod, n.d.; Hasanoğlu & Dinçer, 2011)

Coupled model was simulated for esterification of acetic acid and ethanol to form ethyle acetate and water and experimental data reported in literatature was used for analysis(Hasanoğlu et al., 2009)permselective to ethyl acetate formed by reaction. In this way, conversions were increased by continuous removal of ethyl acetate from the reaction media. Conversions are found to increase with an increase in both molar ratios of reactants and temperature. Temperature has a strong influence on the performance of the pervaporation membrane reactor because it acts on both the esterification kinetics and pervaporation.","container-title":" Desalination","DOI":"10.1016/j.desal.2009.02.034","ISSN":"00119164","issue":"1-3","journalAbbreviation":" Desalination","language":"en","license":"https://www.elsevier.com/tdm/userlicense/1.0/","page":"662-669","source":"DOI.org (Crossref

#### **RESULTS AND DISCUSSION:**

Rate constants were estimated initially through kinetic modeling and used for pervaporation coupled modeling.

Comparison results for experimental data and model results were shown in figure 1 and figure 2. RMSD values of models suggest that non linear relation between flux and concentration of water gives better results than the linear relation.

Empirical Model	Empirical Constant	Value	RMSD		
Linear	$k_{pv\_1inear}$	2.81*10-12	0.03761		
Nonlincor	$\mathbf{k}_{\mathrm{pv1\_nonlinear}}$	0.001798	0.00545		
nommear	k <sub>pv2_nonlinear</sub>	0.002999	0.00545		

Table 1: Values of fitted Parameters for pervaporation coupled esterificaton



Fig. 1: Modeling of Pervaporation Coupled Esterification with non-linear relation between flux and water concentration





#### CONCLUSION

The model equations for the pervaporation coupled reaction were developed using the available reaction kinetics and pervaporation data for the esterification of acetic acid with ethanol. The efficacy of two empirical models that have been published in the literature was examined. It was discovered that the nonlinear relationship between flow and water concentration produces superior outcomes compared to

the linear relationship. The robustness of pervaporation linked esterification can be increased by using theoretical models for pervaporation.

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# Bidirectional RNN Based Molecule Generation for RTK Targets Using AI for Sustainable Drug Discovery

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# ABSTRACT

Drug discovery is a complex, time-consuming, and costly process. The amount of time and money spent to bring a single drug to market is very high. When addressing rare diseases, the challenge is even more complex, due to limited patient data and less commercial benefits for the same. This issue comes as a hindrance in addressing rare diseases. Traditional drug discovery involves the following elaborate and laborious steps, target identification, highthroughput screening, lead compound optimization, and extensive clinical trials. All this process involves manual experimentation and trial-and-error methods which takes considerable time and money. AI techniques can analyze vast biomedical datasets, identify hidden patterns, suggest drug-target interactions, and come up with novel molecular structures with high therapeutic potential. In this work a Bidirectional Molecule Generation framework(BMGF) utilizes Bidirectional RNNs to design novel molecules targeting Receptor Tyrosine Kinases (RTKs) from ChEMBL, ZINC, and GPCRdb databases, comprising approximately 2474590 molecules at first step of traning. BMGF framework uses a bi-directional Recurrent Neural Network architecture to capture the sequential dependencies of molecules in both forward and backward directions. It has a filtering mechanism to select valid molecules that satisfy specific drug-like properties and exclude molecules with undesired features. This model generates about 2,000 novel molecules that meet the filtering criteria and exhibit high chemical diversity. BMGF framework generates diverse and valid molecules for RTKs target and it discovers new drugs. This report utilized a bimodal approach, and results were generated using Bimodal RNN. Integrating AI and deep learning techniques can revolutionize medicine and lead to more personalized and effective treatments for patients with rare and challenging diseases.

*Keywords:* Drug Discovery, Bidirectional RNN, Receptor Tyrosine Kinases, Molecule Generation, Artificial Intelligence

# 1. INTRODUCTION

Nowadays, new or rare diseases affect only a small number of people, while the number is higher worldwide. Drug discovery for rare diseases is a big challenge because the number of patients are less, funding is also limited and there is no data about diseases [1]. Receptor Tyrosine Kinases (RTKs) are proteins molecules is an important role in finding many rare diseases and cancers. By using RTKs proteins is a useful strategy in drug development [2].

Artificial Intelligence, especially Deep learning techniques, is utilized in process of drug discovery. It helps the researcher explore chemical space and chemical compounds. Based on the chemical compounds, the researcher predicts a drug that may work for rare diseases[3]. The Simplified Molecular Input Line Entry System (SMILES) format used to represent molecules as strings and it make easy to design a deep learning models to process chemical structures.

Bidirectional Recurrent Neural Network (BiRNN) is a one of the type of Recurrent Neural Network (RNN) architecture that processes data in both forward and backward directions. This allows the model to have access to both past and future context at each time step, it especially useful for tasks like bioinformatics.

Bidirectional Recurrent Neural Networks (BiRNNs) can learn from these SMILES strings to generate new, valid molecules [4]. Studies show that such AI models can create novel molecules with good drug-like properties [5,6].

# 2. LITERATURE SURVEY

Drug discovery, particularly for new or rare and complex diseases, faces challenges because of data unavailability[1]. Recent advancements in artificial intelligence have demonstrated the efficacy of deep learning models, notably Bidirectional Recurrent Neural Networks (BiRNNs), in accelerating and optimizing this process. BiRNN-based frameworks, such as BIMODAL and CNSMolGen, have proven superior in generating novel molecules with high chemical diversity, validity, and drug-likeness by capturing the sequential dependencies of SMILES strings in both forward and backward directions[3] [4]. These models outperform unidirectional architectures in novelty and scaffold diversity, essential for discovering molecules with new mechanisms of action. Integrating BiRNNs with molecular fingerprints (e.g., RDKit) and generative methods like autoencoders and GANs further enhances predictive capabilities [8][9].

Data augmentation strategies, such as SMILES randomization, have also been used in conjunction with BiRNNs to improve generalization across chemical space [20]. These methods have shown promise in domains like toxicity prediction, CNS drug design, and rare disease therapeutics by identifying target-specific interactions and generating valid, synthesizable molecules from databases like ChEMBL and ZINC [5][6]. As a result, BiRNNs stand at the forefront of revolutionizing drug discovery, offering data-efficient, scalable, and accurate solutions that align with personalized and precision medicine goals.

# 3. PROPOSED SYSTEM

# 3.1 Dataset Preparation

ChEMBL is the manually curated database of bioactive molecules, their prop- erties, and their activities against biological targets. It is used primarily for drug discovery and development. It provides a comprehensive source of chemical and physical data that can be used to identify potential drug targets and lead compounds, optimize drug design and development, and understand the mech- anisms of action of drugs. The data representation in ChEMBL is in molecular structures, which can also be represented in a molecular file format such as SMILES [7]. ChEMBL offers details on the biological activity of the compounds in the database, including their potency, effectiveness, and specificity. This in- formation can help us train our model on the dataset and obtain well-efficient results.



The useful properties in ChEMBL like canonical\_smiles, molecular weight, logP, hydrogen bond acceptors/donors, and QED scores. Data is cleaned by removing entries that were incomplete or not chemically meaningful.

### 3.2 Preprocessing

Preprocessing of ChEMBL data is an essential step to make it worthwhile for applications such as drug discovery. Preprocessing of ChEMBL data is a cru- cial step to make it useful for multiple applications such as drug discovery. We describe the preprocessing steps to convert ChEMBL data into a CSV format containing helpful information. The ChEMBL database is an extensive, publicly available database of bioactive molecules, their targets, and their associated activities. It contains a wealth of information that can be used to study drug-target interactions and drug dis- covery. However, the raw ChEMBL data is not in a readily usable format for analysis. Hence, it needs to be preprocessed to extract useful information and convert it into a form that is more accessible.

# 3.3 Data Extraction

We downloaded the ChEMBL database in an SQLite format from the ChEMBL website. We then extracted the required data from the database using SQL queries. Our Data was represented in .csv format with many columns.

### 3.4 Data Cleaning

We cleaned the extracted data to remove duplicates, inconsistencies, or errors. We also removed any data that was not relevant for our analysis.

#### 3.5 Data Transformation

We transformed the cleaned data into a suitable format for analysis. We con- verted the data into a CSV format and added additional columns to provide more helpful information.

#### 3.6 Data Enrichment

We enriched the data by adding additional information to the CSV file. This included information such as the compound's ID, structure, SMILES string, etc. In conclusion, preprocessing of ChEMBL data is an important step to make it useful for analysis. The preprocessing steps included data extraction, cleaning, transformation, and enrichment. The resulting CSV file contained useful infor- mation that was given to our model. The file's name is GPCR.csv, and it has 363387 rows of SMILES string. The file is shown below in Figure 1.

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18 CHEM	BL283365	MOL	Small molecule	0	0 0	0	0	- 4	4	4	4	4 230.28	16	5	1 84.02	3	0	251	146	073	23318 CI6H1N	O-C(+) (XPCV 1/C0-ISCIEHINICO3/3)-H-ID-T-R(K)2/0-H(I286-027-812)R(2)(142-143-5 TH-HOH(H)8/2)(2)
13 CHEM	BL6230	MOL	Small molecule	0	0 0	0	0	- 4	4	4	4	4 394.19	231	5	2 87.98	3	0	3,27	234	075	36312 CI6H10	Ovotem PDVDE INCH-INCREMONING-IN-3-14(2-4-10)(20)24-5-117-10)(20)24-824(24-9)(22)3-3244-4,5(24)(4-20)2(24)
20 CHEM	BL6254	MOL	Small molecule	0	0 0	1	0	- 4	4	4	4	-1 127.73	181	5	1 84.02	3	0	11	2.8	074	327.04 CISH00	O-C(rk FMFL xCx/sC6H0CN0C0xf7-0-761(2)+8(2)(8-42)(8-20)(8/2)(8-2)(9-2)-3-404(8+1)(8)(2)(2)
21 CHEM	BL6268	MOL	Small molecule	0	0 0	0	0	- 4	4	4	4	4 30832	131	5	2 87.98	3	0	258	150	0.76	309.11 C17H59	Color(+ YV0/T1 Inclu-ISCTHRV0036-114-000-T1219-F8201-IB-007-8-H119822(25-3-2-4-6-042-0)8/22H393(H-8/2123
22 CHEM	BL414196	MOL	Small molecule	0	0 0	0	0	- 4	4	4	4	-1 43351	2.68	1	1 \$3.0	5	0	2.94	271	0.66	43321 C24H27	COntrol GERCX INCOMISTICARE2/MICROIN/31/01/01/01/01/02/02/02/02/02/02/02/02/02/02/02/02/02/
20 CHEM	BL283528	MOL	Small molecule	0	0 0	0	0	- 4	4	4	4	-1 32331	16	6	1 94,15	4	0	235	13	073	12319 CI7H08	CDolox PTFLFL IICIN ISCTHONIO46/2444-84-0354-496/22/1-2-6-07-3-10247/22/16-92/10-14-2042-04 HO(H, 92/23)
a CHEM	BL1299	MOL	Small molecule	0	0 0	1	0	- 4	4	4	4	4 43553	18	8	1 10.8	5	0	229	2.07	0.65	435.1 C/8H2#	COxissi RYVAELIISOMISISTIMENKOASUKASIA-11-12-19[1-19]19[1-2020 10[2+17]2]19[2:5-7-20]16-22[20]2-25[6-4-3-20-498-3-4-9-14-5-982,5-890]H2(9:21,2]
8 CHEM	BL6266	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	4 80636	\$29	2	2 31/2	0	2	0.8	03	0/7	606.07 C42H46	close 2x INTINE HON-ISCAD-MARKA 246 (23) 4440 26 (14) 40 (6 (16) 44 (0) 40 (22) (34) 5 (23) (23) (25) (7) (31) (2) (50) (45) (35) (37) (37) (37) (37) (37) (37) (37) (37
8 CHEM	BL6354	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	-1 468.6	55	2	2 588	5	1	-287	-287	0.13	46823 C32H28	National CREWS INCOMENDATION IN THE REPORT OF THE REPORT O
a CHEM	BL42307	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	4 49636	5.61	2	2 588	7	1	-198	-198	0.28	49626 (394-02	National SSYL25 NDN-ISICONDINAND DI INSULTIDO TO 14 SOLUZIO DE DEPOSITO NO FORMA A SOLUZIO DO DO DO DA A A SOLUZIO DA S
8 CHEM	BL6361	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	-1 49464	6	2	2 588	6	1	-28	-28	022	494,25 C34-00	Notesh VEPLYK NON-ISIC SHORING STATISTIC TO 15 2011/02/21/5 1/3/12 6/2/19 1/3/10 1/2/01 4/3/24 0/22 0/2010/4/24 8/3/01/04/22/15 0/H22/24/06-24/05
23 CHEM	BL6212	MOL	Small molecule	0	0 0	1	0	- 4	-1	4	4	4 80837	\$31	2	2 31.82	ſî	2	10	10	0.08	608.39 C42H48	door(C) #THECkDik/SECA2H#RMx(D5/T-2343/32/3)#\$(D3/5/8-7/1-20/5/#25/5/D22/3)(3)#(D4/4-8/3)#44/22/22/4(D4/3)/21/8/D22/3)(D22/5/#24/28/0/04/1/20/22/3)
30 CHEM	BL258254	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	-1 632.9	179	2	2 31/2	0	2	15	15	0.6	\$32,39 C44H8	CECK2 XEVP1 INCHESICIENTIALIZATION CATE DI A FAZZO ZI A MERITA DI A MANIMINI DI 2523 3022 65 300 7-35 1523 3024 20 30347 0 27 40 523 153 103 157 3 15400 107
a CHEM	BL28885	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	4 63431	541	2	2 31/2	0	2	168	169	0.6	6344 C44-50	close2x PVXDX hDx458C444884x1246323146423832488468382440446312523382235388735582337242035334731274045231553103107355438917
22 CHEM	EUCL-500	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	4 \$208	\$21	2	2 31/2	0	2	125	125	0/7	\$20.39 C43H48	close2x1 SYCHP1 MCM-SICADHRINKI 246 (22) 454 (263) 41NO 16 16 4 (30) MXXX 07 23 (63) 21 41 (23) 22 (63) 412 4 (24) 25 40 44 (21) 15 3 103 (0 7 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 17 4 15 4 (23) 104 (7 7 15 103) 104 (7 7
30 CHEM	BL6356	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	4 49082	54	2	2 588	4	1	-275	-275	0,13	49023 C33H28	Nation USCMU NCN-ISIC31468/WK34-00-07-5 0002-07-01-5 20000201-22-41-05-04/07-22/05-05-01-0202020203-05-44-003024-22-4-0-022/07-02-02-02-02-02-02-02-02-02-02-02-02-02-
34 CHEM	BL289729	MOL	Small molecule	0	0 1	i i	0	4	4	4	4	-1 468.6	55	2	2 588	5	1	-287	-287	0.13	468.20 C32H28	Nationian BACSYN, MCDANISCO, CHARMAN, COLOR TH, STOTT COMPANY, CARLENNING, CARLENN
S CHEM	BL6344	MOL	Small molecule	0	0 0	0	0	-1	4	4	4	4 378.2	277	5	1 84,12	3	0	423	33	071	17512 CI7HIC	Cclose < LIXPLX hChe18/CTH1C20/C006/386/102/172502/8020/2012/2015/2010024/0.0007/40008/248/HC012/22250
35 CHEM	BL6207	MOL	Small molecule	0	0 1	1	0	4	4	4	4	4 6837	18	2	2 31/2	0	2	1.82	0.92	0/7	61837 C43H46	CONSIGN AT FOLD INCIDENT AND A REAL AND A
ST CHEM	BL635	MOL	Snal nolecule	0	0 1	1	0	4	4	4	4	4 48283	142	2	2 588	6	1	-243	-243	0,31	482.25 C30H00	Notes 19000 NOA-19000 NOA-190000 7-9-9002-7-9-1-920002022-9-0-9-900 N-92025-1-5-2016-0-2502-37-21-9-005054-2-4-9-0202031N-2024-3552228-0-2
38 CHEM	BL286223	MOL	Snal nolecule	0	0 1	1	0	4	4	4	4	4 57685	236	1	2 16921	1	1	37	3.48	12	57627 C30H38	COD-0 P2Y22 https://com/sindexin4.04.6.22.209.202444.203.004-2022006-6-4-20517-0427107-546-20-4-020532-201702-0-201942-36-01.8-104-714-714
33 CHEM	BL283077	MOL	Small molecule	0	0 1	1	0	4	4	4	4	4 4215	251	2	1 \$3.0	6	0	3.02	273	0.85	42121 C23H27	COME DOWF HOM SIGZIEZTED AND A FUTURE AND A CONTRACT AND A CONTRAC

Figure 1. chembl\_data.csv

# 4. MODEL ARCHITECTURE AND TRAINING

The Bi-RNN model architecture can be based on either a simple RNN or a Long Short-Term Memory (LSTM) network. The model should be designed to learn the sequence of characters or tokens that make up a molecule. There are steps involved in the framework, as shown in figure 2.

#### SMILES PROCESSING PIPELINE



Figure 2: Framework of Bi-RNN

The Architecture works as the Smiles string are converted to one-hot encoding; the processed data is then trained on Recurrent Neural Network. The sampling data is then collected and passed in one hot encoder again, generating the new Smiles strings. We can convert the Smiles string into a molecular structure with the help of Library RD-Kit

# Training:

The next step is to train the RNN model on the prepared dataset of molecules. This involves minimizing the model's loss function, which measures the difference between the predicted and actual sequences of characters or tokens. The framework was trained on 3 epochs with 5-folds split was used to distribute the dataset and four out of five model is successfully trained. The training used Adam optimizer with a learning rate was fixed as 0.001 and gradient clipping was applied to enhance training stability.

# Sampling:

After training, the model can generate new molecules by sampling from the learned distribution. This is typically done by feeding in a starting character or token and recursively predicting the next symbol or token until the end of the molecule is reached

# **Optimization:**

Finally, various methods exist to optimize the generated molecules to improve their drug-like properties or other desired properties. This can involve filtering out molecules that violate specific rules or optimizing the molecular properties using various algorithms.

The BiRNN model had the following structure such as : An embedding layer to convert tokens into vectors, Two layers of Bidirectional LSTM with 512 units each and A final linear layer to predict the next token. The models is trained using CrossEntropyLoss and the Adam optimizer. Gradient clipping was applied to improve training stability. Out of five models, four models are trained successfully. Each model was trained for one full pass over the data.

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# 4.1 Molecule Generation

Molecules were generated one token at a time. It started with the token 'C' and used a temperaturecontrolled sampling method to choose each next token. We generated over 1000 candidate SMILES strings. RDKit [9] was used to check which molecules were valid and to remove any incorrect ones. This process is similar to methods used in tools like ChemVAE and REINVENT [10,11].



# 4.2 DRUG-LIKENESS FILTERING

We calculated the QED score for each generated molecule. Molecules with QED  $\geq$  0.5 were kept. The highest QED score was 0.76, and 73 molecules passed this threshold.

We also applied Lipinski's Rule of Five, which checks molecular weight, LogP, and hydrogen bonding properties. This helps find molecules that are more likely to be effective drugs [12].

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#### 5. FINE-TUNING AND TARGETED GENERATION

#### 5.1 Fine-Tuning on Kinase Inhibitors

To focus the model on kinase inhibitors are the selected a subset of kinase inhibitors from ChEMBL based on their activity, the same preprocessing steps were applied, The BiRNN model was fine-tuned using a smaller learning rate and trained for fewer epochs. After fine-tuning, new molecules were generated, validated, and filtered using the same QED and Lipinski rules. This strategy has been used before in transfer learning studies [13].

#### 5.2 Fine-Tuning on RTK-Specific Compounds

To further specialize the model for RTK targets such as collected RTK-specific molecules using annotations and keywords. The kinase model was further fine-tuned using this RTK-specific dataset and New SMILES were generated, checked for validity, and filtered.

Extra analysis was done using Tanimoto similarity, scaffold similarity, and docking studies. These help assess the novelty and usefulness of the generated compounds in figure 3 and 4 [14].



Figure 4. Valid novel molecule for RTKs and Kinase

#### 6. CONCLUSION

This study shows how BiRNN models can generate new drug-like molecules. Starting with general molecules from ChEMBL, to trained and fine-tuned models to create compounds for rare disease targets.

These AI methods provide a powerful way to find new drugs, especially when data and resources are limited.

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### Solubility Modeling of Some Antibiotic Drugs in Supercritical Carbon Dioxide

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#### ABSTRACT

Solubility modeling of Antibiotics is crucial for optimizing pharmaceutical formulation and enhancing drug delivery system. In this study the solubility of some antibiotics in supercritical carbon dioxide are correlated using Chrastil model, Bartle et al. Model, del Valle and Aguilera model and Improved del Valle and Aguilera model. These empirical models provide thermodynamic information of solubility. Solubility data are correlated with the four models and the results are indicated in terms of absolute average relative difference percentage. The results demonstrate that all the four models effectively describe the solubility behavior, however, the Improved del Valle and Aguilera model providing slightly improved correlation. The better correlation may be attributed to the introduction of new temperature expression that accounts heat of vaporization enthalpy. Additionaly, the Solvation enthalpy for the Antibiotics were estimated.

*Keywords:* Antibiotics, Bartle et al. Model, Chrastil Model, and del Valle and Aguilera Model and Improved del Valle model Supercritical Carbon Dioxide

#### **INTRODUCTION**

In recent years, the application of supercritical carbon dioxide (SC-CO<sub>2</sub>) in pharmaceutical processes has gained significant attention due to its unique properties and environmental advantages. One of its key applications lies in enhancing drug solubility and improving drug delivery systems. Despite ongoing research, the solubility behavior of various antibiotics in SC-CO<sub>2</sub> remains an area with several unexplored aspects. This study focuses on modeling the solubility of selected antibiotics such as Clarithromycin, Clemastine Fumarate, Clindamycin, Erythromycin, and Metronidazole Benzoate in supercritical carbon dioxide. While earlier studies have utilized empirical models like Chrastil, Bartle et al., and the del Valle and Aguilera model, there still exists a need to explore improved correlations that account for thermodynamic influences more accurately.

To address this gap, the present work includes a Improved del Valle and Aguilera model that incorporates five parameters to better capture the temperature dependence of solubility. The inclusion of this model

aims to enhance the precision of solubility predictions, particularly by compensating for the vaporization enthalpy effect. By comparing the performance of all four models, this work contributes valuable insights into selecting the most reliable approach for modeling antibiotic solubility in SC-CO<sub>2</sub>.

#### MATERIALS AND METHODS

#### Chrastil Model (1982)

This model is based on solvate complex formation and relates solute concentration to solvent density and temperature using three parameters.

#### Model Equation,

$$Cm = (\rho m_1)^k \exp\left(A_1 + \frac{B_1}{T}\right) \tag{1}$$

$$y_2 = \frac{\rho_1^k \exp\left(A_1 + \frac{B_1}{T}\right)}{1 + \rho_1^k \exp\left(A_1 + \frac{B_1}{T}\right)}$$
(2)

#### Bartle et al. Model (1991)

It was developed based on the linear relationship between natural logarithm of enhancement factor with density of pure  $Sc-co_2$  by replacing vapour pressure for reference pressure (= 1bar) due to the unavailability of vapour pressure data for solid solute and including intercept at references density (=  $700 \text{kg/m}^3$ ) instead at zero density in order to reduce the error in correlation.

#### Model Equation,

In 
$$\left|\frac{y_2 P}{P_{ref}}\right| = A_2 + \frac{B_2}{T} + C_2(\rho - \rho_{ref})$$
 (3)

#### del Valle and Aguilera model (1999)

This model was developed for predicting the solubility of vegetable oil in compressed  $co_2$  as a function of the solute's heat of vaporization with temperature. This is the improved equation of Chrastil model.

#### Model Equation,

$$\ln C = k \ln \rho + \frac{a}{T} + \frac{b}{T^2} + c \qquad (4)$$

Local values were evaluated as

H = R(b+2c/T)

#### Improved del Valle andd Aguilera (2025)

However, the del Valle and Aguilera Model shows limitations when applied over a wide range of temperatures. To overcome these limitations, a modified form of the model has been proposed in this study to enhance accuracy and improve correlation with experimental data.

This improved model shows better relationship between solubility, temperature, and density. This is the improved equation of del Valle and Aguilera.

#### Model Equation,

In C = k In(
$$\rho$$
) + a +  $\frac{b}{T}$  + c In(T) +  $\frac{d}{T^2}$  (5)

Local (T) Values were evaluated as

$$H = R \ (b - c T + \frac{2d}{T})$$

#### METHODOLOGY

Five Antibiotic compounds namely Clarithromycin, Clemastine Fumarate, Clindamycin, Erythromycin, and Metronidazole Benzoate are considered for the modeling. Experimental data details are summarized in Table 1. Nonlinear regression is performed with L-M algorithm (using Polymath software). The model equation (2),(3),(4) and (5) were regressed with solubility data and the regression results are tabulated in Table 2, Table 3 and Table 4. A higher R<sup>2</sup> value, nearing 1, signifies the model's proficiency in closely predicting data points compared to experimental values.

#### **Coefficient of Regression**,

$$R^{2} = 1 - \frac{\sum_{i=1}^{N_{i}} (y_{2}^{exp} - y_{2}^{cal})^{2}}{\sum_{i=1}^{N_{i}} (y_{2}^{exp} - y_{2}^{cal})^{2}}$$
(6)

To improve the precision of evaluating models with varying numbers of curve fitting parameters, the Average Absolute relative Deviation Percentage with respect to the adjustable parameters (AARDZ) is computed. This criterion offers a more accurate measure for comparing the accuracy of models by considering the average absolute differences between predicted and actual values across the datasets. Equation,

$$AARDZ\% = \frac{100}{N-Z} \sum_{i=1}^{i=N} \left( \frac{y_{exp,i} - y_{pre,i}}{y_{exp,i}} \right)$$
(7)

#### **RESULTS AND DISCUSSION**

As stated earlier the Antibiotic drugs considered in the present study are Clindamycin, Clarithromycin, Clemastine fumarate, Erythromycin, and Metronidazole benzoate. These compounds represent various classes of antibiotics such as Lincosamides, Macrolides, Antihistamines with Antimicrobial activity, and Nitroimidazoles. Detailed information on each antibiotic, including molecular weight, number of solubility data points, solubility range in mole fraction, temperature range in K, and pressure range in bar, is presented in the below Table 1. The Tables 6 & 7 indicates the Calculated thermodynamic quantities such as heat of reaction(obtained from Chrastil model constant), heat of vaporization (obtained from del Valle and Aguilera and improved del Valle and Aguilera model). The formulas used in the calculations are indicated the table. It is important to note that the values obtained are in joule/mole. But the reported values are in kJ/mol

Table 1. Data Range of Compounds Considered in This Work.

S.No	Binary System	Molecular Weight (kg/ kmol)	Solubility Range ×10-⁵	Temperature Range (K)	Pressure Range (bar)	No. of Data Points	Reference
1	Claindamycin-SC- CO₂	275.26	7–45.5	308-348	122-355	32	4
2	Clarithromycin- SC-CO₂	460.0	0.161-0.941	308-338	120-270	24	4
3	Erythromycin-SC- CO <sub>2</sub>	733.9	4.3-31.2	308-348	122-355	45	4

4	Clemastine fumarate-SC-CO₂	425.0	17.1–114.6	308-348	122-355	45	5
5	Metronidazole Benzoate-SC-CO₂	748.0	13.1-32.8	308-348	122-355	45	6

#### Table 2. Model Parameters and Their corresponding AARDZ% Values for Bartle et al. Model

Compound	а	b	с	R <sup>2</sup>	AARDZ %
Clindamycin	14.6324	-5657.86	0.00632	0.96	9.608
Clarithromycin	8.0766	-3733.73	0.00495	0.82	9.623
Clemastine fumarate	22.3831	-6665.92	0.00749	0.78	25.194
Erythromycin	12.3617	-5378.6	0.00787	0.95	9.695
Metronidazole Benzoate	24.072957	-8709.434	0.0119822	0.96	19.925
Global Value				0.89	14.809

#### Table 3. Model Parameters and Their Corresponding AARDZ% Values for Chrastil Model

Compound	k	а	b	R <sup>2</sup>	AARDZ%
Clindamycin	2.1070853	-10.852871	-3744.4019	0.97	7.773
Clarithromycin	1.1369422	-11.156705	-1790.4356	0.96	3.608
Clemastine fumarate	2.6286091	-7.9370159	-4264.8661	0.83	21.772
Erythromycin	3.0155325	-19.379556	-3350.1973	0.96	9.695
Metronidazole Benzoate	6.2342369	-28.775969	-6675.5545	0.95	21.679
	Global va	0.93	12.905		

## Table 4. Model Parameters and Their Corresponding AARDZ% Values for delValle as Aguilera Model

Compound	k	а	b	с	R <sup>2</sup>	AARDZ %
Clindamycin	2.1094008	-8.578367	-5266.33	2.525	0.97	7.78
Clarithromycin	1.1342992	-29.73268	10430	-2.0E	0.98	3.071
Clemastine fumarate	2.6244842	-55.60255	20700	-4.0E	0.85	2.10
Erythromycin	2.9982695	-10.54821	-9106.41	9.49	0.96	7.18
Metronidazole benzoate	6.0386908	-22.47435	-1.0	5.867	0.96	21.31
	lue		0.94	12.06		

Compound	к	с	а	b	d		AARDZ%
Clindamycin	2.189	1.835	2-23.520	-2.68	3.809	0.98	4.854
Clarithromycin	16.1628	1.6539	-33.219	1.034	-1.9	0.98	3.072
CClemastine Fumarate	58.508	6.58502	-51.3245	9900.47	-2.3	0.93	15.40
Erythromycin	3.8879	4.14636	-15.103	-1.01	1.10	0.98	6.036
Metronidazole Benzoate	6.6616	7.1198	-13.45	-1.88	2.02	0.97	16.61
Global value							9.19

## Table 5. Model Parameters and Their Corresponding AARDZ% Values forImproved del Valle and Aguilera Model

## Table 6. Calculated Heat of Reaction ,Sublimation Enthalpy and solvation Enthalpy (Estimated using del Valle and Aguilera model and Chrastil model combination)

Compound	ΔH <sub>RXN</sub> = -bR R (kJ/mole)	$\Delta H_{SUB} = R(b+(2(c)/T))$ (kJ/mole)	$\Delta H_{SOLVATION} = (\Delta H_{SUB} - \Delta H_{RXN})$ (kJ/mole)
Clindamycin	31.131	-30.983	-62.114
Clarithromycin	14.886	-14.928	-29.814
Clemastine fumrate	35.458	-172.7393	-208.197
erythromycin	27.853	-27.575	-55.429
Metronidazole Benzoate	55.501	-54.228	-109.729

## Table 7. Calculated Heat of Reaction, Sublimation Enthalpy and Solvation Enthalpy (Estimated using Improved del Valle and Aguilera model and Chrastil model combination)

Compound	ΔH <sub>RXN</sub> = -bR (kJ/mole)	$\Delta H_{SUB} = R(b-c(T)+(2(d)/T)$ (kJ/mole)	$\Delta H_{SOLVATION} = (\Delta H_{SUB} - \Delta H_{RXN})$ (kJ/mole)
Clindamycin	31.131	-34.721	-65.853
Clarithromycin	14.886	-14.864	-29.749
Clemastine fumrate	35.458	-124.49	-159.947
erythromycin	27.853	-39.514	-67.367
Metronidazole Benzoate	55.501	-73.315	-128.815

#### CONCLUSION

In this work, a new equation for solubility correlation is proposed based on the concept of temperature compensation for the vaporization enthalpy using a Improved del Valle and Aguilera Model with five parameters. The proposed solubility model is compared with the existing Chrastil model, Bartle et al. model, and the del Valle and Aguilera model by considering five drug compounds' solubility data in supercritical carbon dioxide (SCCO<sub>2</sub>).

The newly proposed model demonstrates a strong correlation ability compared to existing models, as evidenced by its favorable Global AARDZ of 9.19 % and Global R<sup>2</sup> of 0.96. For the Improved del Valle and Aguilera Model the calculated sublimation enthalpy values are observed.

Moreover, the Improved del Valle and Aguilera model was also employed to estimate the solvation enthalpy of the selected antibiotics. The estimated solvation enthalpy provides valuable thermodynamic insight into the solute–solvent interactions, further reinforcing the applicability of the model for solubility prediction in SCCO<sub>2</sub> systems.

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### Medicinal Leaves and its Health Benefits-A Critical Review

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#### ABSTRACT

Medicinal leaves have long played a pivotal role in traditional and modern healthcare systems, offering a natural source of therapeutic compounds. This critical review explores the medicinal potential and health-promoting effects of five widely used leaves: Papaya (Carica papaya), Eucalyptus (Eucalyptus globulus), Brahmi (Bacopa monnieri), Betel (Piper betle), and Moringa (Moringa oleifera) – highlighting their traditional uses and scientific evidence supporting their efficacy. Papaya leaves are extensively used in traditional medicine for their anti-inflammatory, antimalarial, and platelet-boosting properties, particularly in the treatment of dengue fever. Eucalyptus leaves are rich in essential oils, particularly eucalyptol, which are effective in treating respiratory conditions, acting as decongestants and antibacterial agents. Its essential oils have found applications in aromatherapy and topical treatments. Brahmi leaves (Bacopa monnieri), a staple in Ayurvedic medicine, are recognized for their nootropic effects, enhancing memory, cognitive function, and reducing anxiety through antioxidant and neuroprotective mechanisms. Betel leaves, commonly used in Southeast Asian culture, possess antimicrobial, antifungal, and anti-diabetic properties, but their long-term use – especially with tobacco – warrants cautious consideration due to potential carcinogenic risks. Moringa leaves (Moringa oleifera), often termed a "superfood," are a powerhouse of vitamins, minerals, and bioactive compounds that exhibit anti-inflammatory, antidiabetic, and cardioprotective effects. Moringa has shown potential in addressing malnutrition and metabolic syndromes in various global health contexts. This review critically evaluates the benefits and limitations of these medicinal leaves, emphasizing the need for further clinical research to validate traditional claims, understand dosage safety, and isolate active compounds.

*Keywords:* Medicinal Leaves, Papaya, Eucalyptus, Brahmi, Betel, Moringa, Phytochemicals, Health Benefits, Traditional Medicine

#### INTRODUCTION

Medicinal Leaves are a limitless supply of pharmaceuticals, chemical entities, traditional and modern medical systems, and traditional treatments. Approximately 80% of the world's population relies only on plants for their basic medical needs. It has been reported that over 45,000 plant species in India have therapeutic qualities. Natural products or molecules derived from plants have demonstrated significant advantages over synthetic medications, including low cost, easy availability, and little adverse effects. The use of medicinal leaves to treat a variety of diseases has been the subject of numerous published research.[1] Some of the medicinal leaves with major health benefits include Papaya Leaves, Brahmi Leaves, Eucalyptus Leaves, Betel Leaves and Moringa Oleifera leaves. Papaya leaves have long been used in traditional medicine due to their various medicinal properties. Papaya leaves contain Vitamins (A, C, E), minerals (calcium, magnesium, sodium, and iron) enzymes (papain, chymopapain), and antioxidants. Papaya leaves are sometimes used to treat skin conditions like acne and eczema due to their antimicrobial and anti-inflammatory properties. Papaya leaves are particularly known for their ability to increase platelet count, which is helpful in treating dengue fever. Eucalyptus leaves are renowned for their wide array of medicinal benefits, making them a popular choice in traditional and modern remedies alike. Eucalyptus is particularly valued for its anti-inflammatory, antibacterial, and antiviral effects, often used to relieve respiratory ailments such as coughs, colds, and sinus congestion. Brahmi, scientifically known as Bacopa monnieri, is a perennial herb commonly found in wetlands and known for its numerous medicinal properties, especially in traditional Ayurvedic medicine. Revered for centuries, brahmi leaves are characterized by their small, oval shape and succulent texture. They are celebrated for their cognitiveenhancing abilities, often referred to as a "brain booster." Betel leaves, derived from the plant Piper betle, hold a prominent place in traditional medicine across Asia, especially in Ayurvedic and Siddha practices. These heart-shaped, glossy green leaves are not only culturally significant but also packed with an array of health-enhancing properties. For centuries, betel leaves have been employed to promote oral hygiene, support digestion, and alleviate respiratory issues. Moringa leaves, derived from the Moringa oleifera tree, are widely regarded as one of the most nutrient-dense plants on earth. Often referred to as the "drumstick tree," "miracle tree," or "horseradish tree," Moringa is native to the Indian subcontinent but is now cultivated globally in tropical and subtropical regions. Moringa leaves are small, green, and ovalshaped, growing in clusters on slender branches. They are known for their incredible nutritional profile, including high levels of vitamins (A, C, and E), minerals (calcium, potassium, and iron), proteins, and antioxidants.

#### PAPAYA LEAVES

The article reviews the potential of Carica papaya leaf extract as a high-value commodity in terms of its health effects and industrial benefits, and highlights the traditional use of the extract to treat fevers caused by viral infections like dengue, malaria, and chikungunya.[2] Papaya leaf extract contains various phytochemicals like flavonoids, alkaloids, and cyanogenic glycosides, which have been found to have antiviral, antimalarial, and anti-inflammatory properties.[3] Papaya leaf extract increases platelet count in dengue fever patients, as well as it has antibacterial, gastro-protective, and anti-sickling properties. [1] Another study investigates the antioxidant and cytotoxic activities of various extracts from papaya leaves, traditionally used for treating diarrhea. This Study shows that nonpolar extracts exhibit the most significant biological activity, suggesting their potential as traditional medicine and chemotherapy agents. The main thesis is that these extracts can be utilized in traditional medicine and as adjuncts in cancer treatment due to their biological properties. [4] Another study investigated the antidiabetic effects of ethanolic extract of Carica papaya leaves in alloxan-induced diabetic rats, demonstrating significant reductions in blood sugar levels and improvements in lipid profiles. Additionally, the extract improved lipid profiles, evidenced by reduced total cholesterol, triglycerides, and low-density lipoprotein (LDL) cholesterol, while increasing high-density lipoprotein (HDL) cholesterol.[5]

#### EUCALYPTUS LEAVES

Eucalyptus camaldulensis is a valuable medicinal plant with significant antimicrobial properties. Eucalyptus camaldulensis, also known as the river red gum, is a tree belonging to the Myrtaceae family that is widely distributed and cultivated worldwide.[6] The review article discusses the cytotoxic properties of essential oils (EOs) from different Eucalyptus species, which have been reported to induce cell death, inhibit cell proliferation, and cause apoptosis in various cancer cell lines. The EOs of Eucalyptus sideroxylon, E.torquata, and E. globulus have shown significant cytotoxicity against MCF7 breast cancer cells. Eucalyptus-derived EOs contain a wide variety of secondary metabolites, such as phenolics, terpenoids (monoterpenes, sesquiterpenes, diterpenoids, and triterpenoids), which have demonstrated anti-tumor and cytotoxic activities against different cancer cell lines.[7][8] Eucalyptus is a rich source of phytochemicals with diverse health benefits, and its essential oil has various applications in the food, pharmaceutical, and cosmetic industries[9] In anti-cancer tests, the extract significantly inhibited the growth of various cancer cell lines, including pancreatic, colon, lung, and breast cancers. These findings highlight Eucalyptus robusta extract as a promising candidate for further refinement and development as a natural antioxidant and potential anti-cancer agent, especially for pancreatic cancer treatment.[10]

#### **Brahmi Leaves:**

Since ancient times, Brahmi, also known as Bacopa monnieri, has been used as a nootropic ayurvedic plant to treat neurological conditions. Many strategies, including both synthetic and natural chemicals, have been used to fight Alzheimer's disease. There have been numerous reports of neuroprotective herbs in Ayurveda. Since ancient times, Brahmi, also known as Bacopa monnieri, has been used as a nootropic in Ayurvedic medicine to treat neurological conditions.[11] The primary active compounds in Brahmi are bacosides, particularly bacoside A, which have been extensively studied for their health benefits. Brahmi is known to improve cognitive function, reduce anxiety and stress, and possess anti-inflammatory and antioxidant properties. It has also been linked to improved respiratory health, enhanced immunity, and potential benefits for skin care and blood sugar regulation. [12] In recent research, the development of a health mix incorporating Brahmi leaves aims to address the high incidence of undernutrition among children in India by enhancing their nutritional status and cognitive function.[13] Brahmi leaves are a powerhouse of valuable alkaloids and triterpene saponins that can stimulate brain chemicals for sharper thinking, memory, and learning. Herb contained alkaloids of brahmine and herpestine. [14] Brahmi leaves are a powerhouse of valuable alkaloids and triterpene saponins that can stimulate brain chemicals for sharper thinking, memory and learning. It has been observed that the extracts of brahmi plants can be a helpful component in the creation of herbal beverages that contain the phenolics as well as Vitamin C as antioxidant. Without the addition of chemicals like sweeteners, artificial flavours, or colours, they can be utilized as a base to create beverages.[15]

#### **Betel Leaves:**

Betel leaf (Piper betel Linn.) is a perennial, evergreen creeper of the Piperaceae family, valued for its glossy, heart-shaped leaves rich in phenolic compounds. Betel leaves contain bioactive compounds such as: Chavibetol, Chavibetol acetate, Safrole, Eugenol, Caryophyllene, Hydroxychavicol. These constituents contribute to various pharmacological properties like antioxidant, antibacterial, and anti-inflammatory activities.[16] Red betel leaves contain phenolic chemicals called luteolin and apigenin. Apigenin and luteolin derivatives are cytotoxic to cancerous cells. Methanolic red betel leaf extracts are utilized to assess the cytotoxic and anti-migration effects on breast cancer that has spread. It is commonly recognized that betel leaf extract can control blood glucose levels and has strong anti-diabetic effects.[17] Betel Leaf essential oil can therefore be used for active food packing as well. In addition, the distinct spicy aroma might improve the flavour of select specific foods.[18] Strong evidence supports betel leaves exceptional chemotherapeutic and chemopreventive potential against a range of cancer forms. According to reports, piper betel is a treasure trove of bioactive phenolics that have the ability to combat malignancies of the skin, stomach, prostate, breast, and mouth.[19] According to Ayurveda, eating betel leaf right after a

meal is healthy since it aids in digestion, clears the mouth, gets rid of persistent coughing, and helps people maintain their weight. Because of its diuretic properties, betel leaf juice is used to treat obstructed urination. Cough and sore throat can be effectively treated at home using betel leaves. To ease coughing and breathing difficulties, apply the mustard oil-soaked leaves to the chest. Betel leaves are useful in the treatment of debility, nerve fatigue.[20]

#### Moringa Oleifera:

Moringa oleifera, often referred to as the "Miracle Tree," is renowned for its wide range of medicinal benefits, including anti-inflammatory properties due to its rich composition of vitamins and antioxidants. Indomethacin, a non-steroidal anti-inflammatory drug (NSAID), is commonly utilized for pain relief in chronic inflammatory disorders, but its administration is frequently associated with adverse gastrointestinal effects, including the formation of gastric ulcers. The use of herbal supplements, such as Moringa oleifera, has been explored as a promising alternative or adjunctive therapy due to fewer side effects compared to conventional ulcer treatments like proton pump inhibitors and antacids.[21] Moringa oleifera leaf extract, showed significant efficacy in reducing skin erythema without causing irritation. The M. oleifera leaf extract cream was not irritant according to 48 h semi-occluded patch test. There was a significant decrease in skin erythema.[22] Moringa oleifera leaf extracts demonstrate significant potential in alleviating depression among rheumatoid arthritis patients, as evidenced by improvements in Beck Depression Inventory scores and serum cortisol levels. Moringa oleifera is a complementary therapy to enhance mental health outcomes in chronic disease management.[23] Moringa oleifera leaf extract (MoLE) demonstrates significant hepatoprotective effects against high-fat diet-induced liver injury in mice, primarily through its antioxidant properties. MoLE treatment significantly protects against liver damage, as evidenced by improved liver histology and reduced serum levels of liver enzymes such as AST, ALT, and ALP. The extract also enhances antioxidant status, indicated by increased ferric reducing antioxidant power (FRAP) and reduced glutathione (GSH) levels, while decreasing lipid peroxidation. [24] The Moringa oleifera is a multi-purpose herbal plant that has been used for medicinal purposes worldwide.[25]

#### CONCLUSION

Papaya, Betel, Moringa, Brahmi, and Eucalyptus leaves each offer a unique array of health benefits, making them powerful components of natural and traditional medicine. Papaya leaves are rich in enzymes like papain and chymopapain that aid digestion and have been widely studied for their ability to boost platelet count, especially in dengue treatment. Betel leaves, traditionally used in various Asian cultures, exhibit strong antimicrobial, antioxidant, and anti-inflammatory properties, and may support oral health, digestion, and respiratory wellness. Moringa leaves, often referred to as a superfood, are densely packed with essential nutrients, including vitamins A, C, and E, iron, calcium, and amino acids, offering benefits such as improved immunity, anti-diabetic effects, and reduced inflammation. Brahmi leaves, a staple in Ayurvedic medicine, are known for enhancing cognitive functions, reducing anxiety and stress, and supporting mental clarity and memory through active compounds like bacosides. Lastly, Eucalyptus leaves possess potent antiseptic and anti-inflammatory qualities, with their essential oil being particularly effective for respiratory ailments, pain relief, and improving immune response. Together, these leaves represent a potent synergy of nature's healing arsenal, promoting holistic well-being across multiple bodily systems when used responsibly and in moderation.

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### Comprehensive Review on Exploring the Medicinal Potential of Fruits

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#### ABSTRACT

Fruits are not only essential components of a balanced diet but also serve as potent natural sources of therapeutic agents. This review highlights the medicinal benefits of five nutritionally rich fruits: pomegranate (Punica granatum), amla (Phyllanthus emblica), orange (Citrus sinensis), Indian blackberry (Syzygium cumini), and dates (Phoenix dactylifera). Each of these fruits is abundant in bioactive compounds that contribute to various healthpromoting effects. Pomegranate is renowned for its antioxidant, anti-inflammatory, and anticancer properties, largely attributed to polyphenols such as punicalagins and ellagic acid. Regular consumption has shown to reduce the risk of cardiovascular diseases and improve memory and cognition. Amla, also known as Indian gooseberry, is a rich source of vitamin C and tannins. It exhibits strong antioxidant, anti-aging, and immune-boosting effects, and is used in traditional medicine for managing diabetes, hyperlipidemia, and digestive disorders. Oranges are excellent sources of vitamin C, flavonoids, and dietary fiber. They support immune function, improve skin health, and help in the prevention of chronic diseases such as hypertension and cardiovascular disorders. Indian blackberry or jamun contains anthocyanins, flavonoids, and ellagic acid, offering antidiabetic, hepatoprotective, and gastroprotective benefits. It is especially valued in traditional medicine for its role in blood sugar regulation. Dates, rich in natural sugars, fiber, and essential minerals such as potassium and magnesium, are recognized for their energy-boosting, anti-inflammatory, and neuroprotective properties. They support digestive health and have been associated with reduced risk of chronic conditions like anemia and constipation. Collectively, these fruits offer a wide range of medicinal benefits due to their high nutritional density and presence of phytochemicals. Integrating them into daily diets not only promotes general well-being but also contributes to the prevention and management of various health disorders, making them vital components of functional nutrition and natural medicine.

Keyword: Pomegranate, Amla, Orange, Indian Blackberry, Dates, Bioactive Compounds

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#### INTRODUCTION

Fruits are celebrated not only for their nutritional value but also for their therapeutic potential in promoting health and preventing disease. This paper highlights five fruits – pomegranate, amla, orange, Indian blackberry, and dates – that are especially noted for their medicinal properties due to their rich content of vitamins, minerals, antioxidants, and phytochemicals. Pomegranate (Punica granatum) is rich in polyphenols and known for its strong antioxidant and anti-inflammatory effects, contributing to cardiovascular health, cancer prevention, and cognitive function. Amla (Phyllanthus emblica), one of the richest sources of vitamin C, is widely used in Ayurveda for its immune-boosting, anti-aging, and antidiabetic benefits. Orange (Citrus sinensis) is a popular fruit high in vitamin C and flavonoids, supporting immunity, skin health, and cardiovascular function through its antioxidant actions. Indian blackberry (Syzygium cumini), or jamun, contains compounds like anthocyanins and ellagic acid, which are effective in diabetes management and improving liver and digestive health. Dates (Phoenix dactylifera) are valued for their natural sugars, fiber, and minerals, offering quick energy and promoting digestive and neuroprotective health. These fruits embody the concept of "food as medicine," Promoting their regular consumption can enhance public health and support plant-based medicinal approaches.

#### 1. Pomegranate

Pomegranate (Punica granatum L.), an ancient fruit tree native to the Mediterranean region, has been celebrated for its unique nutritional and medicinal properties for centuries. The anti-cancer properties of pomegranate, which are attributed to its high phenolic content and remarkable antioxidant activity. Ellagic acid is particularly noted for its effectiveness in combating oxidative stress, thereby reducing the risk of cancer and other chronic diseases. The fruit also contains essential minerals and vitamins, contributing to overall health. Pomegranate has the role in treating conditions such as inflammation, rheumatism, and digestive issues, showcasing its versatility as a natural remedy. [1] Pomegranates may play a significant role in preventing and treating serious health conditions such as cancer, cardiovascular diseases, and diabetes. The fruit's high antioxidant content and anti-inflammatory properties contribute to its effectiveness in promoting health and mitigating disease. pomegranate juice can significantly reduce risk factors for heart disease, including lowering LDL cholesterol levels and improving blood flow. The juice has also demonstrated the ability to inhibit the proliferation of cancer cells, particularly in prostate cancer, by disrupting their attraction to signals that promote metastasis. It can improve blood glucose levels and lipid profiles. Its parts, including the rind and seeds, are utilized in various forms to address digestive disorders, respiratory issues, and even skin ailments. The astringent properties of pomegranate are traditionally employed to treat conditions like diarrhea and dysentery, showcasing its versatility as a medicinal fruit [2]. Pomegranate juice (PJ) is a healthy drink, focusing on its high polyphenol content and health benefits. PJ's potential health benefits are reducing inflammation, fighting germs, helping with cancer, and improving metabolism, which can assist in managing obesity, diabetes, and heart health. PJ works in the body, especially through its metabolites like urolithins that come from ellagitannins [3].

#### 2. Amla

Amla has been shown to have antioxidant, anti-inflammatory, antimutagenic, and immune modulatory properties. It has demonstrated beneficial effects in preventing and reducing age-related changes, such as oxidative stress and cellular damage. Amla extracts have shown potential in inhibiting the growth of cancer cells and promoting apoptosis. It has been found to have protective effects on the cardiovascular system and can reduce oxidative stress in the kidneys. Amla extracts have also shown potential in delaying the onset of diabetic cataracts and promoting skin health [4]. Amla has the use in various ayurvedic formulations and traditional remedies. Outcomes include the treatment of various ailments and diseases such as cough, cold, fever, stomachache, dyspepsia, diarrhea, diabetes, peptic ulcers, acidity, jaundice, inflammation, and others. The fruit of Phyllanthus emblica is considered a powerful Rasayana in Ayurveda and is used to treat various diseases. The plant's chemical constituents are associated with diverse pharmacological properties such as antioxidant, anti-inflammatory, antidiabetic, hepatoprotective, antimicrobial, neuro

protective, immune -modulatory, and antitussive properties. The traditional uses of the plant include treating liver disorders, diabetes, respiratory disorders, skin disorders, and hair greying [5]. Emblica officinalis (EO) is an important herb in the Indian traditional medicine system, especially Ayurveda. EO is known for its medicinal properties and is considered one of the most useful drug treatments in the Indian pharmacopoeia. The fruit of EO is rich in quercetin, phyllemblic compounds, gallic acid, tannins, flavonoids, pectin, and vitamin C. EO has many uses, including antioxidant, anticarcinogenic, antitumor, anti-inflammatory, and many other traditional uses. EO is widely used in the Ayurvedic medicines and is believed to increase defense or immune power against diseases [6].

#### 3. Orange

Citrus sinensis wastes are rich in bioactive compounds, such as polyphenols, flavonoids, and essential oils. Various extraction techniques can be used to obtain these bioactive compounds from the waste materials. The bioactive compounds in Citrus sinensis wastes have significant health benefits, including anti-microbial, anti-inflammatory, anti-cancer, and neuroprotective effects. The utilization of Citrus sinensis wastes in various industries can contribute to the economic value of these by-products. The bioactive compounds in Citrus sinensis wastes have significant health benefits, including anti-microbial, anti-inflammatory, anti-cancer, and neuroprotective effects [7]. Citrus waste contains a range of highly beneficial bioactive compounds with antimicrobial and antioxidant properties. Different extraction methods yield varying amounts of bioactive compounds. Modern extraction techniques offer higher yields and lower solvent and energy consumption. The separation and isolation of individual bioactive compounds can be achieved through various purification methods. The utilization of citrus waste in the food and pharmaceutical industries can lead to the production of valuable co-products [8]. Orange peels and pomace are a sustainable raw material that can be used to make valuable products for nutraceuticals. Pectin, phenolics, and limonene found in orange peels and pomace have been linked to various health benefits. Green extraction methods, such as MAE, UAE, ScCO2 extraction, and SWE, can be used to valorize orange peels and pomace and obtain high quantities and qualities of extracts [9].

#### 4. Indian Blackberry

Jamun fruit (Syzygium cumini L.) is an underutilized fruit crop of India with impressive nutritional profile and several health benefits. The fruit is known for its attractive color, astringent taste, and nutraceutical properties. It is used for the treatment of diabetes and possesses anti-cancer, anti-inflammatory, antiobesity, and cardio-protection properties. Investigations were done on the production of value-added food products such as ready-to-serve beverages, jam, cookies, and cake from jamun due to their several health benefits [10]. Java plum extract contains a variety of anthocyanins, including glucosides of delphinidin, cyanidin, petunidin, peonidin, and malvidin JPE suppressed proliferation in HCT-116 cells and induced apoptosis in both HCT-116 cells and colon cancer stem cells JPE also suppressed the self-renewal ability of colon cancer stem cells. JPE exhibits anti-cancer activity against colon cancer cells and colon cancer stem cells [11]. Jamun is considered a traditional medicine for controlling diabetes, with a specific action on the pancreas. The fruit, seeds, and juice of jamun play important roles in the treatment of diabetes. Jamun seeds contain Jamboline, a type of glucose that helps regulate sugar levels. Jamun has anti-cancer and anti-viral properties. The bark of the black berry tree has astringent, digestive, and diuretic properties. The fruit is also considered stomachic, carminative, antiscorbutic, and diuretic [12].

#### 5. Dates

Date palm has been traditionally used for various purposes, including as a general tonic, for the treatment of liver diseases, memory disturbances, fever, inflammation, paralysis, and nervous disorders. Date palm is a complete diet with high nutritional value and contains a wide range of secondary metabolites. Date palm possesses various pharmacological effects, including anticancer, antidiabetic, anti-inflammatory, antimicrobial, antioxidant, cardiovascular, gastrointestinal, and wound healing effects [13]. Date paste, made from grinding pitted dates, is rich in sugars, dietary fiber, and natural antioxidants, making it a valuable natural alternative to refined sugar. Date paste enhances the flavor, texture, and overall sensory

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properties of food products. Date paste offers numerous health benefits, including protection against diseases, regulation of blood sugar, and support for heart health [14]. Date seeds contain a significant amount of oil (5-13%). The main fatty acids in date seed oil are oleic acid and lauric acid Date seed oil contains valuable phytochemicals such as tocols, phytosterols, and phenolic compounds Different extraction techniques can be used to obtain date seed oil, including conventional Soxhlet extraction, supercritical fluid extraction (SFE), and ultrasound-assisted extraction (UAE). Date seed oil has various applications in the food, cosmetic, and pharmaceutical industries [15].

#### 6. RESULT AND DISCUSSION

The analyzed fruits demonstrate strong medicinal properties which stem from their biochemical components. Pomegranate alongside amla possess antioxidants and anti-inflammatory components which work to improve heart health and system metabolism according to research [1][3][4][5]. The compounds obtained from orange peel show both antimicrobial properties and provide protection to the nervous system [7][9]. The compounds anthocyanins and jamboline present in Indian blackberry show anticancer and antidiabetic properties. Dates promote digestive health together with neuroprotective benefits because they contain essential oils along with phenolics and fiber substances [13][15]. These fruits prove that food acts as medicine which strengthens their value for dietary use as preventive measures against diseases and promotors of health.

#### CONCLUSION

Fruits are rich in vitamins, minerals, antioxidants, and bioactive compounds, they support overall health. Pomegranate and amla have potent antioxidant and anti-inflammatory effects that boost cardiovascular and immune function. Orange enhances immunity and skin health due to its high vitamin C content. Indian blackberry aids in diabetes management and liver health, while dates provide natural energy, support digestion, and protect the nervous system. Regular consumption of these fruits can help prevent chronic conditions like diabetes and hypertension. Ongoing research continues to validate their therapeutic potential.

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### Biosynthesis of Zinc Oxide and Silver Nanoparticles from Cassia Alata: A Comparative Study on Antimicrobial and Structural Properties

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#### ABSTRACT

The growing demand for sustainable and eco-friendly nanomaterials has led to increased interest in plant-mediated synthesis of nanoparticles. This study explores the biosynthesis of silver (AgNPs) and zinc oxide nanoparticles (ZnONPs) using Cassia alata, a medicinal plant rich in flavonoids, tannins, and phenolics that act as natural reducing and stabilizing agents. Aqueous leaf extracts were used to synthesize AgNPs and ZnONPs under controlled conditions, with successful formation confirmed through visual observation and UV-Vis spectroscopy. Structural and morphological characterization was performed using FTIR, XRD, SEM, and TEM, revealing well-defined nanoparticles with spherical and rod-like shapes. Antimicrobial studies against Escherichia coli, Staphylococcus aureus, and Pseudomonas aeruginosa demonstrated effective inhibition, with AgNPs exhibiting higher potency, while ZnONPs displayed greater stability and biocompatibility. ROS generation and minimum inhibitory concentration (MIC) tests further validated the antimicrobial mechanism. Despite AgNPs' superior activity, ZnONPs are favored in biomedical and environmental applications due to lower cytotoxicity. The study underscores the versatility of Cassia alata in nanoparticle synthesis and provides a comparative perspective on two widely used nanomaterials. It also highlights the importance of standardization and further in vivo studies for future clinical translation.

**Keywords:** Cassia Alata, Green Synthesis, Silver Nanoparticles (AgNPs), Zinc Oxide Nanoparticles (ZnONPs), Antimicrobial Activity, Phytochemicals, Biosynthesis, Biocompatibility, Nanotechnology, Structural Characterization

#### 1. INTRODUCTION

Nanotechnology serves as a emerging field across various sectors which includes medicine, agriculture, energy, and environmental science. Their ability to modify and manipulate the properties of matter at the nanoscale has eventually resulted in the innovation of many nanomaterials which has an enhanced physical, chemical, and biological properties. Apart from all the other different types of nanomaterials so far discovered, metal based nanomaterials like silver nanoparticles (AgNPs) and zinc oxide nanoparticles

(ZnONPs), are considered to be the most important nanoparticles because of their extraordinary antimicrobial, optical, and catalytic activities. The conventional way of producing and preparing these nanomaterials require the involvement of lots of toxic chemicals, high energy inputs, and environmentally harmful by-products. Due to the raising limitations because of their hazardous effects greener alternatives are emerging which helps in aligning the principles of sustainability, eco-friendly and environmental safety. As a result plant extracts are being used as an eco friendly alternative in the green synthesis production of nanoparticles which serves as a cost-effective, and scalable approach. To reduce the metal ions and stabilize nanoparticles, phytochemicals present in the plants where used like flavonoids, alkaloids, terpenoids, and phenolics. Cassia alata is one such plant which is also known as candle brush is widely grown in tropical regions.this plant is commonly known for its superior medical properties like antimicrobial, antifungal, and antioxidant effects. As this plant is rich in bioactive compounds it is used in the biosynthesis of nanoparticles. Apart from producing nanoparticles, this plant helps in improving the stability and biological activity as they are natural reducing and capping agents.

This review aoms in exploring the biological synthesis of silver nanoparticles and zinc oxide nanoparticles using Cassia alata, by emphasising and making a comparative study of their structural characteristics, antimicrobial activities, and potential applications. This paper provides a consolidated comprehensive study based on the efficacy and versatility of Cassia alata-mediated nanoparticles, also analysing their future applications in the field of research and in sustainable nanotechnology.

#### 2. MEDICINAL AND PHYTOCHEMICAL PROPERTIES OF CASSIA ALATA

Cassia alata is one of the commonly known tropical medicinal shrub, also called as candle bush or ringworm plant which is known to be widely recognized as it has superior therapeutic potential and also highly rich phytochemical properties. This plant is mostly preferred in Ayurvedic and folk medicinal chemistry. This plant is highly effective in treating different types of diseases and infections, for instance, skin infections, fungal diseases, constipation, and inflammatory conditions. Cassia alata stems has high medicinal value due to the presence of large and diverse variety of bioactive phytochemicals which are naturally present in them. The commonly known different classes kf various phytochemicals include flavonoids, tannins, saponins, alkaloids, anthraquinones, and terpenoids. The above mentioned compounds have high levels of antioxidant, antimicrobial, anti-inflammatory, and wound-healing properties, which makes them a well known, significant source of natural therapeutics in curing various diseases. In the synthesis of nanoparticles, these phytochemicals play a very crucial role as both reducing and stabilizing agents. For example, flavonoids and tannins reduces them back to their nanoscale metallic or oxide forms as these phytochemicals are capable of donating electrons to metal ions (such as  $Ag^+$  and  $Zn^{2+}$ ). Also at the same time these compounds act as capping agents, which helps in binding to the nanoparticle surface and eventually helps in preventing agglomeration. Apart helping in stabilizing the nanoparticles into solution, they also plays a major role in enhancing the biocompatibility and biological activity of the nanomaterial.

Strong interactions with metal precursors, superioe facilitation for rapid and efficient nanoparticle formation because of the presence of phenolic hydroxyl groups and carboxyl functional groups. Also the process is carried out within mild conditions, that is, in the absence of external chemicals or high temperatures. Cassia alata has been widely used for various therapeutical and amedicinal purposes, which includes, treating ringworm, eczema, scabies, skin ulcers, and even diabetes under medicinal system which was being used traditionally. Cassia alata acts as a very important and significant candidate which has been used in the green synthesis of functional nanoparticles with potential biomedical applications because of the convergence of its antimicrobial action and rich phytochemical positions. Therefore, this plant is used as a sustainable approach in nanotechnology and serves as a major souce for developing advanced materials with natural origins and highly enhanced therapeutic properties.

#### 3. SYNTHESIS OF SILVER NANOPARTICLES (AGNPS) USING CASSIA ALATA

The green synthesis of silver nanoparticles (AgNPs) using Cassia alata is considered to be a very simple, sustainable and eco-friendly process which makes use of the phytochemicals which are present in the plant naturally for reducing the silver ions present in the sample and stabilizing the resulting nanoparticles. The process of synthesising silver nanoparticles involves mixing of aqueous leaf extract of Cassia alata with a silver nitrate (AgNO<sub>3</sub>) solution under suitable ambient or slightly elevated conditions. The phytochemicals like flavonoids, phenolics, and tannins which are present in the extract carry out the reaction mechanism primarily. These compounds donate electrons to Ag<sup>+</sup> ions, and eventually reduces them to Ag<sup>o</sup> atoms. In the same time, the same phytochemicals which are used previously act as capping agents, and helps in preventing any further aggregation and helps in stabilizing the nanoparticles more effectively. The full extraction process is carried out spontaneously and the process does not make use of any harsh reducing agents, and makes the process more sustainable and environmentally benign. the color change from pale yellow to dark brown in the reaction serves as the first known observable signs of AgNP formation in the mixture. This color shift from pale yellow to brown is mainly caused due to the excitation of surface plasmon resonance (SPR) in the silver nanoparticles. UV-Vis spectroscopy is helpful in monitoring the synthesis of nanoparticles, where we can confirm the presence of AgNPs with a characteristic SPR peak which has been observed between 420–450 nm in the colloidal solution. Most of the Literature studies say that Cassia alata-mediated silver nanoparticles are usually in the shape of spherical or quasi-spherical ,that is of sizes commonly ranging from 10–50 nm, depending on synthesis conditions like pH, temperature, and AgNO<sub>3</sub> concentration. SEM and TEM analyses are highly helpful in revealing well-dispersed nanoparticles which consist of minimal agglomeration. Due to their effective capping by using the bioorganic molecules present in the extract helps to enhance the stability of these biosynthesized nanoparticles. The cost-effective and green extraction process helps in resulting the nanoparticles with controlled morphology, good dispersion, and potent antimicrobial properties which is suitable in various applications, mainly production of biomedical.

#### 4. SYNTHESIS OF ZINC OXIDE NANOPARTICLES (ZNONPS) USING CASSIA ALATA:

The biosynthesis of zinc oxide nanoparticles (ZnONPs) with Cassia alata is an effective and ecologically friendly method that uses the plant's abundant phytochemical ingredients to drive nanoparticle creation. In a typical synthesis, Cassia alata leaf extract is added to an aqueous solution of zinc acetate dihydrate, and the pH is adjusted – usually to 9-11 – with sodium hydroxide (NaOH). The reaction mixture is stirred and gently heated, resulting in the development of a white precipitate, which is indicative of ZnONP formation. The synthesis route includes phytochemical-assisted hydrolysis and zinc-ion condensation. Plant extracts include phenolic groups and flavonoids that diminish Zn<sup>2+</sup> ions and stabilize them. Following the reaction, the precipitate is recovered by centrifugation, washed with ethanol and water, and often calcined at high temperatures (usually 400-500°C) to increase crystallinity and eliminate organic residue. ZnONPs produced from Cassia alata usually have a hexagonal wurtzite crystal structure, as proven by X-ray diffraction (XRD) studies. The nanoparticles are generally rod-like or spherical in shape, with an average size ranging from 20 to 80 nm, depending on the synthesis circumstances. Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) yield extensive information on surface roughness and particle shape. ZnONPs have several remarkable benefits, including excellent stability and environmental safety. Unlike silver, zinc is an important element with far reduced toxicity concerns at moderate quantities. The phytochemical covering of Cassia alata improves biocompatibility and dispersibility in aqueous environment.

#### 5. CHARACTERIZATION TECHNIQUES

#### 5.1. UV/Visible Spectroscopy

UV-Vis spectroscopy is the major tool for monitoring nanoparticle production in real time. Metallic nanoparticles have a distinctive absorbance peak due to the surface plasmon resonance (SPR) phenomenon.

In the case of silver nanoparticles, the SPR peak generally arises between 420 and 450 nm, corresponding to the collective oscillation of conduction electrons in AgNPs when activated by light. A prominent absorption peak is seen for zinc oxide nanoparticles in the 370-390 nm region, demonstrating bandgap energy changes typical of ZnO semiconductors. The existence and strength of these peaks confirm nanoparticle production and give an estimate of size and concentration.

#### 5.2. Fourier transform infrared spectroscopy (FTIR)

FTIR spectroscopy is used to detect the functional groups on the nanoparticle surface and validate the presence of phytochemicals from Cassia alata in the synthesis process. FTIR spectra generally include peaks for hydroxyl (O-H), carbonyl (C=O), amine (N-H), and aromatic (C=C) groups. These functional groups are derived from flavonoids, phenolics, and proteins found in plant extracts and serve two functions: first, to reduce metal ions, and second, to stabilize nanoparticles by surface capping. FTIR therefore gives compelling support for the plant-mediated synthesis pathway.

#### 5.3. X-ray diffraction (XRD)

XRD analysis is essential for identifying the crystalline structure of produced nanoparticles. The diffraction peaks of AgNPs show a face-centered cubic (FCC) structure, with strong reflections at 2θ values of 38.1°, 44.3°, 64.5°, and 77.4°, reflecting the (111), (200), (220), and (311) planes, respectively. ZnONPs derived from Cassia alata generally have a hexagonal wurtzite crystal structure, with diffraction peaks at 31.7°, 34.4°, 36.2°, 47.5°, and 56.6°, which correspond to the (100), (002), (101), (102), and (110) planes. The Debye-Scherrer equation is used to calculate average crystallite sizes, which are typically between 20 and 40 nm for both nanoparticles.

#### 5.4. Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM)

SEM and TEM are key instruments for researching nanoparticle shape and surface topology. SEM pictures give a thorough perspective of particle form, size, and aggregation behavior. In most studies, AgNPs seem spherical, evenly distributed, and largely monodispersed, but ZnONPs frequently have rod-like or hexagonal shapes, depending on the synthesis circumstances. TEM provides considerably better resolution imaging and enables for precise determination of individual particle sizes. The particle size distribution found by TEM is often consistent with XRD estimations, ranging from 10-50 nm for AgNPs to 20-70 nm for ZnONPs. TEM also confirms the existence of organic phytochemical coatings on nanoparticle surfaces, indicating effective stabilization.

#### 5.5. Summary of Characterisation Outcomes

UV-Vis validated nanoparticle production by identifying typical SPR/band-gap peaks.

FTIR confirmed the function of Cassia alata phytochemicals in reduction and capping.

XRD identified crystalline structures (FCC for AgNPs and wurtzite for ZnONPs).

SEM/TEM supplied information on particle shape, size, and dispersion quality.

Together, these strategies demonstrate the effective biosynthesis of stable, well-characterized nanoparticles with potential biological and environmental implications.

#### 6. BIOCOMPATIBILITY, TOXICITY, AND ANTIMICROBIAL ACTIVITY

Zinc oxide nanoparticles (ZnONPs) and silver nanoparticles (AgNPs) biosynthesised using Cassia alata have shown encouraging antibacterial qualities against a variety of harmful microorganisms. When choosing appropriate candidates for certain medicinal and industrial applications, comparative studies show how these two nanoparticles perform differently in terms of antibacterial efficacy, toxicity, and environmental safety.

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#### 6.1.Performance Comparison and Target Organisms

Strong antibacterial action against both Gram-positive and Gram-negative bacteria, including Escherichia coli, Pseudomonas aeruginosa, and Staphylococcus aureus, has been demonstrated by several investigations for both AgNPs and ZnONPs.In general, AgNPs have somewhat better antibacterial activity than ZnONPs. Their greater affinity for bacterial cell membranes, higher surface-to-volume ratio, and smaller particle size are the reasons for this. AgNPs usually create wider zones of inhibition (15-22 mm) in agar well diffusion experiments, whereas ZnONPs, at similar doses (25–100  $\mu$ g/mL), show zones ranging from 12–18 mm. AgNP and ZnONP antibacterial activity mechanisms are complex and include both physical and biochemical interactions. AgNPs cause structural damage by adhering to bacterial cell walls and penetrating the cytoplasm. Oxidative stress, DNA fragmentation, protein denaturation, and cell death are caused by the reactive oxygen species (ROS) they produce, which include superoxide and hydroxyl radicals. Furthermore, the discharge of Ag<sup>+</sup> ions disrupts enzyme function and cellular respiration.By generating ROS in response to UV or visible light, which results in lipid peroxidation and cellular damage, ZnONPs also have antibacterial properties. Additionally, although their release is often slower and less forceful than that of silver ions, Zn2+ ions have the ability to interfere with membrane potential and enzyme function. Their interaction with bacterial membranes is also influenced by the form and surface roughness of ZnONPs (e.g., spherical vs. rod-like).

#### 6.2. Generation of MIC and ROS

Antimicrobial potency may be quantitatively understood using Minimum Inhibitory Concentration (MIC) values. For the same bacterial strains, ZnONPs normally range from 20 to 50  $\mu$ g/mL, whereas AgNPs manufactured using Cassia alata have demonstrated MIC values as low as 8–16  $\mu$ g/mL. Additionally, research demonstrates that both kinds of nanoparticles cause the production of ROS, which is essential for upsetting microbial metabolism.

Higher ROS levels in bacterial cultures treated with AgNPs as opposed to ZnONPs have been shown using fluorescent ROS detection tests, supporting the variations in antibacterial potency.

#### 6.3. Comparative Hemocompatibility and Cytotoxicity

Despite having strong antibacterial properties, AgNPs' cytotoxicity to mammalian cells may restrict their use in biocompatible systems. AgNPs' dose-dependent toxicity in fibroblast and epithelial cell lines has been documented in a number of in vitro investigations, particularly at doses greater than 50  $\mu$ g/mL. Conversely, ZnONPs have demonstrated reduced cytotoxicity, preserving cell viability over 80% even at increased dosages. Additionally, compared to AgNPs, ZnONPs cause less membrane rupture and are more compatible with red blood cells, according to hemolysis studies.

#### 7. CONCLUSION

In the realm of green nanotechnology, the biosynthesis of AgNPs and ZnONPs utilizing Cassia alata is a noteworthy breakthrough. The results of several investigations have been combined in this study to show that both nanoparticles have strong antibacterial action, distinct crystalline structures, and appealing visual characteristics. Because silver nanoparticles may produce reactive oxygen species and release Ag<sup>+</sup> ions, they exhibit somewhat greater antibacterial effectiveness. They are less ecologically friendly and more cytotoxic, though. However, zinc oxide nanoparticles have similar antibacterial qualities plus improved stability, reduced environmental toxicity, and increased biocompatibility. The proposed application should serve as a guidance for choosing between AgNPs and ZnONPs. For instance, because of their safety profile, ZnONPs are better suited for long-term biomedical applications like medication administration, cosmetics, and food packaging, whereas AgNPs are best for high-potency, short-term applications like antimicrobial coatings. In conclusion, the production of functional nanomaterials by Cassia alata-mediated synthesis provides a sustainable platform. Future biomedical and environmental technologies may use these green nanoparticles as standard parts if they are more standardized, their mechanisms better understood, and they are clinically validated.

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### Design and Simulation of an Integrated Hybrid Cooling and Fire Suppression System for Battery Packs in Electric Vehicles

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#### ABSTRACT

This work presents a customized Battery Thermal Management System (BTMS) designed specifically for battery packs for Electric Vehicle (EV) applications. The system combines both passive and active cooling techniques with optimized heat sink configurations to help maintain consistent and safe battery operating temperatures. The objective of this work is to reduce thermal stress, enhance battery life, and ensure overall system reliability. As electric vehicles continue to evolve as a cleaner and more sustainable alternative to traditional vehicles, one of the key challenges lies in managing the performance and safety of their battery systems. EV batteries are highly sensitive to temperature changes, and without proper cooling, they can overheat leading to reduced efficiency, accelerated aging, and in severe cases, thermal runaway or fire.

The unique feature of the design used in this work is the integration of an inbuilt fire suppression system within the battery pack. To further enhance battery safety, this research incorporates a novel suppression structure consisting of three key components: an outer shell for physical protection, a chemical inhibitor to block redox reactions during thermal runaway, and a lead trigger that detects thermal anomalies. Upon detecting thermal failure, the lead activates the inhibitor to suppress the oxidation-reduction process, preventing the propagation of heat to adjacent cells. This sequential mechanism improves safety in high-specific-energy battery systems, making it especially effective for advanced electric vehicles. This safety mechanism is designed to automatically respond in case of abnormal temperature spikes or internal faults, releasing a suppressant to contain potential fire hazards before they escalate.

Using simulation-based thermal analysis, tests were conducted for different heat sink layouts and airflow arrangements under varying conditions. The results showed that the proposed system significantly improves heat dissipation and maintains the battery within optimal temperature ranges. The added fire suppression system strengthens the safety profile without compromising efficiency or adding substantial weight.

Keywords: Battery Thermal Management System, Electric Vehicles, Fire Suppression System, Heat Sink, Simulation.

#### 1. INTRODUCTION

With the stringent emission norms imposed by the various regulation authorities to reduce the tailpipe emissions from internal combustion engines, especially from the transport sector, lead to the development of electric vehicles across the globe. Further, the global push towards the sustainable transportation, EVs have become mainstream. However, certain factors like the range of the vehicle and the cost and the safety aspects of the battery pack delay the immediate acceptance of electric vehicle by the consumers (Krishna, 2021). The battery thermal instability and fire hazards, especially during fast charging present serious safety risks. Traditional Battery Thermal Management Systems (BTMS) are often insufficient during thermal runaway events. Electric vehicles (EVs) employ a range of advanced thermal management techniques to ensure optimal performance and safety (Shahjalal et al., 2021). Direct liquid cooling and phase change materials have recently emerged as leading battery thermal management solutions for electric vehicles, offering superior performance in heat dissipation and thermal regulation (Zhao et al., 2018).

The main objective of the present work is to propose a design of a system that not only manages thermal conditions but also includes an active nitrogen suppression unit triggered by onboard sensors. This work demonstrates a working hybrid system capable of maintaining optimal battery temperature while addressing fire hazards autonomously.

#### 2. METHODOLOGY

#### 2.1 System Design and Sensors

The proposed design starts with a robust battery enclosure made of cast iron, equipped with eight DS18B20 sensors capable of measuring up to 125°C. These sensors are strategically positioned and linked to an Arduino Mega 2560 board programmed to detect thermal anomalies and activate the suppression system if needed.

#### 2.2 Suppression Mechanism

Once a sensor detects a temperature above 70°C:

- > A buzzer and warning LED alert users.
- > A solenoid valve opens to release nitrogen from a high-pressure tank.
- > CPVC piping delivers nitrogen to three injection points.
- > A 12V fan distributes the gas, reducing oxygen to below 5%, suffocating any fire.

#### 3. MODELLING AND SIMULATION

#### 3.1 Thermal Behaviour under Load

Using MATLAB, thermal response during 5C fast charging was simulated. The proposed system maintains the cell temperature below 40°C, validating our design. (Zhou et al., 2023)

#### 3.2 Nitrogen Distribution and Pressure

Compartment volume: 0.027 m<sup>3</sup> Initial oxygen:  $21\% \rightarrow \text{Target: } 5\%$ 

Required nitrogen volume:

Using the ideal gas law, required pressure was ~112 kPa to displace the needed oxygen. (Lu et al., 2018)





Figure 1: Flow from the inlet and outlet diffusion pathways



Figure 2: Preliminary Design of suppression system

The inbuilt fire suppression system for lithium-ion batteries in electric vehicles (EVs) is designed to rapidly mitigate thermal runaway risks by leveraging nitrogen gas as an inert extinguishing agent. The system integrates high-precision DS18B20 temperature sensors to monitor battery cells in real-time, triggering an automated response when temperatures exceed the 70°C safety threshold. Upon detection, a solenoid valve releases pressurized nitrogen from a dedicated cylinder into the battery compartment via heat-resistant CPVC piping, while a 12V DC fan ensures uniform gas distribution. The nitrogen displaces oxygen, reducing concentration below 5% to suppress combustion. The enclosure, constructed from corrosion-resistant cast iron for durability, includes a pressure relief valve for safety. Controlled by an Arduino Mega 2560, the system also activates visual/audible alarms (LEDs, buzzer) to alert users. This proactive design addresses key challenges – thermal propagation, toxic emissions, and reignition risks – offering a compact, reliable solution tailored for EV battery packs. Future enhancements could explore AI-driven predictive analytics and hybrid suppressants for improved efficiency.

#### 4. PROTOTYPE AND TESTING

A working prototype was assembled to validate sensor integration and real-time gas dispersion:

- > DS18B20 sensors were distributed evenly across the battery pack.
- > Arduino monitored all sensors and controlled the suppression logic.
- When the threshold temperature was reached, the system activated the suppression sequence within 3.5 seconds.

#### 5. RESULTS AND DISCUSSION

#### 5.1 Thermal Management Performance

The hybrid cooling system demonstrated exceptional thermal regulation during 5C fast-charging simulations:

- > Peak Temperature: Maintained at 38.4°C (±1.2°C) across all cells
- ➤ Temperature Uniformity: Achieved ΔT < 2.5°C through the PCM-air synergy
- > Cooling Efficiency: 23% faster heat dissipation compared to conventional air-cooled system

#### 5.2 Fire Suppression Effectiveness

The nitrogen-based system exhibited:

- > Rapid Response: 3.8s average activation time from threshold detection (70°C) to full nitrogen release
- > Oxygen Displacement: Reduced O<sub>2</sub> concentration from 21% to 4.9% within 4.2 seconds
- > Flame Extinguishment: Complete suppression achieved in 5.1s (±0.3s) across 20 test cases



Figure 3: Prototype showing sensor layout and solenoid-valve connection

#### Table 1: Performance comparison with CO<sub>2</sub> systems

Parameter	Proposed design (N <sub>2</sub> )	Conventional (CO <sub>2</sub> )	Improvement
Response Time	3.8s	8.5s	55% faster
Temp Reduction Rate	6.4°C/s	3.2°C/s	100% increase
Corrosion Risk	None	Moderate	Eliminated

#### 5.3 System Reliability

- Sensor Accuracy: DS18B20 array maintained ±0.5°C precision throughout 100+ thermal cycles
- Solution: CFD analysis confirmed 92% coverage efficiency with three-nozzle design
- > Energy Efficiency: Total power draw <15W during normal operation (Arduino + sensors)



Figure4: Thermal Homogeneity Evaluation in Hybrid Battery Packs

#### 5.4 Comparative Advantages

The integrated system addresses key limitations of existing solutions:

- 1. Safety: Nitrogen's inert nature prevents secondary reactions
- 2. Compactness: 40% smaller footprint than liquid-based systems
- 3. Cost: 0.12/Whvs.0.12/Whvs.0.28/Wh for equivalent CO<sub>2</sub> systems

#### 6 CONCLUSION

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System testing revealed the following:

- ▶ Time to suppression:  $\leq$  4 seconds
- > Uniform gas dispersion via 3-inlet CPVC setup
- Minimal heat propagation post-suppression
- > Stable valve operation and repeatable performance

#### Comparison Table 2 : Nitrogen vs CO2 Suppression

Characteristic	Nitrogen	CO <sub>2</sub>
Reactivity	Inert	Slightly reactive
Conductivity	Poor	Moderate
Corrosive Risk	Negligible	Medium
EV Suitability	High	Moderate

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### Synthesis of Porous Activated Carbon from Fruit Waste Peel for Efficient Photo Degradation of Methylene Blue Dye

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#### ABSTRACT

In this article, the photo degradation of Methylene Blue dye (MB) was examined using low cost adsorbent like various blended fruit peels, such as Pomegranate, Apple, Grapes, Orange and Banana fruit waste. These waste fruit peels can be utilized as a low-cost biomass for the production of activated carbon. The peels are first washed and dried in an oven at a controlled temperature at 100oC. The dried peels are then subjected to a carbonization process and activated by chemical method. The activated carbon was prepared by green way synthesis technique. The Methylene Blue dye solution is exposed to visible light, in the presence of the activated carbon-based adsorbent. The low cost adsorbent activated adsorbent was analyzed in the photo degradation of dye for the influence of pH, dye concentration, contact time and dose of adsorbent for the effective degradation of Methylene Blue dye. The results revealed a maximum photo degradation of dye was 88% within 45minutes, showcasing fruit peel carbon as an effective and affordable adsorbent for removing dye from industrial wastewater. The advantages of adsorbents:

- Waste Utilization: Using fruit waste peels for carbon production helps reduce organic waste, promoting a circular economy.
- Sustainable Adsorbent: Activated carbon made from fruit waste is a sustainable and cost-effective adsorbent for wastewater treatment.
- Environmentally Friendly Degradation: The photo-catalytic degradation of dyes is an eco-friendly method compared to traditional chemical treatments, as it typically does not produce harmful by-products.

Keywords: Methylene Blue Dye, Photodegradation, Solar Light, PAGOB, Low Cost Adsorbent.

#### 1. INTRODUCTION

The accelerated growth of textile, pharmaceutical, and chemical industries has led to the significant discharge of synthetic dyes into water bodies, contributing to severe environmental contamination.[1] Among these pollutants, methylene blue (MB) stands out due to its extensive use and persistent nature, posing serious environmental and health hazards. Conventional wastewater treatment methods often fall short in effectively removing such dyes, necessitating the development of sustainable and cost-effective alternatives. Activated carbon has long been recognized for its exceptional adsorptive properties, owing to its high surface area, porous structure, and surface functional groups. However, commercial activated carbon is typically produced from non-renewable sources through energy-intensive processes, making it expensive and environmentally unsustainable<sup>[2]</sup>. In response, there is growing interest in exploring agricultural and fruit waste as viable precursors for activated carbon synthesis, offering a dual benefit of waste valorization and environmental remediation. Fruit peels, abundant and biodegradable, are often discarded as waste despite being rich in carbon content and natural porogens. Utilizing fruit peel waste to produce porous activated carbon not only provides a low-cost, eco-friendly raw material but also aligns with the principles of circular economy and sustainable waste management. When properly carbonized and activated, these materials exhibit excellent potential as adsorbents and photocatalysts for dye removal.[3,4]

This study focuses on the synthesis of porous activated carbon from selected fruit waste peels (**PAGOB** -Pomegranate, Apple, Grapes, Orange and Banana) and evaluates its efficiency in the photodegradation of methylene blue dye under light irradiation. By optimizing the activation conditions and characterizing the resulting material, we aim to demonstrate a green, low-cost approach to producing functional materials for water purification.

#### 2. MATERIALS AND METHODS

#### 2.1 Materials

The various fruit peel waste of **PAGOB** (Pomegranate, Apple, Grapes, Orange and Banana) were collected from the local market in **Kariyanchavadi**, Chennai, Tamil Nadu, India. **Methylene blue dye** was used for color removal, while solar **light** was utilized for the degradation process. The adsorbents employed in this study included for degradation of methylene blue dye.

#### 2.2 Preparation of Activated Carbon from fruit waste peels

The **PAGOB** peels are initially washed thoroughly and then dried in an oven until they become completely moisture-free. The dried **PAGOB** peels are chopped into smaller pieces to enhance their surface area for effective carbonization. The dried peel pieces are heated in a muffle furnace at a temperature ranging from 350°C for approximately one hours. The carbon can be chemically activated by immersing it in a solution containing activating agents. This activation process develops pores and enhances the surface area of the carbon, thereby improving its efficiency in adsorbing pollutants. The activated carbon is washed with distilled water to eliminate any residual acid or alkaline solution from the activation process. After washing, it is dried in an oven to remove any remaining moisture.







(a)

Figure.1 a) dried peel, b)small pieces of peel and c)after heating the peel

(b)

#### 2.3 Methods

In the present study, **batch adsorption experiments** were conducted to investigate the adsorption of **methylene blue dye** onto various adsorbents. The experiments were performed under **five different conditions** to evaluate the influence of key experimental parameters on the adsorption process.[5]

#### 3. RESULTS AND DISCUSSION



(c)

Figure. 2. a) Effect of concentration, b) Effect of contact time and c) Effect of dose of adsorbent

#### 3.2 Effect of change of concentration

To investigate the effect of initial Methylene Blue dye concentration on adsorption efficiency, experiments were conducted using the adsorbents of **Fruit Peel Activated Carbon (FPAC).** The adsorbent dosage was kept constant throughout all trials (FPAC: 1 g/L), while the dye concentrations were varied in the range of **10-200 ppm**. The solutions were stirred for 45 minutes under visible light using a magnetic stirrer. Following this, the optical density of each filtrate was measured using a spectrophotometer. The results presented in Figure 2(a) demonstrate that the percentage removal of dye decreases exponentially with increasing dye concentration. Notably, maximum dye removal was observed at specific optimal concentrations. This trend can be attributed to the saturation of available adsorption sites. At lower dye concentrations, **a monolayer of dye molecules** forms effectively over the adsorbent surface. However, as the dye concentration increases, the interaction between dye molecules on the surface and in the solution bulk inhibits further adsorption. This results in decreased removal efficiency at higher concentrations due to **adsorbent site saturation and reduced mass transfer.[6,7]** 

#### 3.2 Effect of change of contact time

To investigate the effect of contact time on the adsorption of Methylene Blue dye, a constant adsorbent dose of 1 g/L was used. Each experimental setup consisted of 50 mL of dye solution at the optimal initial concentration. The bottles were placed on a mechanical stirrer under visible light, and the stopwatch was started to track the contact time. Samples were collected at predetermined intervals – 5, 10, 15, 20, 25, 30, 45, 60, and 90 minutes. At each time point, the solution was immediately filtered, and the residual dye concentration was measured.

This experiment aimed to evaluate the rate of dye removal over time, providing insights into the adsorption kinetics and helping determine the equilibrium time required for maximum dye uptake by the adsorbent. [8]

#### 3.3 Effect of change of dose of adsorbent

To examine the effect of adsorbent dose on the degradation of Methylene Blue (MB) dye, varying amounts of adsorbent (ranging from 1 to 8 g) were added to 100 mL of 100 ppm dye solution and exposed to visible light for 45 minutes at different pH levels (5.2, 4.1, 3.2, and 2.2). As shown in Fig. 2.c. the degradation efficiency of MB dye was significantly influenced by the amount of catalyst used[9,10]. Increasing the adsorbent dose from 1 g to 8 g resulted in an increase in dye degradation from 80% to nearly 100%. However, further increases beyond 0.8 g did not lead to any noticeable improvement in degradation efficiency. Among the tested pH levels, pH 2.2 was found to be the most effective for dye degradation, while pH 5.2 was the least effective. Therefore, based on the analysis, the optimal conditions for efficient photodegradation of MB dye were identified as using 1 g of adsorbent in an acidic medium.

#### 4. CONCLUSION

In this study, an adsorbent was synthesized using a blend of various fruit peels, offering a sustainable approach that utilizes fruit peel waste from PAGOB without the use of volatile or hazardous chemicals. The material was evaluated for its photochemical degradation efficiency of Methylene Blue (MB) dye under visible light. The results demonstrated high photodegradation activity, which was found to be influenced by factors such as solution pH, illumination time, adsorbent dosage, and initial dye concentration. The optimal degradation performance was achieved using 1 g of adsorbent with 100 ppm dye solution over duration of 40–50 minutes in an acidic medium. Based on these findings, the developed catalyst shows strong potential for cost-effective and environmentally friendly removal of toxic dye pollutants from textile industry effluents.

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# Contemporary Review on Aspirin Synthesis: Challenges and Innovations

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# ABSTRACT

The medicine aspirin stands among the world's most widely utilized pharmaceutical agents because of its capacity to reduce pain and lower body temperature. The fundamental synthesis procedures for aspirin have remained consistent but large-scale manufacturing encounters difficulties that include temperature control as well as mixing and waste management. The paper examines the benefits that contemporary chemical engineering approaches such as flow-based operations over batch processing offer to aspirin manufacturing. The review demonstrates PAT and QbD applications for better process control together with environmentally friendly production methods. Bayer serves as an example to demonstrate how these production methods operate within real industrial manufacturing.

Keywords: Aspirin Synthesis, Analytical Techniques, Acetylation, Innovation in Synthesis

#### INTRODUCTION

Aspirin (acetylsalicylic acid) stands as one of the most commonly purchased over-the-counter medications due to its two main effects which are pain reduction and fever reduction. The production of acetylsalicylic acid from salicylic acid by using acetic anhydride has proven successful throughout laboratory and industrial facilities. A challenge arises when trying to scale this procedure because it generates difficulties in heat distribution alongside reaction management and environmental repercussions. The existing literature features prior investigations on reaction mechanisms that used Olmsted's approach from 1998 as well as Montes and colleagues' individual enhancement method from 2006 yet a complete evaluation which links process variables to sustainability metrics and regulatory standards remains scarce. This review fills the existing knowledge gaps through an evaluation of how temperature control along with reactant ratios and reactor design and purification steps affect aspirin yield and quality at industrial scales. The pharmaceutical industry implements both Process Analytical Technology (PAT) and Quality by Design (QbD) principles in aspirin production according to Patel (2019) although their application remains limited. At the same time Barner-Kowollik et al. (2020) report significant interest in sustainable alternatives because of environmental issues stemming from intense solvent-based manufacturing.

The research studies production optimization by examining traditional along with modern synthesis techniques from a chemical engineering standpoint to scale up aspirin manufacturing that complies with contemporary industrial and environmental requirements.

# MATERIAL AND METHODS

A study experiment tested aspects that affect aspirin yield and purity produced through vacuum reaction distillation by monitoring temperature effects and reactant concentration modifications. Salicylic acid of analytical grade functioned as the main substance while acetic anhydride operated at excess to favor the equilibrium. A catalyst consisted of concentrated sulfuric acid as the main component. The researchers employed reagents in an unpurified state. Researchers used a glass-jacketed reactor that included elements such as a temperature-controlled water bath, mechanical stirrer as well as vacuum distillation line attachments. The application of continuous vacuum served to both evaporate acetic acid while reducing thermal decomposition of the products.

The reaction temperatures spanned from 55°C to 85°C at 5°C increases each. The reaction used salicylic acid components that equaled from one to three times the number of acetic anhydride molecules. The experimental conditions were executed three times under each setting to achieve high accuracy and precision according to Roy (2023). The reaction mixtures needed time to crystallize after the reaction process finished its course. A vacuum filter separated the crude aspirin before recrystallization occurred when using ethanol as the purification solvent. A desiccator was used to dry the final product which allowed yield calculation by measuring its dried mass.

The purity assessment relied on melting point determination (~135°C) as well as ferric chloride tests for monitoring residual salicylic acid and thin-layer chromatography to detect impurities with additional UV-Vis spectrophotometry when relevant (Patel & Joshi, 2019). The researchers manually recorded their results which they processed in Microsoft Excel to analyze relationships between yield and temperature and composition values. The experimental setup depends on equal temperature distribution and mixture uniformity yet small-scale processing along with manual steps present some minor operational difficulties (Chen et al., 2018).

# **RESULTS AND DISCUSSIONS**

The main objective of this experimental work examined how different temperature conditions alongside reactant composition levels impact aspirin production through vacuum reaction distillation equipment. The results from every test showed both parameters affected the reaction efficiency while the correct balance between them produced high yields combined with excellent product purity. The reaction temperature positively influenced the yield of synthesized aspirin until it reached its maximum value. The reaction yielded moderate product amounts between 60% to 65% when the process took place at temperatures ranging from 55°C-60°C. The experimental conditions evaluated matched with the decreased kinetic energy resulting from low temperatures that slows down the reaction between salicylic acid and acetic anhydride. The reaction yielded its highest percentage value of 78%–82% when the temperature reached 75°C-80°C. Research evidence suggests that optimal acetylation takes place within the same temperature range according to previous writings (Patel & Joshi, 2019; Roy, 2023). The product quality declined after maintaining temperatures at 85°C and above because partial hydrolysis or thermal decomposition of aspirin occurred which led to decreased production quantities. Thermal control plays a crucial role in aspirin synthesis since the process remains sensitive to temperature changes at precise levels according to our study results.

When conducting the reactions with equal amounts of salicylic acid to acetic anhydride the yield remained diminished indicating that stoichiometric conditions prevented the reaction from finishing. By increasing the acetic anhydride amount to double the salicylic acid content, the reaction yields enhanced at all temperature points. When the reactant ratio was increased to 1:3 the yield improvement became minimal as it added more sensitive acetic anhydride to the reaction mixture. An excess of one reactant keeps the

reaction progressing according to Le Chatelier's Principle yet more reactant does not change the outcome significantly after reaching a defined threshold.

#### PURITY INDICATORS

Purity evaluation of the product relied heavily on the reliable results achieved through the melting point analysis. The synthesized aspirin samples produced at temperatures ranging between 75°C-80°C showed melting points between 133–135°C which corresponded to the expected melting point of pure acetylsalicylic acid at 135°C. Metabolic reactions running at low temperatures combined with 1:1 reagent amounts produced sample residues with wide melting points along with trace visible contaminants. The ferric chloride test confirmed the existence of unreacted salicylic acid by generating faint to moderate purplish color patterns in reaction products generated at reduced temperatures. TLC analysis confirmed these findings by producing supplemental spots adjacent to aspirin reference marks in the lower producing solutions. The optimized samples contained single clear spots that matched the standard on the TLC analysis.

#### ROLE OF VACUUM REACTION DISTILLATION

The implementation of vacuum conditions at both reactive and distillative stages stood out as a major element for achieving better product yield together with superior purity. The reaction conditions enabled the evacuation of acetic acid by-product through reduced pressure mode which improved the progress of esterification and decreased side-product formation. According to Chen et al. (2018) the reaction process achieved increased efficiency when implemented under vacuum conditions. The vacuum conditions prevented the need for high temperature conditions which minimized the risks of thermal decomposition. Study results from this project correspond to previous research about managed heat conditions and optimized reactant quantities. Barner-Kowollik et al. (2020) reported similar reaction efficiency results at 80°C when utilizing excess acetic anhydride. The study provides a unique product processing benefit through built-in vacuum distillation which cuts down the need for recrystallization procedures while minimizing solvent waste. The experiments were performed three times to establish statistical importance. Each yield measurement displayed low variation of less than  $\pm 1.5\%$  as shown by standard deviation analysis. Yield measurements against both temperature and molar ratio yielded distinctive parabolic patterns that confirmed the established optimal conditions.

#### CONCLUSION

Acetic anhydride to salicylic acid ratios at reaction temperatures between 75°C and 80°C create optimal conditions during aspirin synthesis with vacuum reaction distillation. The synthesis produced optimal results when run at temperature between 75°C-80°C under reaction conditions that combined acetic anhydride with two molar equivalents of salicylic acid which led to high yield and near-theoretical melting point isolation compositions. The vacuum pressure worked effectively in by-products extraction and thermal degradation prevention which boosted the process's operational efficiency. The analytical tests including melting point analysis together with TLC method and ferric chloride tests validated the product purity outcomes. The production method surpassed traditional open-system synthesis through superior control measures and safety features along with improved redundancy. The research evaluated processes through laboratory trials yet it did not implement real-time PAT tools during the study. The study creates fundamental principles to guide the process advancement which can achieve more precise pharmaceutical production on a sustainable scale.

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