

**USE OF PHOTOCATALYTIC TRANSESTERIFICATION METHOD FOR
PRODUCTION OF BIODIESEL FROM
SILK COTTON-SEED OIL BY THE USE OF ZnO AND TiO₂ CATALYST**

Submitted in partial fulfillment of the requirements for the award of Bachelor of
Technology degree in Chemical Engineering

by

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of A. CHRISTILA FERNANDO (40190006), V. PAVI NAMRUTHAA (40190016), and R. RAKSHANA (40190020). USE OF PHOTOCATALYTIC TRANSESTERIFICATION METHOD FOR PRODUCTION OF BIODIESEL FROM SILK COTTON-SEED OIL BY THE USE OF ZnO AND TiO₂ CATALYST who carried out the project entitled under my supervision from NOVEMBER 2023 to APRIL 2024

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ABSTRACT

Biodiesel, derived from renewable feedstocks such as silk cotton seed oil, presents an environmentally friendly and domestically produced alternative fuel with numerous benefits, including reduced greenhouse gas emissions, enhanced energy security, and support for sustainable agriculture. Traditional biodiesel production methods often involve chemical catalysts and heat in transesterification reactions. However, photo-transesterification offers an innovative approach utilizing light energy, particularly ultraviolet (UV) or visible light, to drive the reaction. Techniques such as FTIR (Fourier Transform Infrared Spectroscopy), GC-MS (Gas Chromatography-Mass Spectrometry), and IC engine studies are conducted. FTIR helps identify functional groups in biodiesel, GC-MS analyses its chemical composition, while IC engine studies evaluate its combustion properties and engine performance. These characterization studies play a crucial role in assessing biodiesel's suitability as a sustainable alternative fuel. The identification of compounds in biodiesel production is crucial for understanding its composition and yield. In our study, we confirmed the presence of glycerol through the detection of characteristic peaks in the FT-IR spectrum. Specifically, the OH group of glycerol was identified at approximately 3007.44 kg/m^2 , while the corresponding C-H peak appeared at 2853.17 kg/m^2 in the bottom layer. Moreover, the absence of an OH peak alongside the presence of ester (1647 kg/m^2) and C-H ($2853.17\text{--}2853.44\text{ kg/mol}$) peaks in the bottom layer suggests the prevalence of chain fatty ester compounds, which are indicative of biodiesel composition. To optimize the yield percentage, experiment with variations in molar ratio, catalyst weight percent, and reaction time. Currently, the highest yield of 81% was achieved at a molar ratio of 1:24, catalyst weight percent of 5%, and reaction time of 180 minutes. Adjusting these parameters slightly, along with optimizing temperature, and minimizing byproducts, could enhance yield further.

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LIST OF ABBREVIATIONS

CPSO	<i>Ceiba pentandra</i> seed oil
FE-SEM	Field scanning electron microscopy
EDX/EDS	Electron beam field dispersive x-ray spectroscopy
BET	Brunauer-Emmett-Teller
GCMS	Gas chromatography mass spectroscopy
BJH	Barrett-Joyner-Halenda
FTIR	Fourier transform infrared spectroscopy
FAME	Fatty acid methyl esters and glycerol
VOCs	Volatile organic compounds
DSSCs	Dye-sensitized solar cells
TEM	Transmission electron microscopy
FFAs	Free fatty acids
TG	Triglycerides
ANOVA	Analysis of variance
ASTM	American society for testing and material
EN	European
CYTOP™	Cyclic transparent optical polymer
TBADT	Tetra-n-butylammonium decatungstate
TDDFT	Time dependent density functional theory
FAO	Food and agriculture organisation
PSO	Program specific outcomes
SSA	specific surface area

RT	Retention time
SFC	Sulfated Fatty Acid Methyl Ester
IP	Indicated Power
BP	Brake Power
FP	Friction Power
IMEP	Indicated Mean Effective Pressure
BMEP	Brake Mean Effective Pressure
FMEP	Friction Mean Effective Pressure
IThEff	Indicated thermal efficiency
BThEf	Brake thermal efficiency
SFC	specific fuel consumption
HBP	High-Pressure Pump
HJW	High-pressure Jet Wash
HGas	Hydrogen Gas
Hrad	Heat Release Rate
MFB	Mass fraction burned
TDC	Top dead centre
IVO	Intake valve opening
CHR	Cumulative heat release
NHR	Net heat release
RoPR	Rate of pressure rise
MGT	Mean gas temperature

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CHAPTER 1

INTRODUCTION

1.1 BIODIESEL

Biodiesel is a renewable and environmentally friendly alternative to traditional fossil fuels, especially petroleum-based diesel. This is a biofuel made from organic materials, usually vegetable oil, animal fat, or recycled cooking oil. The production of biodiesel is a process called transesterification, where this raw material is chemically processed to break down the triglyceride structure into biodiesel and glycerol. One of the major advantages of biofuel over conventional diesel fuel is its ability to reduce greenhouse gas emissions. Because biodiesel comes from a renewable resource, the carbon dioxide released during combustion is part of the natural carbon cycle. Instead, burning fossil fuels releases carbon dioxide that has been stored for millions of years, thereby exacerbating climate change. Biodiesel can be used in diesel engines without modification because it is convenient and suitable for transport infrastructure. There are also properties that improve fuel lubrication, thus extending the life of diesel engines.

The widespread use of biodiesel is driven by a variety of factors, including concerns about energy security, the desire to reduce dependence on fossil fuels, and the need to address environmental issues. Governments and corporations around the world are looking for ways to integrate biomass into their energy production in order to create a more sustainable and energy-efficient future. Although the impact is positive, challenges such as the supply of raw materials, production costs, and competition for raw materials for food production must still be overcome. Biodiesel production is an economical primary fuel source. Research and development continue to improve the efficiency and sustainability of biodiesel production, making it a key driver in the transition to clean and sustainable energy solutions. Biodiesel production is an environmentally friendly and sustainable process that involves converting renewable raw materials, such as vegetable oil, into environmentally friendly and sustainable products. Recycled animal fat, or cooking oil, is an alternative to conventional diesel fuel.

The most important way to improve environmental sustainability in biodiesel production is to diversify livestock feed and focus on non-food sources such as

waste oil and algae. To reduce carbon emissions and encourage the switch to green fuels, governments continue to implement regulations and incentives to encourage the biofuels industry. The goal of technical research and development is to ensure the full life of the organism while researching new raw materials and increasing the efficiency of production. In addition, due to developments in engine technology, it is possible to blend other biodiesel with conventional diesel fuel. Eco-design technologies have revolutionized the landscape of bio-based manufacturing, incorporating sustainable and eco-friendly practices throughout the process. The key is the selection of raw materials, using non-food sources such as algae, jatropha, or cooking oil to reduce the pressure on food resources.

Biodiesel also provides energy security by diversifying fuel sources and relying on domestic raw materials. Since biodiesel is compatible with existing diesel engines and infrastructure, this will allow for better transferability and wider adoption. In addition, the lubricating properties of biodiesel extend the life of the engine, thus reducing maintenance costs.

The conversion process is key to biodiesel synthesis and requires the use of environmentally friendly catalysts such as enzymes or solid catalysts, thus making cleaning more efficient than traditional chemical machinery. Innovations such as supercritical water technology improve environmental efficiency by operating at low temperatures and pressures, saving energy, and reducing the overall ecological footprint. Biomass synthesis has become an environmentally friendly activity, optimizing the use of various raw materials and reducing waste, thereby encouraging biological sustainability. The focus on waste utilization, including the conversion of waste cooking oil and animal fats into biomass, demonstrates commitment to circular economy principles.

Energy-efficient processes, often incorporating renewable energy sources, help reduce the carbon footprint of biodiesel production. In addition, water recycling and biological plant life conservation practices. Life cycle analysis is a comprehensive tool that evaluates the entire biodiesel production process, from raw material cultivation to end-use disposal. By incorporating these environmentally friendly technologies, the biodiesel industry is paving the way to an environmentally responsible and sustainable future in the alternative fuel sector. Alternative catalysts

in biodiesel production represent a major leap towards more sustainable and efficient processes.

Biodiesel continues to be a significant player in the renewable fuels market, although its exact status may vary depending on factors such as government policies, technological advancements, and market demand. Biodiesel production and consumption have been steadily increasing over the years due to various factors such as concerns about climate change, energy security, and the desire to reduce reliance on fossil fuels. Many countries have implemented policies and incentives to promote the use of biodiesel as part of their efforts to reduce greenhouse gas emissions and meet renewable energy targets. These policies include blending mandates, tax incentives, subsidies, and renewable fuel standards.

Ongoing research and development efforts are focused on improving biodiesel production processes, increasing feedstock diversity, enhancing fuel properties, and reducing production costs. These advancements contribute to the continued growth and viability of biodiesel as a renewable fuel option. Despite its benefits, biodiesel faces several challenges, including competition with other biofuels such as ethanol, concerns about land use change and deforestation associated with certain feedstocks, and fluctuations in feedstock prices. The global biodiesel market is influenced by trends in energy markets, environmental regulations, agricultural policies, and trade dynamics. Emerging economies, in particular, are expected to play a significant role in driving future growth in biodiesel production and consumption.

1.2 RESEARCH OBJECTIVE

The objective of our project is to produce biodiesel by photocatalytic method using ZnO and TiO₂ catalyst. The chosen of these two catalysts is due to their photocatalytic performances. The use of photocatalytic transesterification method is due to their several potential advantages over traditional transesterification methods. This method utilizes light-activated catalysts to facilitate the transesterification reaction. Photocatalytic reactions can exhibit higher selectivity towards the desired product, leading to fewer undesirable by-products or impurities in the biodiesel. This can result in higher yields and improved product quality and

also occurs under milder reaction conditions which leads to reduced energy consumption and lower operating costs.

However, it's essential to note that photocatalytic transesterification is still an emerging technology and may face challenges such as the development of efficient and stable photocatalysts, optimization of reaction conditions, and scalability for industrial-scale production. Additionally, the economic viability of photocatalytic transesterification compared to traditional methods will depend on factors such as catalyst cost, energy requirements, and process efficiency. Further research and development efforts are needed to fully realize the potential benefits of this approach for biodiesel production.

The use of silk cotton seed oil is due to their Abundant Availability .it is cultivated worldwide, and silk cotton seed oil is readily available as a byproduct of cotton production. This abundant supply makes cottonseed oil a potentially sustainable feedstock for biodiesel production. It typically has a high oil content, making it an efficient feedstock for biodiesel production. This means that a significant amount of biodiesel can be produced from a given quantity of cottonseed oil. By utilizing cottonseed oil for biodiesel production, waste from the cotton industry can be minimized, contributing to waste reduction and resource efficiency.

1.3 PROBLEM STATEMENT

Biodiesel production, a promising alternative to fossil fuels, faces a critical hurdle: maintaining optimal reaction temperatures during transesterification. Deviations from this ideal range can significantly impact the process, leading to lower biodiesel yield, unwanted byproducts, and increased energy consumption. This inefficiency necessitates the development of improved temperature control strategies. By achieving precise temperature control, we can unlock the full potential of biodiesel production, maximizing yield, minimizing waste, and optimizing energy usage, ultimately paving the way for a more sustainable and efficient biofuel industry

The problem statement for biodiesel production involves several key aspects, including the reversible nature of the transesterification reaction, challenges associated with commercial-scale batch processing, and the utilization of photocatalytic reactors with ZnO and TiO₂ catalysts. Additionally, issues related to glycerol formation present further complications.

Biodiesel production through transesterification involves a reversible reaction, which complicates the achievement of high yields and purity levels. This reversibility necessitates the careful optimization of reaction conditions to Favor the production of biodiesel over the reactants, thus maximizing efficiency.

Commercial-scale biodiesel production often relies on batch processing due to its simplicity and flexibility. However, this approach poses challenges such as limited scalability, longer processing times, and difficulties in maintaining consistent reaction conditions, leading to variations in product quality and yield.

The utilization of photocatalytic reactors presents a promising alternative for biodiesel synthesis, with ZnO and TiO₂ emerging as catalysts due to their photocatalytic properties. However, challenges remain in optimizing reactor design, catalyst loading, and reaction parameters to achieve high conversion rates and selectivity while minimizing energy consumption and catalyst degradation.

Furthermore, glycerol formation during transesterification poses a significant problem, as it affects product purity and complicates downstream processing. Strategies to mitigate glycerol formation, such as catalyst modification or process optimization, are essential to enhance the overall efficiency and economic viability of biodiesel production processes.

The analysis of biodiesel production experiments reveals significant challenges in achieving optimal yield and maintaining temperature control. Despite variations in parameters such as molar ratio, catalyst weight percentage, and reaction time, the highest yield was not consistently attained across experimental conditions. For instance, while the yield peaked at 67% with a molar ratio of 1:24 and a catalyst weight percentage of 5%, it fluctuated in other scenarios, indicating a lack of consistency in achieving maximum yield. Additionally, the experiments did not explicitly address temperature control, which is crucial for maintaining reaction stability and maximizing yield. Without precise control over temperature, variations in reaction conditions may occur, potentially impacting yield and product quality. Therefore, the results underscore the challenges associated with optimizing yield and controlling temperature effectively in biodiesel production, highlighting the need for further research and refinement in these areas.

1.4 PHOTOCATALYTIC TRANSESTERIFICATION:

Photocatalysis represents an innovative biotechnological approach that integrates photocatalytic principles into traditional conversion methods. This method uses light energy to initiate the conversion reaction using a catalyst, usually an inorganic material such as titanium dioxide (TiO_2). Solar power absorbs photons from a light source (usually ultraviolet or visible light) and produces electron-electron pairs that participate in redox reactions. When producing biodiesel, this process controls the conversion of triglycerides (found in vegetable oils and animal fats) into biodiesel and glycerine. Photocatalytic distillation has many advantages, including simple reaction conditions, reduced energy consumption, and the potential of solar energy as a sustainable light source. This approach is expected to improve the efficiency of biodiesel synthesis and reduce the environmental impact associated with traditional conversion methods. Ongoing research in this area includes optimization of photocatalytic materials, reaction parameters, and scale-up to pave the way to greener and more sustainable biomass production.

Esterification plays a crucial role in the production of esters, organic compounds that find extensive applications across multiple industries. Catalysis accelerates the reaction between carboxylic acid and alcohol, producing ester and water as by-products. The general formula for the neutralization reaction is carboxylic acid + alcohol catalyst. A catalyst (usually sulfuric or hydrochloric acid) helps remove water molecules from the reactants to form the ester. Esters have a sweet odour and are easy to detect, making them useful in hunting and the arms control industry. Importantly, the esterification reaction is reversible.

Under certain conditions, the ester can be hydrolysed to the carboxylic acid and the parent alcohol. Transfection is the basic chemical process used to produce biodiesel, converting triglycerides in fats and oils into biodiesel (methyl fatty acids or ethyl esters) and glycerol. In the presence of a catalyst, the glycerol moiety of triglycerides is replaced by alcohol (mainly methanol and ethanol). Biodiesel and glycerine are the general formulas for transesterification. Enzymes (usually sodium or potassium hydroxide) aid in digestion by breaking down the complex structure of triglycerides into biomolecules and glycerol. This technology is important in the biodiesel industry because it produces high-quality biodiesel that can be used as a

renewable and clean alternative to conventional diesel fuel. Biodiesel is popular because it is compatible with a variety of raw materials, such as vegetable oils, animal fats, and recycled cooking oils, contributing to the efficiency and sustainability of biodiesel production. The by-product glycerine can be used for other purposes and adds value to the entire biodiesel production process. As biodiesel becomes increasingly popular as an environmentally friendly fuel source, research is underway to improve the efficiency and sustainability of the conversion process.

1.5 SILK COTTONSEED OIL (*ceiba pentandra* silk oil):

In biodiesel production, silk cotton seed oil extracted from *Ceiba pentandra* seeds offers several advantages. Firstly, it is a non-edible feedstock, thus avoiding competition with food crops and addressing concerns regarding food security. Additionally, silk cotton seed oil possesses favourable characteristics such as high lipid content and low free fatty acid levels, making it suitable for biodiesel production. Furthermore, *Ceiba pentandra* trees are fast-growing and resilient to harsh environmental conditions, contributing to sustainable feedstock cultivation. The use of silk cotton seed oil in biodiesel production promotes environmental sustainability by reducing greenhouse gas emissions and dependence on fossil fuels. However, challenges may arise in large-scale production, such as variability in seed oil composition and limited availability of *Ceiba pentandra* seeds in certain regions, necessitating further research and development to optimize its utilization in biodiesel production

Silk cotton seed oil extracted from *Ceiba pentandra* seeds presents several advantages for biodiesel production. Firstly, *Ceiba pentandra* trees are widely distributed in tropical and subtropical regions, ensuring a sustainable and abundant source of feedstock. Additionally, silk cotton seeds contain a high oil content, typically ranging from 18% to 30%, making them valuable for oil extraction purposes. Importantly, as non-edible seeds, they alleviate concerns about competing with food crops for fuel production, promoting sustainable agricultural practices. Moreover, the low levels of free fatty acids in silk cotton seed oil reduce the need for extensive pre-processing steps, streamlining biodiesel conversion processes. Environmentally, biodiesel derived from silk cotton seed oil offers benefits such as reduced greenhouse gas emissions and decreased air pollution compared to

conventional diesel fuel. Furthermore, economically, its utilization can create opportunities for farmers, particularly in regions where *Ceiba pentandra* trees are cultivated, thus contributing to rural development and income generation.

However, several challenges accompany the use of silk cotton seed oil in biodiesel production. Firstly, the variability in oil composition due to factors like geographic location and tree genetics can impact biodiesel yield and quality, necessitating careful monitoring and optimization of production processes. Additionally, the availability of *Ceiba pentandra* seeds may be seasonal, and harvesting them can be labour-intensive, posing logistical challenges for consistent feedstock supply. Furthermore, despite its favourable characteristics, silk cotton seed oil may require specialized processing techniques to overcome challenges such as high viscosity and impurities. Competition with other uses, such as traditional medicine and fibre production, also exists, potentially affecting the availability of seeds for biodiesel production. Addressing these challenges will require further research and development to optimize production processes, improve extraction efficiency, and ensure sustainable practices throughout the supply chain.

1.6 CATALYST:

In the process of photocatalytic transesterification for biodiesel manufacture, zinc oxide (ZnO) and titanium dioxide (TiO₂) assume vital roles as catalysts, leveraging their unique attributes under light exposure. ZnO and TiO₂ serve as photocatalysts, initiating chemical reactions when exposed to light energy. Upon illumination with ultraviolet (UV) or visible light, both catalysts generate electron-hole pairs, thereby creating reactive species like superoxide radicals (O₂⁻) and hydroxyl radicals (-OH). These reactive species play a crucial role in catalysing the transesterification reaction by interacting with triglyceride molecules present in vegetable oils or animal fats. Through this process, triglycerides are converted into biodiesel and glycerol. ZnO and TiO₂ offer distinct advantages, including high surface area, stability, and cost-effectiveness, making them well-suited for catalysing the transesterification reaction under photocatalytic conditions. Additionally, their ability to generate reactive oxygen species under light illumination enhances the efficiency of the biodiesel production process. Thus, ZnO and TiO₂ play integral roles as photocatalysts in facilitating the conversion of triglycerides into biodiesel,

contributing to the advancement of sustainable and environmentally friendly fuel production technologies.

1.7 CHARACTERIZATION

Various characterization techniques are employed in biodiesel production and catalyst analysis to understand their properties and optimize production processes. For catalyst analysis, techniques such as Field Emission Scanning Electron Microscopy (FESEM) are utilized to examine catalyst surface morphology, providing insights into particle size, shape, and distribution, crucial for catalyst stability and activity assessment. Energy-Dispersive X-ray Spectroscopy (EDX) is employed to determine the elemental composition of catalyst materials, identifying active species and impurities essential for understanding catalyst performance. Additionally, Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) analyses assess catalyst surface area, pore size distribution, and volume, optimizing catalyst design for enhanced catalytic efficiency in biodiesel production.

OVAT (One Variable at a Time) experimentation is crucial in biodiesel production research. It involves adjusting one variable while keeping others constant to precisely assess its impact on yield, quality, and efficiency. By focusing on individual factors, researchers gain insights into process dynamics, simplify experimental design, and achieve cost and time efficiency. Overall, OVAT facilitates systematic exploration and optimization of biodiesel production processes.

On the other hand, for biodiesel analysis, Fourier Transform Infrared Spectroscopy (FTIR) is employed to analyse functional groups in biodiesel, confirming successful transesterification and providing insights into purity and composition. Gas Chromatography-Mass Spectrometry (GC-MS) determines the fatty acid methyl ester (FAME) profile of biodiesel, aiding in feedstock identification and assessing quality and properties crucial for engine performance. Additionally, Internal Combustion (IC) engine testing evaluates biodiesel performance under real-world conditions, assessing parameters like power output, fuel efficiency, and emissions characteristics. These combined characterization techniques play a vital role in understanding biodiesel properties, catalyst behaviour, and optimizing production processes for efficient and sustainable biodiesel production.

CHAPTER 2

LITERATURE SURVEY

Michael Rahul Soosai et al. (2022) have published a journal titled "Integrated global optimization and process modelling for biodiesel production from non-edible silk-cotton seed oil by microwave-assisted transesterification with heterogeneous calcium oxide catalyst." The creation of biodiesel, an environmentally benign and sustainable alternative to conventional diesel fuel, is the main topic of this study. Transesterification, a chemical procedure used to make biodiesel, involves reacting triglycerides (fats and oils) with an alcohol and a catalyst to create biodiesel and glycerine. In this investigation, microwave help is used throughout the transesterification process. Chemical processes can be accelerated by microwave heating, potentially increasing the yield of biodiesel. A heterogeneous calcium oxide catalyst is used to catalyse the transesterification reaction. Solid heterogeneous catalysts may be easily removed from the result and are appropriate for reuse because they are not dissolved in the reaction mixture.

Adriana Zaleska et al. (2014) have published a journal titled "Photocatalytic Conversion of Glycerol to Hydrogen in Batch and Semi-Batch Laboratory Reactors." In this study, glycerol is generated as a by-product during biodiesel production. It can be further processed to produce value-added products like glycerol carbonate or glycerol-based esters. Photocatalytic conversion of glycerol into hydrogen is a promising approach for utilizing glycerol to produce a clean energy carrier. A glycerol is exposed to light (usually UV or visible light) in the presence of the photocatalyst. The photocatalyst absorbs photons and generates electron-hole pairs, initiating redox reactions.

Mohammad M. Hussain et al. (2020) have published a journal titled "Application of Heterogeneous Catalysts for Biodiesel Production from Microalgal Oil." In this study, the advancement of efficient and economical catalysts with an environmentally friendly action is capital to address the problems associated with biodiesel assembly from microalgal oil. Recently, admitting a few difficulties, heterogeneous catalysts have appeared to have an advantage over constant catalysts for biodiesel

production from third-bearing feedstocks like microalgae. Acid-base catalysts are one of the abeyant catalysts due to their adeptness to activate both chargeless free fatty acids (FFAs) in esterification reactions and triglycerides (TG) in transesterification reactions at the aforementioned time. For an effective bartering catalyst, recyclability, lower costs, and agitator lifetime are awful capital, as these accept an above-board approach to the total amount of action accompanying biodiesel production.

David W. Rooney et al. (2023) have published a journal titled "Biofuel production, hydrogen production, and water remediation by photocatalysis, biocatalysts, and electrocatalysis." In this study, photocatalysis can be employed to convert biomass, such as lignocellulosic materials or algae, into biofuels like bioethanol or biodiesel. Photocatalysis can be used to split water into hydrogen and oxygen using sunlight. Biocatalysts can involve the use of enzymes, microorganisms, or plants to degrade or remove pollutants from water. Electrocatalysis is used in electrochemical water treatment systems, such as electrocoagulation and electrooxidation, to remove pollutants. In this process, an electric current is passed through water using an electrolyser, which contains electrocatalysts at the anode and cathode.

Zahra Moafi et al. (2023) have published a journal titled "Biodiesel Production Using a Photocatalytic Process." In this study, the generated electron-hole pairs participate in a series of redox reactions. The triglycerides undergo transesterification with the alcohol to produce biodiesel (methyl or ethyl esters) and glycerol, just as in traditional biodiesel production. After the photocatalytic reaction, the mixture is typically separated into biodiesel, glycerol, and any unreacted starting materials. The crude biodiesel produced in this process may require further purification steps, such as washing and drying, to remove impurities and residual catalysts. The photocatalyst can often be regenerated and reused, reducing the need for continuous catalyst replacement.

M. Bowker et al. (2016) The publication released a journal named "Production of H₂ through the photocatalytic conversion of cellulose and untreated biomass utilizing Ni, Pd, Pt, and Au on titania." In this study, we take Titania (TiO₂) is often used as a photocatalyst due to its ability to absorb UV light effectively. Additionally, noble metal

nanoparticles (Ni, Pd, Pt, and Au) are deposited or loaded onto the TiO₂ surface to enhance its photocatalytic activity. Cellulose, or raw biomass, is typically pre-treated to break it down into smaller, more reactive components. This can involve mechanical or chemical treatments to increase the accessibility of the biomass to the catalyst. The photocatalytic reforming reaction takes place in a reactor or chamber under controlled conditions. When the catalyst is exposed to UV or visible light, it absorbs photons and generates electron-hole pairs, initiating various chemical reactions with the biomass.

Jinshu Huang et al. (2022) “Research Progress on the Photo-Driven Catalytic Production of Biodiesel”. In this study they had Compared to accepted thermal-driven catalysis, photo-driven catalysis has the advantages of green, environmental protection, and energy-saving by using Cr/SiO₂ catalyst, ZnO/SiO₂ catalyst and COF-based photocatalysts.

Abdul Basit Naveed et al. (2023) “TiO₂/g-C₃N₄ Binary Composite as an Efficient Photocatalyst for Biodiesel Production from Jatropha Oil and Dye Degradation”. In this study It is acclaimed that the nanocomposites acquire decidedly high photocatalytic activities and the present abstraction reveal that the synthesis of nanocomposites for biodiesel assembly an organic dye abasement is an able adjustment to cope with the challenges accompanying to blooming activity and an apple-pie environment with the use of g-C₃N₄/TiO₂ composite material.

Marek Stelmachowski et al. (2014) “The Photocatalytic Conversion of (Biodiesel Derived) Glycerol to Hydrogen”. In this study the Pt-TiO₂ photocatalysts accept a best hydrogen change amount up and the ability of hydrogen production was afflicted by glycerol antecedent concentration and photocatalysts content with the use of Pt-TiO₂ photocatalysts.

Ahmed I. Osman et al. (2023) “Biofuel production, hydrogen production and water remediation by photocatalysis, bio catalysis and electrocatalysis”. In this study Recent advancements in photocatalysis, bio catalysis and electrocatalysis accept

been evaluated, and the challenges faced in ecology catalysis accept been identified. Research is currently focused on developing efficient photocatalysts through doping, coupling with added nanomaterials, precipitation with metal particles, clear advance designs and heterojunctions by using Heterojunction-based photocatalysts, Electrocatalysis and Bio catalysis.

c.Hardacre et al. (2016) “H₂ production by the photocatalytic reforming of cellulose and raw biomass using Ni, Pd, Pt and Au on titania” In this study, we clearly demonstrated that hydrogen can be produced by photo modifying a mixture of cellulose and water over a TiO₂ catalyst supported with metal nanoparticles. Importantly, catalysts based on non-noble and platinum group metal materials are highly effective. This process has high potential for realizing an efficient hydrogen economy, as hydrogen can be produced directly from the main component of biomass (cellulose).

Dominic Okechukwu Onukwuli et al. (2016) “Optimization of biodiesel production from refined cotton seed oil and its characterization”. In this study the assay of about-face (ANOVA) showed that a satisfactory aftereffect was obtained. Moreover, accretion both temperature and KOH absorption college about-face amount was accomplished and time does not assume to accept any significant effect with the help of KOH catalyst.

Omkaresh B R et al. (2021) “Development of heterogenous photocatalytic method for biodiesel production. In this study the actinic CaTiO₃ NPs shows authentic distinct orthorhombic phase. All the diffraction peaks at (101), (121), (404), (321) and (242) were able-bodied indexed and akin with the accepted JCPDS. The actual has amassed structures with baby bean like and cool morphologies. The activity dispersive X-ray spectrum of CaTiO₃ sample assures the actuality of Ca, Ti and O₃ at adapted concentrations by using CaTiO₃.

Hendro Juwono et al. (2023) “Facile Production of Biodiesel from Candlenut Oil (*Aleurites moluccana* L.) Using Photocatalytic Method by Nano Sized-ZnO Photocatalytic Agent Synthesized via Polyol Method” In this study The assembly of biodiesel from candlenut oil application ZnO via photocatalytic-assisted transesterification has been auspiciously agitated out. ZnO was able via the polyol adjustment and apparent a nanoparticle admeasurement of 20–200 nm and an apparent breadth of $15.5 \text{ m}^2 \text{ g}^{-1}$. The pore admeasurement administration of ZnO was mostly about 10–20 nm. Besides, assorted morphologies were generated by polyol methods, including spherical, rods, and hexagonal.

Murat Efgan Kibar et al. (2023) “Assessment of Homogeneous and Heterogeneous Catalysts in Transesterification Reaction”. In this study the absolute after-effects of the biodiesel produced application amalgamate catalysts with altered feedstocks and types of catalysts acclimated were presented. The accent of the alternation amid an agitator and an abutment for amalgamate agitator was additionally discussed. It can be assured that bearing bio-diesel sustainably and economically is an actual adorable ambition that will account humankind’s charge for activity in the approaching and amalgamate catalysts will be the best adorable allotment of the accomplished process.

Claudia Carlucci et al. (2019) “Titanium Dioxide as a Catalyst in Biodiesel Production”. In this study the advantageous in assessing the accessible catalytic pathways in the assembly of biodiesel, exploring decidedly the use of titanium dioxide as catalyst. Furthermore, this assignment focused on the abstraction of the altered strategies to conduct the transesterification acknowledgment advised by titanium dioxide as agitator aural microreactors. The use of micro-technologies in biodiesel assembly and the development of micro-reactors, able of authoritative accessible unpracticable actinic transformations application acceptable techniques, allows an abridgement in assembly costs and a greater aegis of the environment.

Ranggaweny Al-Ghani et al. (2023) “Design of Biodiesel Reactor Using Nano catalyst ZnO from Castor Oil”. In this study the abstraction aims to architecture an architecture for the accomplish of zinc oxide (ZnO) nanocrystals application a

connected afflicted catch basin reactor (CSTR) reactor. By giving abstracts on incoming feedstock and artefact breeze produced by the reactor, accumulation antithesis calculations were acclimated in this study as a criterion to actuate whether the reactor was operating appropriately.

Jabbar Gardy et al. (2016) “Synthesis of $\text{Ti}(\text{SO}_4)_2$ solid acid nano-catalyst and its application for biodiesel production from used cooking oil”. In this study the synthesis of titanium sulphate oxide or titanium oxide sulphate $[\text{Ti}(\text{SO}_4)_2]$ nano-catalyst, which can be used as a solid super acidic heterogeneous catalyst for a variety of applications due to its high activity and stability, has been made possible by employing a novel method of impregnating a sulphate group into TiO_2 -NPs. In comparison to other metal oxide catalysts that have been reported, the nano-catalyst has superior functionality, significantly improved process conditions, and a higher tolerance to FFA. It can be recycled and reused up to eight times without deactivating the active sites. Additionally, the specifications of the produced biodiesel fall within acceptable biodiesel limits after being analysed in accordance with the ASTM and EN standards.

Hang Lu et al. (2001) “Photochemical reactions and on-line UV detection in microfabricated reactors”. In this study the possibility of increasing the efficiency of photon transfer and reactions is one of the advantages of small-scale reactors. Microreactors specifically provide the opportunity to “thin” optically thick solutions in order to make the most of the incident light. Crystallization can be kept away from in the reactor with a nonstop stream framework by controlling residence time, and subsequently the degree of response on-chip, simplifying downstream cycles. In manufacturing these micro reaction gadgets, we have exhibited a low temperature holding of quartz substrates to miniature machined silicon gadgets with a per fluorinated polymer — CYTOP™. In view of its dormancy and cycle effortlessness, CYTOP™ may end up being helpful in numerous other microreactor applications.

Apoorva Maheshwari et al. (2020) “Scalability of photochemical processes”. In this study they demonstrated how a photochemical decatungstate-catalysed, oscillatory plug flow photoreactor can be used to scale up a C(sp³)-H alkylation. Response transformation and yield were examined by changing different boundaries, including home time, throb power, light force, likeness cyclohexane, TBADT stacking and convergence of the substrate. With full substrate conversion and a residence time of 7.5 minutes, we were able to achieve 8.7 g/h, or 36.7 mmol/h, of productivity following the optimization study. This examination exhibited the efficiency of the HANU™ reactor and its scale-up capacities.

Yan Xiong et al. (2021) “Photo-Driven Catalytic Hydrogenation of CO₂ to CH₄ with nearly 100% Selectivity over Ag₂₅ Clusters”. In this study the TDDFT excitation energies of Ag₂₅-CO₂ complex in initial four energized states were 634.22, 633.38, 627.45, and 611.33 nm, separately, which were like those of the initial four invigorated conditions of uncovered Ag₂₅ cluster (637.48, 633.61, 630.51, and 612.72 nm, separately). It could likewise be seen from the front facing orbitals graph That the intramolecular charge-move change comes from the ligand to the metal iotas. Also, the HOMO-LUMO hole of Ag₂₅-CO₂ complex (2.50 eV) was near that of Ag₂₅cluster(2.49eV), demonstrating that the actual adsorption of CO₂ on Ag₂₅ bunch.

CHAPTER 3

AIM AND SCOPE

AIM

- The aim of the study is to produce biodiesel from cotton seed oil utilizing the impregnation method for catalyst preparation with ZnO and TiO₂ nanoparticles.
- Additionally, the study aims to optimize reaction conditions for biodiesel production, including catalyst loading, alcohol to oil ratio, reaction temperature, and reaction time.
- Furthermore, efficient storage techniques for glycerol, a byproduct of the transesterification reaction, will be investigated to ensure stability and potential reuse or appropriate disposal.

SCOPE

- The future of biodiesel has great potential as the world shifts its focus to sustainability and renewable energy alternatives.
- As technology advances and environmental concerns grow, biodiesel has a key role to play in the transition to renewable energy.
- As research continues to improve biodiesel production efficiency and feedstock diversity, biodiesel will become even more cost-effective and sustainable. Governments are tightening emissions regulations and encouraging renewable energy adoption.
- Biodiesel demand is expected to continue to grow as the market continues to expand.
- This expanding market provides a great opportunity for innovation and capital investment.
- All in all, the future of biodiesel looks promising, providing a sustainable answer to tomorrow's energy challenges while promoting economic growth and environmental responsibility.

CHAPTER 4

MATERIALS AND METHODOLOGY

4.1 Materials and Equipment

Material required:

- Silk cotton seeds
- Zinc oxide (ZnO)
- Titanium dioxide (TiO₂)
- Distilled water
- Methanol
- Silk cotton seed oil

Equipment's required:

- Mini mechanical screw press
- Stainless-steel barrel
- Hot plate magnetic stirrer
- Ultra sonicator
- Magnetic pellet
- Hot air oven
- Photocatalytic reactor
- Magnetic pellet
- Separatory funnel
- Round Bottom Flask with Heating Mantle

4.2 Oil preparation

Acquiring: Cottonseed oil is extracted from the seeds of the cotton plant. The cotton seeds are collected from the nearby store.

Drying: The oil extraction process began with sun-drying 40 kg of *C. pentandra* seeds to eliminate moisture content.

Seed Crushing: The dried cotton seeds were then crushed to break them into smaller pieces or flakes. This increases the surface area of the seeds, facilitating the extraction of oil.

Oil Extraction: The crushed cotton seeds were then transferred to mini mechanical screw press. This screw press is characterized by a helical-shaped thread with five threads and a barrel bore diameter of 60 mm, along with a drainage length of 155 mm.

Oil impurity purification: Followed by extraction, the CPSO was stored in a stainless-steel barrel for a duration of 96 hours to facilitate the settling of impurities. Subsequently, the upper layer of the CPSO was isolated for the further experimentation.

4.3 Catalyst Preparation:

Selection of Catalyst: ZnO and TiO₂ were selected as the catalyst for our project this is due to their unique properties that make them effective for the photocatalytic processes. Both ZnO and TiO₂ have wide bandgaps, which means they can absorb a significant portion of the solar spectrum, including ultraviolet (UV) radiation and for their excellent photostability, meaning they can maintain their structural integrity and catalytic activity under prolonged exposure to light.

Precursor & Support Material Selection: Precursor is nothing but a starting material or an active substance. In this study we have used two components (ZnO & TiO₂). The selection of active substance is based on their electronic structure and chemical properties of these materials. On the basis of photocatalytic property ZnO is generally considered less active than TiO₂ due to factors such as its wider bandgap energy, lower charge carrier mobility, and relatively lower stability under photocatalytic conditions. Therefore, TiO₂ is considered to be the active substance and the ZnO is the support material.

Catalyst Impregnation: Impregnation is a technique used in many fields, such as materials science, chemistry, and engineering. A process in which a substance (the impregnant) is introduced into the pores or cracks of another material (the matrix) to change its properties, improve its performance, or achieve a specific result. These

can be incorporated into solids such as porous materials, powders, and fibres. For impregnating the compounds ZnO and TiO₂ were available in the college laboratory.

10 g of TiO₂ were weighed in a weighing machine and transferred to a conical flask. And the conical flask was placed on a magnetic stirrer with a hot plate and run for 10 minutes. After 10 minutes, ZnO was weighed to 10g and added to the same conical flask. This mixture is stirred for 2 hours. And then it was placed in an ultrasonicator, at 40°C for 20mins. Again, the mixture was stirred for 2 hours in a hot plate magnetic stirrer. After 2 hours, the mixture was filtered by filter paper and the catalyst particles were settled on the filter paper.

Drying: After impregnation, the settled particles in filter paper were dried in a hot air oven at 80°C for 120 mins.

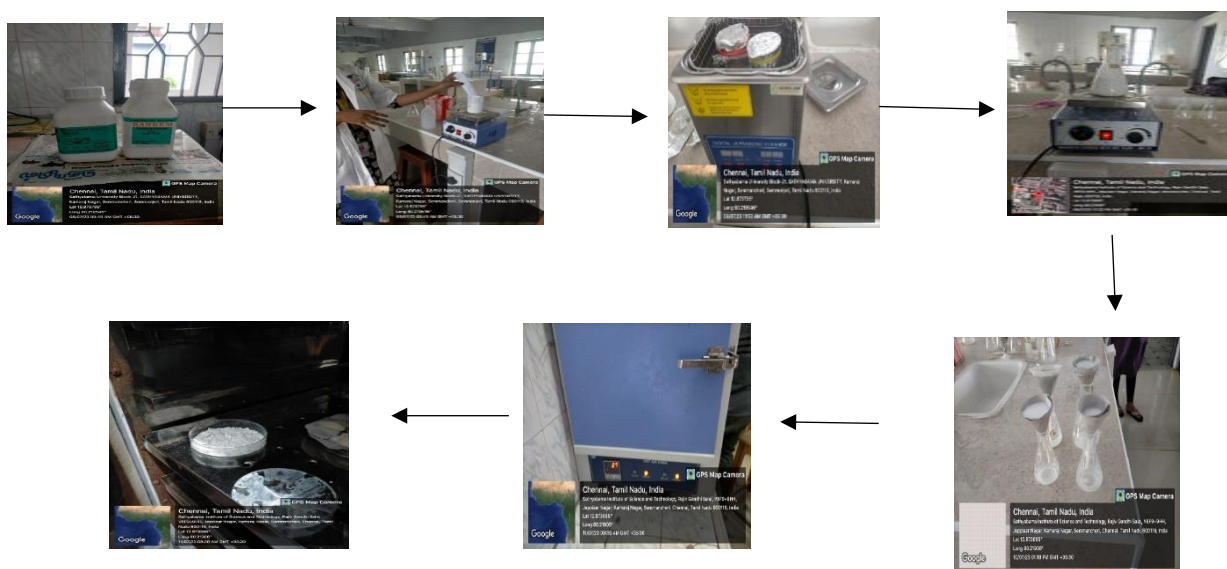


Fig 4.1 Overall pictorial demonstration of catalyst preparation

4.4 Biodiesel Preparation:

Photocatalytic transesterification: A screw cap bottle is filled with 55.26 g pure methanol and 60 g free fatty acid (Cottonseed oil) with 5.18 g catalyst. These calculations are obtained from the optimization study. These catalysts are able to absorb uv light energy and start the chemical reaction. Heat the triglyceride (vegetable oil or animal fats) to reduce the viscosity of the triglyceride. This step is

needed to mix the reactants and make the reaction more efficient. The methanol is the reactant and participates in the reaction to make biodiesel.

Next, the screw cap bottle is covered with aluminium foil, drill a few holes into the aluminium foil. The screw cap bottle is placed on a water bath and place it on the photocatalytic reactor then turn on the UV light, and the water bath is allowed to remain at room temperature for 3 hours. The biodiesel is poured into a graduated cylinder and allow it to settle. The layers (biodiesel glycerol) and volumes (biodiesel, glycerol) of the biodiesel were measured.

Separation: By using filter paper, the catalyst from the mixture were separated. Then it is poured on the separatory funnel. The reaction mixture is allowed to settle and separated into two phases: the upper phase containing biodiesel (FAMES or FAEEs) and the lower phase containing glycerol, a by-product of the photo transesterification reaction.

Glycerol Removal: The glycerol layer from the bottom of the reaction vessel were removed. The removed glycerol can be used for other purposes.

Washing: The separated oil is washed three times with the separatory funnel to eliminate impurities and unreacted alcohol.



Fig 4.2 Overall pictorial demonstration of biodiesel preparation

4.5 Catalyst characterization

FTIR spectrometers are employed to analyze the absorption of infrared light by molecules in a sample, providing insights into its chemical composition. SEM instruments produce high-resolution images of a sample's surface by scanning it with a focused beam of electrons, revealing detailed morphological information.

BET analyzers are utilized to measure the specific surface area of materials through gas adsorption, commonly applied in characterizing porous materials like catalysts. BJH analyzers complement BET analyzers by analyzing the pore size distribution within a sample.

EDX spectrometers detect characteristic X-rays emitted by a sample when bombarded with high-energy electrons, enabling elemental analysis. These instruments are often integrated into SEM setups for simultaneous imaging and elemental mapping.

To analyze the data generated by these instruments, specialized software is employed. This software facilitates tasks such as spectrum analysis, image processing, surface area calculations, pore size distribution analysis, and elemental mapping, depending on the specific technique utilized.

4.6 One variable at a Time Approach

The OVAT framework has served as a guiding roadmap for my research endeavors. This framework systematically delineates the process into four fundamental elements: objective, variables, agreement, and timeframe. Establishing a clear objective was deemed paramount, encompassing tasks such as ensuring adherence to regulatory standards, evaluating product quality, or refining production processes. Through the graphical representation of molar ratio against yield, insightful observations regarding biodiesel production efficiency were made. Detailed analysis of neighbouring ratios and exploration of factors influencing yield contributed to process refinement. Similarly, in the investigation of catalyst weight percentage against yield, significant insights regarding optimal concentration, efficiency, and reaction sensitivity were garnered. Furthermore, the plotting of reaction time against yield facilitated the identification of trends in optimal reaction

duration for maximizing yield, thereby ensuring the reliability and scalability of our analysis in OVAT studies.

4.7 Biodiesel characterization

In biodiesel characterization, understanding its chemical composition and purity is essential for assessing its quality and suitability for various applications. Two widely used techniques for this purpose are FTIR (Fourier Transform Infrared Spectroscopy) and GC-MS (Gas Chromatography-Mass Spectrometry).

FTIR spectroscopy is employed to analyze the functional groups present in biodiesel molecules. By measuring the absorption and transmission of infrared light by the sample, FTIR provides valuable information about the molecular structure of biodiesel. Specifically, it helps identify key chemical bonds such as those associated with esters ($\text{C}=\text{O}$), hydrocarbons ($\text{C}-\text{H}$), and alcohols ($\text{O}-\text{H}$). These functional groups are characteristic of biodiesel compounds, allowing analysts to determine parameters such as ester content, degree of unsaturation, and the presence of impurities or contaminants.

On the other hand, GC-MS is a highly sensitive technique used to analyze the composition of biodiesel at a molecular level. It involves two main steps: gas chromatography separates individual components of the biodiesel sample based on their volatility, while mass spectrometry identifies and quantifies these components based on their mass-to-charge ratios. In the case of biodiesel, GC-MS is particularly useful for analyzing fatty acid methyl esters (FAMES), which are the primary components of biodiesel derived from the transesterification of vegetable oils or animal fats. GC-MS analysis allows for the identification and quantification of specific FAMES present in biodiesel samples, as well as the detection of impurities such as glycerol, free fatty acids, and other contaminants.

4.8 IC Engine characterization

The IC engine under test is a Research Diesel engine, designed to deliver 3.50 kW of power at 1500 rpm. It operates as a single-cylinder, four-stroke engine with a constant speed. The engine is water-cooled and features a cylinder bore of 87.50 mm and a stroke length of 110.00 mm. Its connecting rod length measures 234.00 mm, while the compression ratio stands at 17.50, resulting in a swept volume of 661.45 cc.

For combustion parameters, specific gas constant is set at 1.00 kJ/kgK, with an air density of 1.17 kg/m³. The adiabatic index is 1.41, and the polytropic index is 1.11. The engine operates through 10 cycles, with a cylinder pressure reference of 7 and a smoothing factor of 2. The top dead center (TDC) reference is at -223 degrees.

Regarding performance parameters, the engine is equipped with an orifice diameter of 20.00 mm, along with an orifice coefficient of discharge set at 0.60. The dynamometer arm length is 185 mm, and the fuel pipe diameter measures 12.40 mm. Operating conditions are based on an ambient temperature of 27 degrees Celsius. The engine runs on diesel fuel, with a fuel density of 830 kg/m³ and a calorific value of 42000 kJ/kg. Additionally, there are 360 pulses per revolution. These parameters collectively provide a comprehensive characterization of the engine setup and its operational environment.

CHAPTER 5

RESULTS AND DISCUSSION

5.1 OIL CHARACTERIZATION

5.1.1 *Ceiba pentandra* seed oil extraction

Ceiba pentandra, commonly known as the kapok tree, yields seeds that are processed to extract oil using a screw press. The seeds undergo a meticulous preparation process involving harvesting and cleaning to eliminate impurities. Subsequently, the cleaned seeds are fed into the screw press in batches to ensure consistent extraction. Within the press, mechanical pressure is applied, causing the oil to be expelled from the seeds. The extracted oil is then separated from the solid residue, typically in the form of seed cake or meal. After separation, the *Ceiba pentandra* seed oil is collected and subjected to filtration to remove any remaining particles or impurities, ensuring a high-quality product. The yield of oil obtained from the extraction process is calculated, with an example yield of 18.3% from 40 kg of seeds.

Yield determination: The yield of CPSO was calculated using the below equation.

$$\text{Oil Yield (\%)} = (\text{Weight of Oil Extracted (kg)} \div \text{Weight of Seeds (kg)}) \times 100$$

The weight of the oil extracted = 7.35 kg

The weight of the cotton seed = 40 kg

By using the above equation, the yield of oil is 18.38%

5.1.2 *Physio-chemical properties*

The quality of CPSO (*Ceiba Pentandra* Seed Oil) is crucially defined by its acid value, serving as an indicator of both its purity and quality. High acid values in feedstocks can trigger side reactions, such as saponification, during transesterification processes, consequently diminishing the biodiesel yield. Moreover, biodiesel derived from acidic feedstocks has been noted to potentially

obstruct injectors and induce engine corrosion. The acid value also significantly impacts the Free Fatty Acid (FFA) content. In the case of CPSO, it exhibited an acid value of 13.67 mg KOH/g oil and an FFA content of 6.87%. To enhance biodiesel yield, it is imperative to pre-process CPSO via acid-catalysed esterification to reduce the FFA content to below 1%.

The iodine value serves as an indication of the unsaturated fatty acid content in the feedstock. Oils are categorized based on their iodine value into drying oils (above 125 g I₂/100 g oil), semi-drying oils (110–125 g I₂/100 g oil), and non-drying oils (below 110 g I₂/100 g oil). CPSO exhibited an iodine value of 98.9 g I₂/100 g oil, similar to the 91.6 g I₂/100 g oil reported for *Moringa stenopietala* oil. This places CPSO in the non-drying oil category. Due to its significant carbon double bond content, CPSO can be stored for extended periods without the addition of antioxidants.

The peroxide value provides insight into the hydroperoxide content within the feedstock. Hydroperoxide plays a role in the polymerization process during transesterification, which can lead to the formation of sediments and gums. These sediments have the potential to clog the filter of an internal combustion engine during combustion. CPSO demonstrates a low peroxide value of 3.29 meq O₂/kg oil, indicating robust oxidation stability of the feedstock. This value is comparable to the 2.16 meq O₂/kg oil reported for CPSO.

Additionally, the oil's refractive index (RI) is influenced by factors such as conjugation, unsaturation, molecular weight, and fatty acid chain length. Non-edible oils typically exhibit a refractive index ranging between 1.447 and 1.482. The RI of CPSO, measured at 1.464, falls within this range. It's important to note that the oxidative stability of biodiesel is reliant on the unsaturation level of the feedstock.

The saponification value (SV) represents the amount of KOH in milligrams required to convert 1 gram of oil into soap. This value offers insights into the feedstock's fatty acid chain length and molecular weight. An SV below 195 mg KOH/g oil suggests a high molecular weight feedstock, while a value exceeding 300 mg KOH/g oil indicates a low molecular weight feedstock more suitable for soap production. CPSO exhibits an SV of 189.14 mg KOH/g oil, indicating the presence of high

molecular weight fatty acids. Consequently, CPSO has a lower probability of forming soap and is better suited for the transesterification process, as noted by Haile et al. in 2019. Utilizing the SV, the molecular weight of CPSO was calculated to be 959.13 g/mol.

5.1.3 FTIR (Fourier Transform infrared spectroscopy)

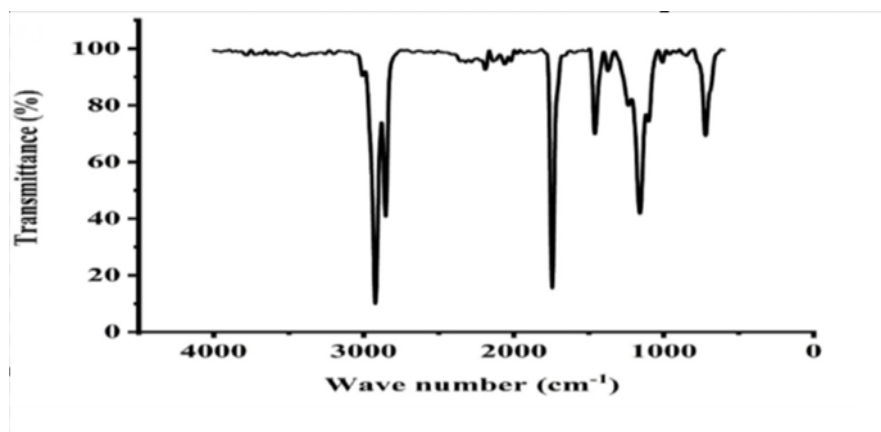


Fig 5.1 FT-IR for oil

The Fourier Transform Infrared (FT-IR) analysis of *Ceiba pentandra* seed oil (CPSO) revealed several significant peaks, each corresponding to specific functional groups present in the molecular structure. The major peaks observed at 2922.16 cm^{-1} , 2854.65 cm^{-1} , and 1458.18 cm^{-1} are indicative of C-H stretching vibrations, particularly associated with alkane ($-\text{CH}_2-$) groups present in the hydrocarbon chains of fatty acids. The peak observed at 1743.65 cm^{-1} corresponds to C=O stretching vibrations, suggesting the presence of ester functional groups characteristic of fatty acids. This peak indicates the existence of carboxylic acid moieties within the molecular structure of CPSO, which is consistent with its composition as a triglyceride-rich oil.

Furthermore, the peaks at 1159.22 cm^{-1} and 1236 cm^{-1} are associated with C-O stretching vibrations, further confirming the presence of ester groups in the CPSO molecules. These peaks specifically suggest 1, typical of triglyceride molecules found in vegetable oils. Lastly, the peak observed at 721.38 cm^{-1} indicates C-C bending vibrations, which further supports the presence of carbon-carbon bonds

within the CPSO molecules. This peak is characteristic of the backbone structure of fatty acids and contributes to the overall structural analysis of the oil. In summary, the FT-IR analysis of CPSO provides valuable information regarding its molecular composition, particularly highlighting the presence of alkane, ester, and carbon-carbon functionalities, which are typical of triglyceride-rich oils like CPSO.

5.1.4 GC-MS (Gas chromatography mass spectroscopy)

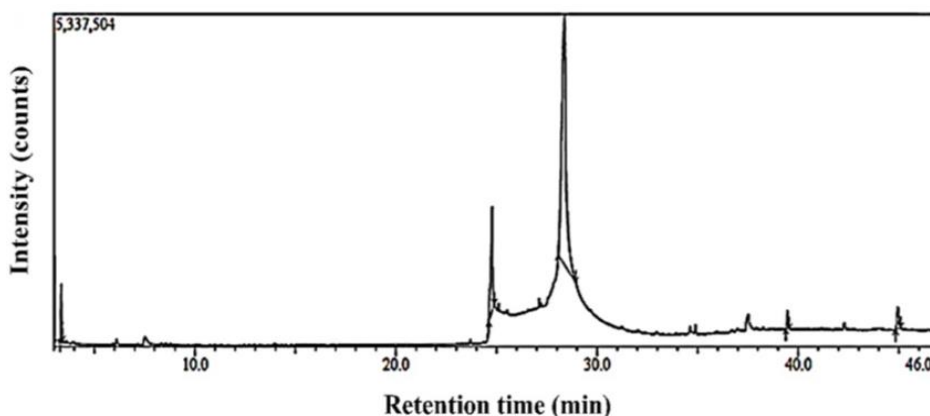


Fig 5.2 GC-MS for oil

The Gas Chromatography-Mass Spectrometry (GC-MS) analysis conducted on CPSO biodiesel elucidated that the predominant component of its fatty acid makeup consists of unsaturated acids, constituting 78.34% of the total composition. Linoleic acid emerged as the most abundant among these, representing 13.94%. This essential omega-6 fatty acid, commonly sourced from plant oils, serves crucial roles in various biological functions. Its incorporation into CPSO biodiesel contributes significantly to its fluidity and stability, attributes highly sought after in biodiesel production. Additionally, the GC-MS analysis highlighted the presence of n-hydroxytryptophan (NHTP) as the primary unsaturated fatty acid in CPSO-derived biodiesel. NHTP, an omega-3 fatty acid known for its monounsaturated nature and potential health benefits, further enhances the fatty acid profile of CPSO biodiesel.

In summary, these findings underscore the favorable fatty acid composition of CPSO-derived biodiesel, indicating its potential as a renewable and sustainable fuel source with versatile applications. Moreover, the balance between unsaturated and saturated fatty acids offers pivotal insights into the distinctive characteristics of

CPSO-derived biodiesel. The prevalence of unsaturated fatty acids hints at its superior performance in terms of cold flow properties and operational adaptability, particularly in lower temperature environments.

Additionally, the incorporation of saturated fatty acids enhances the biodiesel's resistance to oxidation and augments its combustion attributes. Together, the revelations derived from the GC-MS analysis provide a nuanced understanding of the intricate fatty acid profile of CPSO biodiesel, underscoring its potential as an ecologically sound and renewable fuel alternative suitable for a wide array of applications spanning transportation and industrial sectors.

5.2 CATALYST CHARACTERIZATION

5.2.1 Field emission scanning electron microscopy (FE-SEM)

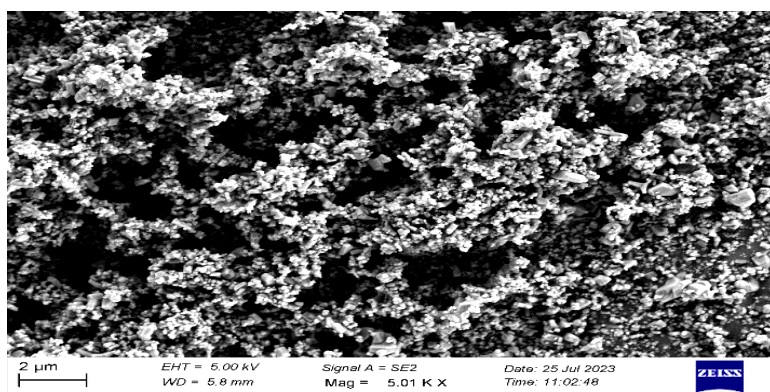


Fig 5.3 FE-SEM image of TiO₂/ZnO composite catalyst

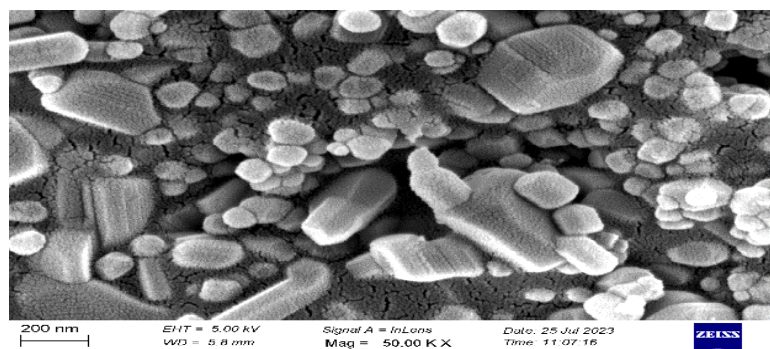


Fig 5.4 FE-SEM image of TiO₂/ZnO composite catalyst

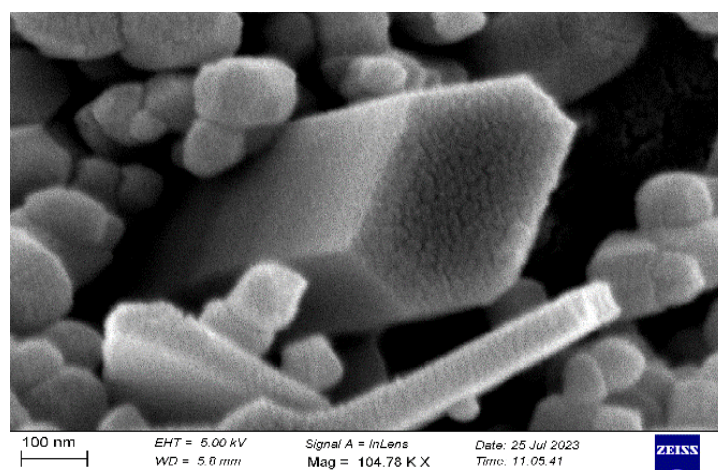


Fig 5.5 FE-SEM image of TiO_2/ZnO composite catalyst

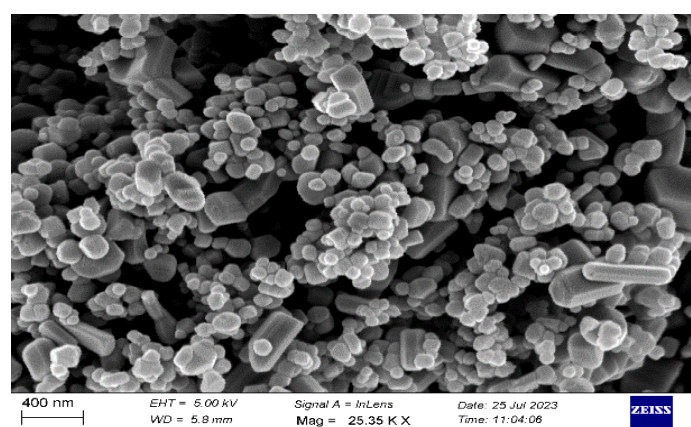


Fig 5.6 FE-SEM image of TiO_2/ZnO composite catalyst

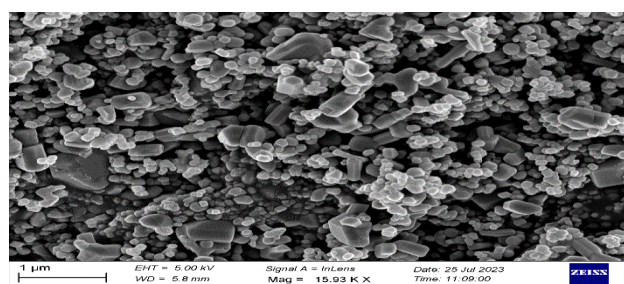


Fig 5.7 FE-SEM image of TiO_2/ZnO composite catalyst

The mixture of TiO_2 and ZnO reveals the presence of both hexagonal and tetragonal crystal structures, as observed through scanning electron microscopy (SEM).

Hexagonal and tetragonal are different crystal structures or polymorphs of a material. Polymorphism refers to the ability of a substance to exist in multiple crystalline forms while maintaining the same chemical composition. The presence of both hexagonal and tetragonal structures suggests that the material can exist in different structural configurations

It indicates that the sample contains multiple phases with distinct crystal structures. Each phase may have unique properties, such as electronic, optical, or mechanical characteristics.

5.2.2 The Energy Dispersive X-ray (EDX)

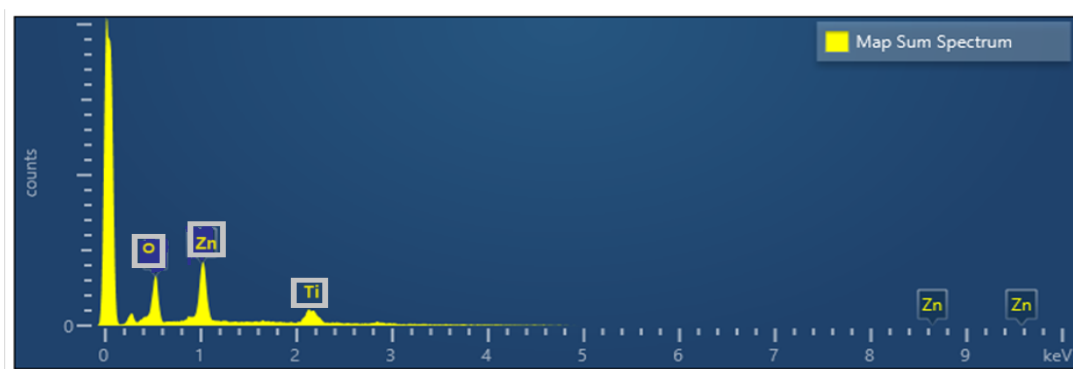


Fig 5.8 Map Sum Spectrum graph for ZnO/TiO_2

Table 5.1 Atomic% for catalyst

Map Sum Spectrum				
Element	Line Type	Weight%	Weight %Sigma	Atomic %
O	K series	29.32	0.80	62.89
Zn	L series	60.18	0.60	21.57
Ti	L series	10.5	0.20	15.54
Total		100.00		100.00

The resulting map displays the composite spectrum obtained from scanning the sample, offering insights into its elemental composition. Peaks within the spectrum indicate the presence of oxygen and zinc, crucial for discerning the chemical structure of the sample. Moreover, line types "K" and "L" correspond to the first and second energy levels, respectively, revealing details about the electronic structure and element distribution within the sample. This comprehensive analysis provides valuable information for understanding the sample's properties and characteristics.

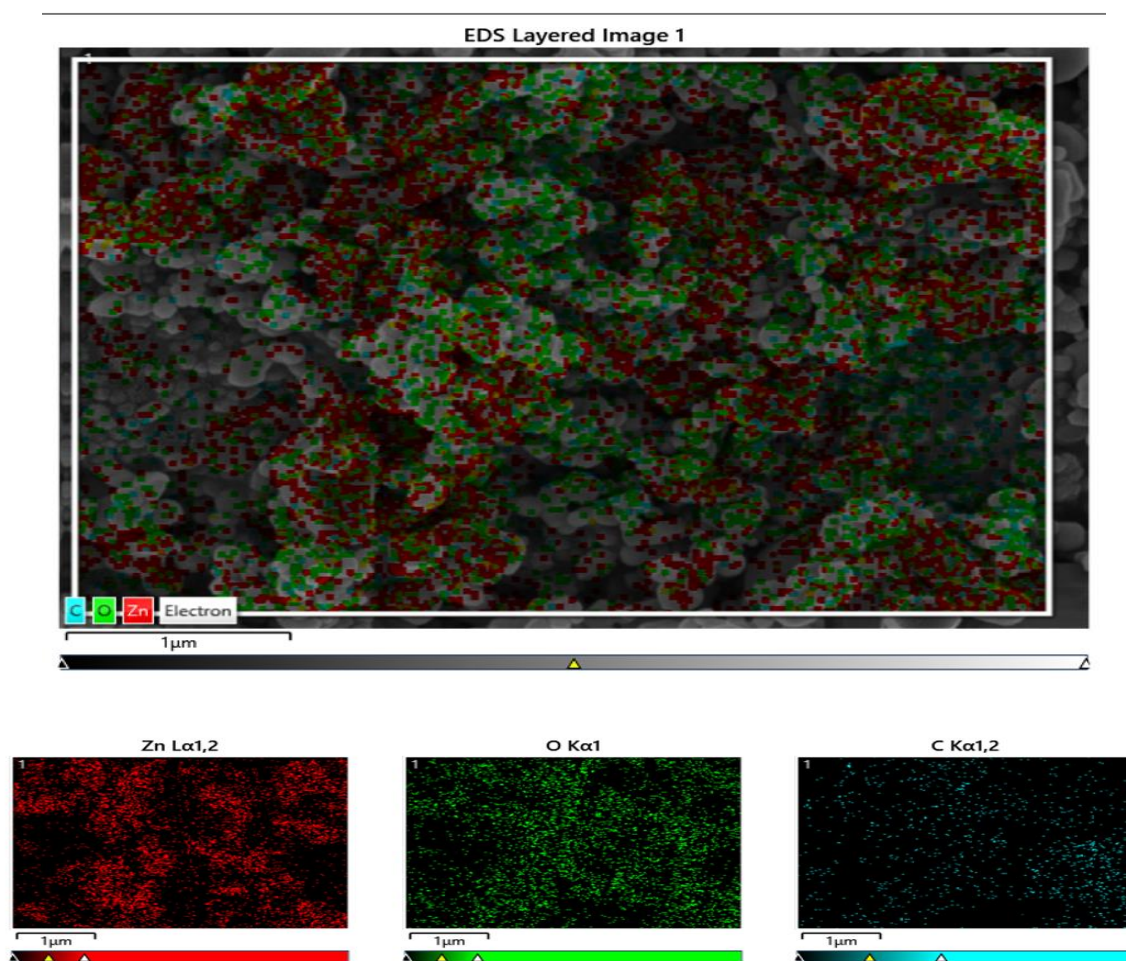


Fig 5.9 EDX elemental map

ZnO nanoparticles are composed exclusively of zinc and oxygen, whereas TiO₂ particles are composed of zinc and oxygen (with trace amounts of carbon). In cases of sample homogeneity, EDX provides elemental maps illustrating spatial distribution of elements to help characterise material heterogeneity. For

stoichiometric compounds such as ZnO or TiO₂, elemental ratios should be close to expected stoichiometrically values.

For example, in the case of ZnO, the expected ratio of zinc to oxygen is approximately 1:1, and in the case of TiO₂, the expected ratio is approximately 1:2. In addition, EDX analysis reveals the presence of titanium or zinc emission lines, which provide important information about surface values and their function as constituents in prepared ZnO/TiO₂ core / shell films, helping to understand structural and compositional properties.

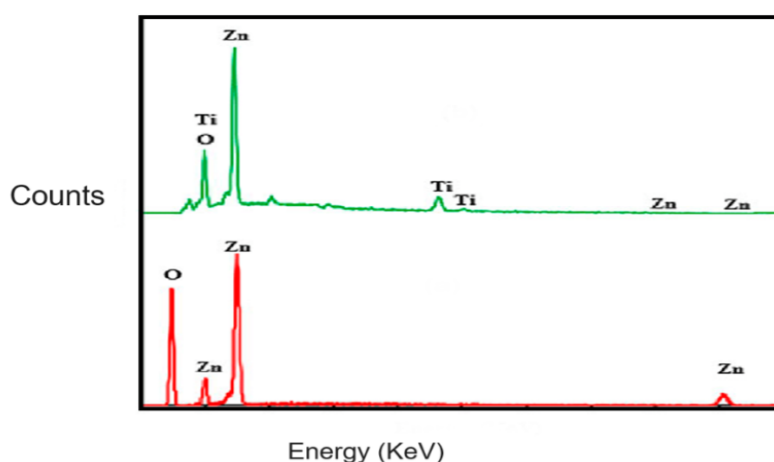


Fig 5.10 Energy Vs Counts

ZnO nanoparticles consist solely of zinc and oxygen, while TiO₂ particles comprise zinc and oxygen, with minimal traces of carbon. When dealing with sample homogeneity, Energy Dispersive X-ray Spectroscopy (EDX) offers elemental maps depicting the spatial distribution of elements, aiding in characterizing material heterogeneity. For stoichiometric compounds like ZnO or TiO₂, elemental ratios should align closely with expected stoichiometric values. For instance, in ZnO, the anticipated zinc-to-oxygen ratio is around 1:1, while in TiO₂, it's approximately 1:2. Furthermore, EDX analysis identifies titanium or zinc emission lines, furnishing crucial insights into surface values and their role as constituents in ZnO/TiO₂ core/shell films, thereby enhancing understanding of structural and compositional properties.

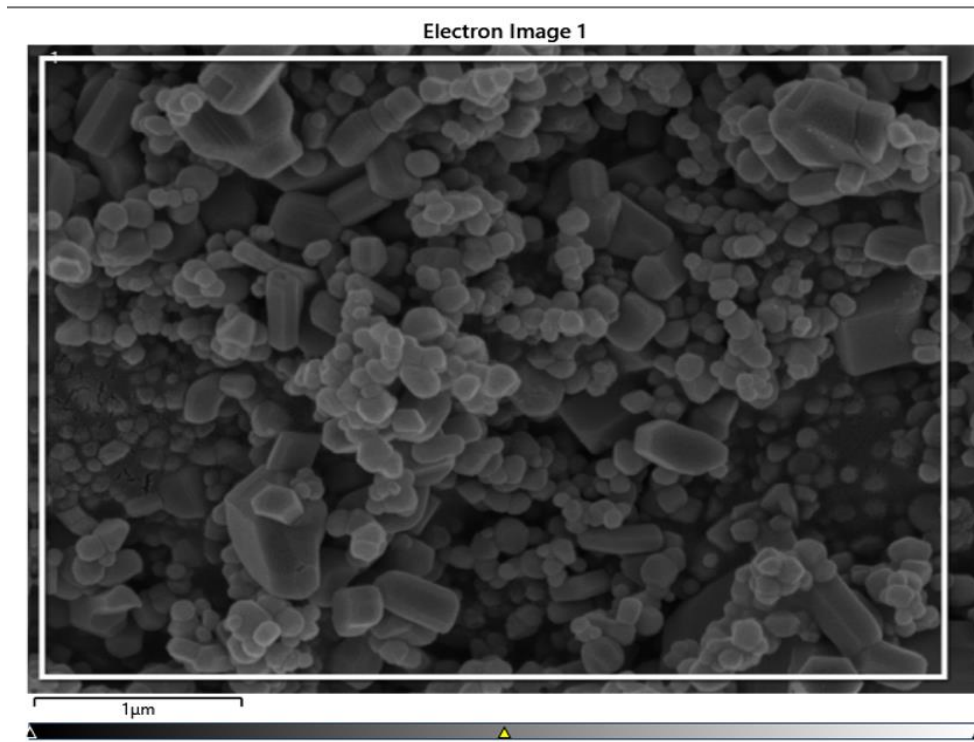


Fig 5.11 Electron Image for ZnO/TiO₂

The sample under consideration exhibits a combination of hexagonal and tetragonal crystalline structures, alongside ZnO crystalline formations. This coexistence indicates polymorphism, implying that the material can manifest in multiple crystalline configurations while retaining its chemical composition. This inherent versatility suggests that the material can assume diverse structural arrangements, each potentially endowed with distinct electronic, optical, or mechanical properties. Moreover, the presence of hexagonal crystal structures alongside titanium dioxide (TiO₂) and zinc oxide (ZnO) in this amalgamation signifies potential phase transformations under specific conditions such as temperature or pressure. Understanding these transformations is paramount for the advancement of novel materials and their utilization across various engineering applications.

5.2.3 BET(Brunauer-Emmett-Teller) Surface Area Analysis

The TiO₂ support has a surface area of 7.6 m²/g, which is similar to the 7.92 m²/g for the ZnO/TiO₂ catalyst. The "C" constant measure is a measure of the interaction of the adsorbent with the adsorbate. Nitrogen adsorption and desorption analysis were used to determine the specific surface area of the nanocomposites with ZnO-doped TiO₂ and their porous structure. These properties play an important role in improving the photocatalyst activity of the materials. The samples are mesoporous, which means they have pores with

sizes between 2 and 50 nanometres. Nucleoside isotherms exhibit a Type IV behaviour that is typical of mesoporous materials.

This means that with increasing pressure, the adsorption rate increases. When compared to pure ZnO, there are significant differences between the two. The nanocomposites have an increased specific surface area (SSA) of 11.401 m²/g of ZnO. The pore volume remains constant at 0.019 meq/g for both. The larger pores in the nano-composites allow for better electron transport through the pores, which helps to increase the catalytic activity of the materials during dye degradation. The larger surface area and active sites increase the catalytic efficiency of the materials.

This section provides a summary of the scientific research that has been done on the specific surface area of nano-composites containing ZnO, the distribution of pore size, and the role of the nano-composite in increasing photocatalyst activity.

In this section, we will also provide information on the isotherms that nitrogen adsorbs-desorbs, as well as the pore radius for synthesized materials, which were determined using the Dubinin-Astakov-Method (DA), which is a widely used approach in pore characterization studies.

5.2.4 Isothermal Curve

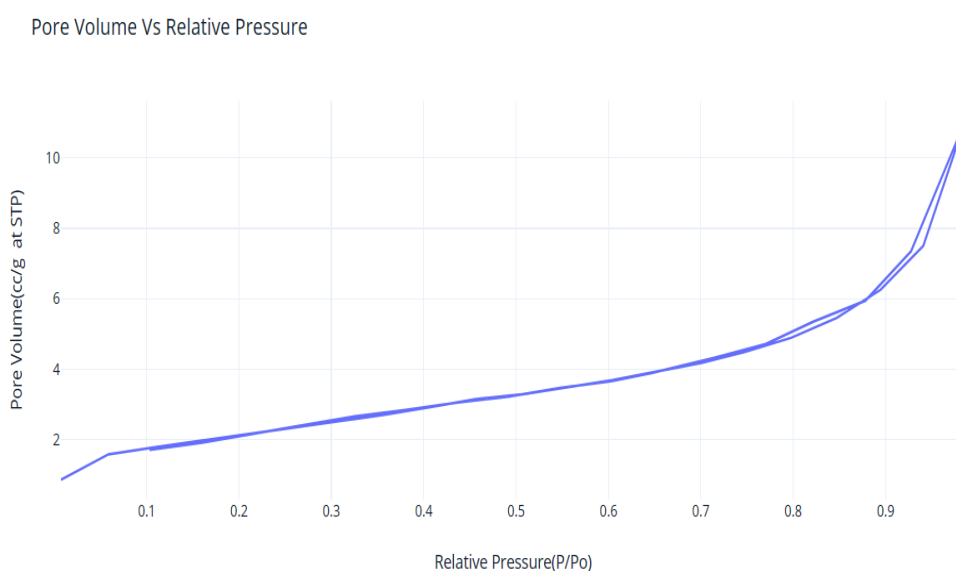


Fig 5.12 Relative Pressure vs Pore Volume

DFT pore size distribution to determine the model pore diameter Desorption vs. Relative pressure (P / P_0) of the optimal catalyst Adsorption vs. P/P_0 of the optimum catalyst the upper line represents desorption, and the lower line represents. The isothermal curve is obtained by measuring the relative pressure of the adsorbent and the corresponding volume at standardized pressure (STP). The relative pressure was measured at various values (0.007189, 0.986303), and the corresponding volumes at standardized pressure varied (0.8695, 0.9999, and 11.0512, respectively), representing the relationship between the relative pressure and the adsorption volume of the adsorbed gas. The isothermal curve is essential for the analysis of adsorption behaviour.

5.2.5 BJH(Barrett-Joyner-Halenda)

BJH stands for "Barrett-Joyner-Halenda." This is a well-known approach in materials science and the characterization of porous materials. In particular, it is used to determine pore size distribution as well as surface area for porous materials such as catalysts and adsorbents, as well as membranes.

Table 5.2 BJH desorption summary

Surface Area	11.401m ² /g
Pore Volume	0.019 cc/g
Pore Diameter (d)	2.275 nm

To determine the specific surface area of these materials, the multipoint BET method was used. The BET method analyses adsorption data at various relative pressures (P/ P_0) between 0.05 and 0.30. The pore size distribution of these materials was determined using the Desorption Isotherm Data. BJH is a widely used approach in the literature for the estimation of pore sizes in adsorption studies.

5.2.6 FTIR (Fourier-transform infrared spectroscopy)

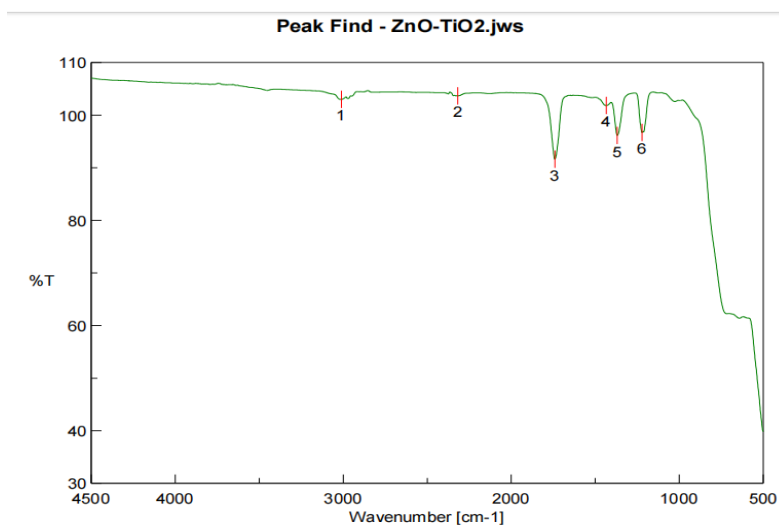


Fig 5.13 FTIR for ZnO/TiO₂

Table 5.3 Position and Intensity data

No.	Position	Intensity	Functional Group
1	3010.34	102.975	C-H stretching vibrations
2	2318.02	103.652	Alkane (C-H)
3	1740.44	91.6123	C=O stretching vibrations
4	1433.82	101.824	Nitro (NO ₂)
5	1369.21	96.1254	Ester
6	1221.68	96.6875	C=O

FT-IR is a powerful analytical tool for determining functional groups and characterizing materials. FT-IR spectrometry for TiO₂-ZnO catalysts in the spectral range 400–4000 cm⁻¹ showed distinct peaks at 1200 cm⁻¹ and 1400 cm⁻¹. These peaks were attributed to TiO₂ stretching vibrations and lattice vibrations, respectively. The band around 1300 cm⁻¹ is associated with bending vibrations of

H₂O/Ti-OH groups. The peaks at 1500 cm⁻¹ and 1400 cm⁻¹ indicate metal oxide (ZnO) formation.

In addition, both samples showed significant absorption peaks in the range of 1700-3000 cm⁻¹ that were associated with Zn–O vibration. The high purity of TiO₂-ZnO compositions in this region was further supported by peaks in the 1700-3000 cm⁻¹ range for ZnO (ZnO) and TiO₂ (TiO₂). These findings highlight the value of FT-IR analysis to characterize the TiO₂/ZnO catalyst and provide insight into its molecular structure and structure.

5.3 OVAT Studies

In biodiesel characterization, the OVAT framework acts as a valuable roadmap, delineating the process into four fundamental elements: objective, variables, agreement, and timeframe. Initially, establishing a clear objective is paramount. This may encompass ensuring adherence to regulatory standards, evaluating product quality, or refining production processes. Plotting a graph between molar ratio and yield for OVAT studies in biodiesel production provides a visual depiction of their relationship. The x-axis typically represents the molar ratio, with data points at ratios like 1:15, 1:18, 1:21, 1:24, and 1:27. Meanwhile, the y-axis displays the corresponding yield percentages, such as 57%, 59%, 62%, 67%, and 64%. This graphical representation allows for several insights to be gleaned.

Table 5.4 Molar ratio and Yield%

S.NO	MOLAR RATIO	YIELD%
1	1:15	57%
2	1:18	59%
3	1:21	62%
4	1:24	67%
5	1:27	64%

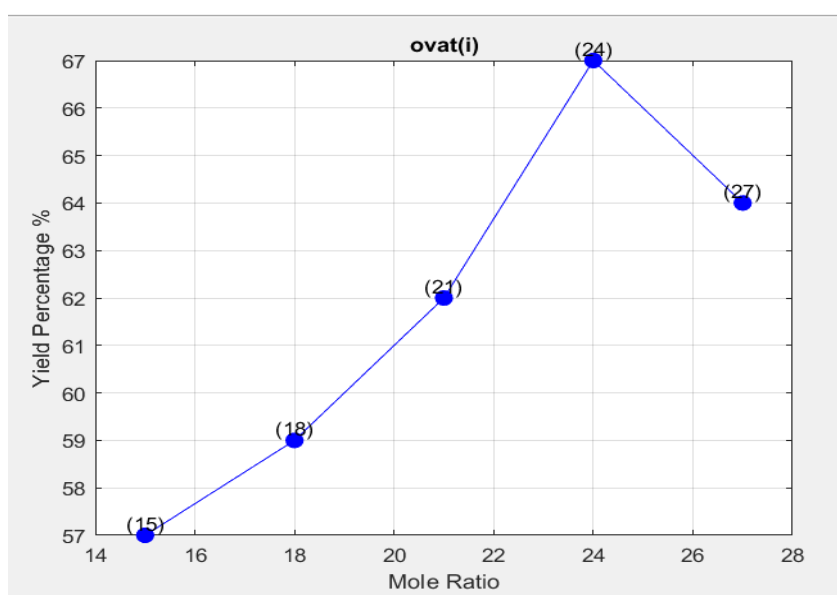


Fig 5.14 Graph plotted between molar ratio and yield percentage

The data point you've provided, with a yield of 67% at a molar ratio of 1:24, offers significant insights into biodiesel production.

Firstly, the 67% yield at the 1:24 molar ratio signifies the efficiency of this specific ratio in generating biodiesel. It suggests that under the given experimental conditions, the reaction at this ratio resulted in a relatively high yield compared to other ratios tested.

Moreover, this data point hints at the potential optimality of the 1:24 molar ratio for biodiesel production in the tested conditions. It may represent a balanced combination of reactant concentrations that promotes efficient conversion to biodiesel.

Comparing the yield at 1:24 with yields at neighbouring ratios can provide further understanding. If the yield at 1:24 stands out significantly from adjacent ratios, it emphasizes the importance of this ratio in influencing yield.

However, if the yield at 1:24 falls short of expectations, adjustments to the production process might be necessary. This could involve refining reaction conditions, optimizing catalyst concentration, or modifying feedstock properties to enhance yield.

Further analysis into why the yield at 1:24 is at its specific level may also be beneficial. Exploring factors such as reaction kinetics, catalyst efficiency, or feedstock characteristics could provide deeper insights and potential avenues for improving yield.

In summary, the yield of 67% at a molar ratio of 1:24 provides valuable information about the efficiency and optimization potential of biodiesel production under the tested conditions. Analyzing the given values for catalyst weight percentage (3%, 4%, 5%, 6%, 7%) and the corresponding yields (67%, 70%, 73%, 71%, 70%), we can plot a graph to visualize the relationship between these variables.

Upon plotting the data points and drawing a trendline, we can observe the following:

Trend Analysis: The trendline might indicate whether there's a linear, exponential, or other type of relationship between catalyst weight percentage and yield.

Optimal Catalyst Weight: We can identify the catalyst weight percentage at which the yield is maximized. This point signifies the most efficient catalyst concentration for achieving the highest yield in the given conditions.

Yield Variation: Any fluctuations or consistent changes in yield with changes in catalyst weight percentage can provide insights into the sensitivity of the reaction to catalyst concentration variations.

Consistency and Stability: If the yields remain relatively consistent or stable across different catalyst weight percentages, it suggests robustness in the reaction process under varying catalyst conditions.

Comparison with Previous Studies: The plotted data can be compared with previous studies or theoretical predictions to validate experimental results and understand how they align with established knowledge.

Table 5.5 catalyst weight% and Yield%

S.No	Catalyst weight%	Yield (%)
1	3	67
2	4	70
3	5	73
4	6	71
5	7	70

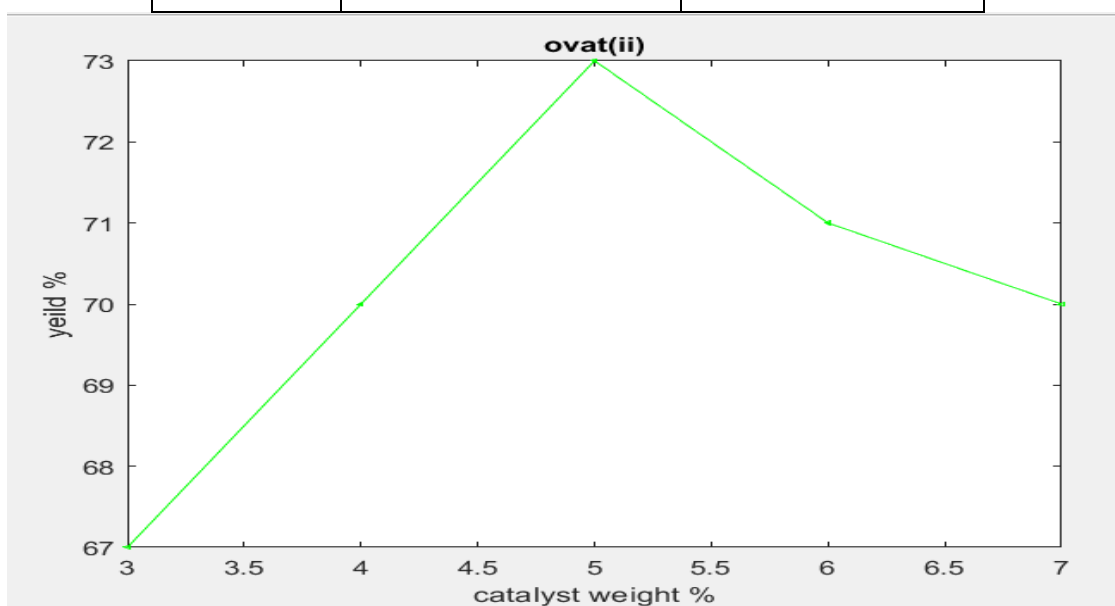


Fig 5.15 Graph plotted between catalyst weight % and yield %

The yield of 73% indicates a relatively efficient conversion of reactants into desired products. This high yield implies that the chosen catalyst concentration effectively facilitates the desired chemical transformation, resulting in a significant proportion of the reactants being converted into the desired products within the given reaction time.

Furthermore, the yield of 73% at 5% catalyst weight suggests that further increasing the catalyst concentration might not lead to a significant improvement in yield or could potentially result in diminishing returns. It's essential to consider factors such as catalyst deactivation or mass transfer limitations beyond this optimal concentration.

In summary, the data point corresponding to a 5% catalyst weight and a yield of 73% highlights the effectiveness of this catalyst concentration in promoting the desired reaction and achieving a high yield of the desired products.

Table 5.6 Reaction time and Yield%

Sl.NO	Reaction Time (min)	Yield (%)
1	120	73
2	150	78
3	180	81
4	210	77
5	240	75

Plotting this data on a graph with reaction time on the x-axis and yield on the y-axis would enable us to observe any trends or patterns.

Analyzing the relationship between reaction time and yield is pivotal in optimizing Ovat studies. The provided dataset includes reaction times ranging from 120 to 240 minutes, with corresponding yields spanning from 73% to 81%. Plotting these data points allows for visualizing trends and drawing conclusions regarding the most effective reaction duration.

Upon plotting the data and observing the resulting graph, several key insights can be drawn:

Firstly, there appears to be an overall increasing trend in yield as reaction time progresses from 120 to 180 minutes. This suggests that within this range, longer reaction times lead to higher yields, with 180 minutes indicating the peak yield of 81%. Beyond 180 minutes, however, there is a slight decline in yield, indicating that excessively prolonged reaction times may not be advantageous and could even be detrimental to the process.

Understanding the optimal reaction time is crucial for Ovat studies, as it directly impacts the efficiency and effectiveness of the reaction. In this case, the data suggest that around 180 minutes represents the optimal reaction time for maximizing yield. Further investigation beyond this timeframe would be necessary to assess the reaction's stability and to ensure that the observed trend holds true over longer durations.

Additionally, the consistency of yield across different reaction times is essential for process reliability. While fluctuations are minimal within the provided dataset, it's imperative to conduct additional experiments to validate the stability of the reaction process under varied conditions.

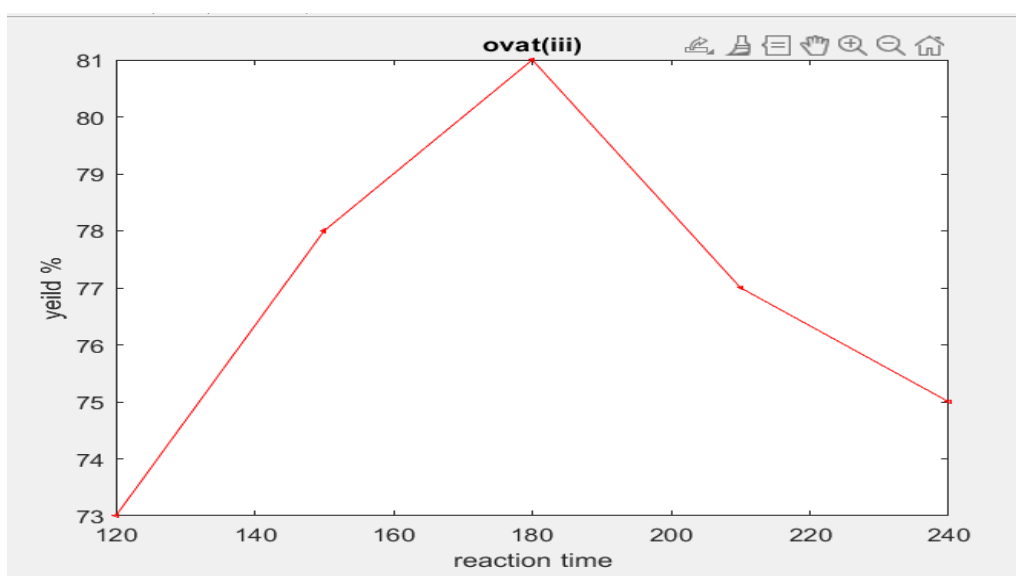


Fig 5.16 Graph plotted between Reaction time and yield %

In conclusion, based on the analysed data, a reaction time of approximately 180 minutes appears to yield optimal results for Ovat studies. However, comprehensive studies encompassing longer reaction times and assessing reaction stability are warranted to fully optimize the reaction conditions and ensure reproducibility and scalability in Ovat studies.

5.4 BIODIESEL CHARACTERIZATION

5.4.1 FT- IR:

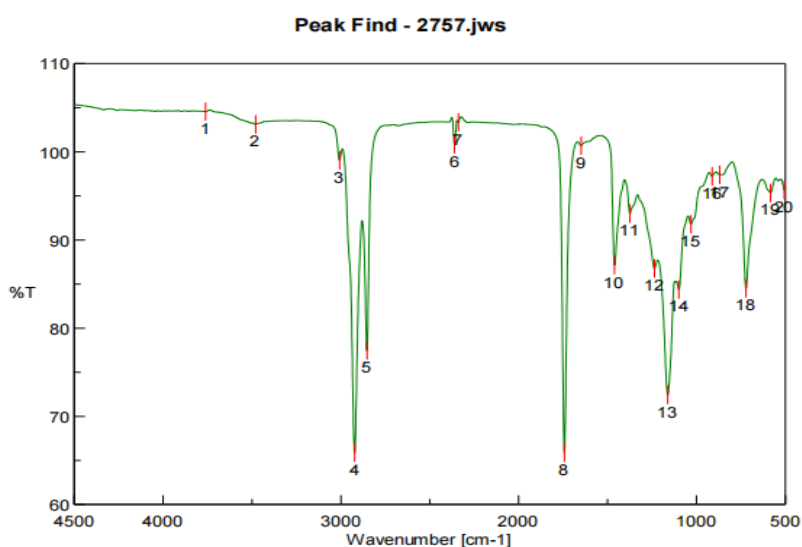


Fig 5.17 FT-IR for biodiesel

In the initial sample, the presence of glycerol is confirmed by a peak corresponding to its OH group at about 3007.44 kg/m². The identification of glycerol as a compound is further confirmed by the corresponding C-H peak at 2853.17 kg/m² in the bottom layer (FT-IR spectrum). The absence of an OH peak, in combination with the ester peak (1647 kg/m²) and C-H peak (2853.17-2853.44 kg/mol) in the bottom layer of biodiesel, is indicative of the presence of a chain fatty ester compound, which is characteristic of biodiesel's composition.

Biodiesel is primarily composed of single-cored carbon atoms (C-C), sp³ hybridized to form C-H bonds. C-H bond stretching is typically between 1374-1460 kg/m². C-O bonds in the ester group are observed in the range 1030.77 – 1374.03 cm⁻¹ and commonly appear as multiple peaks. C=C stretching is observed in the range

1647.88 – 1699.23 cm⁻¹, indicating unsaturated bonds. C-H stretching is seen in the wave number range 583.36 – 1099.43 cm⁻¹. FT-IR values confirm the biodiesel sample's composition and chemical structure. Ester groups derived from glycerol transesterification indicate successful photo-transesterification. The biodiesel sample contains saturated hydrocarbon chains and other characteristic functional groups.

5.4.2 GC-MS (Gas chromatography mass spectroscopy)

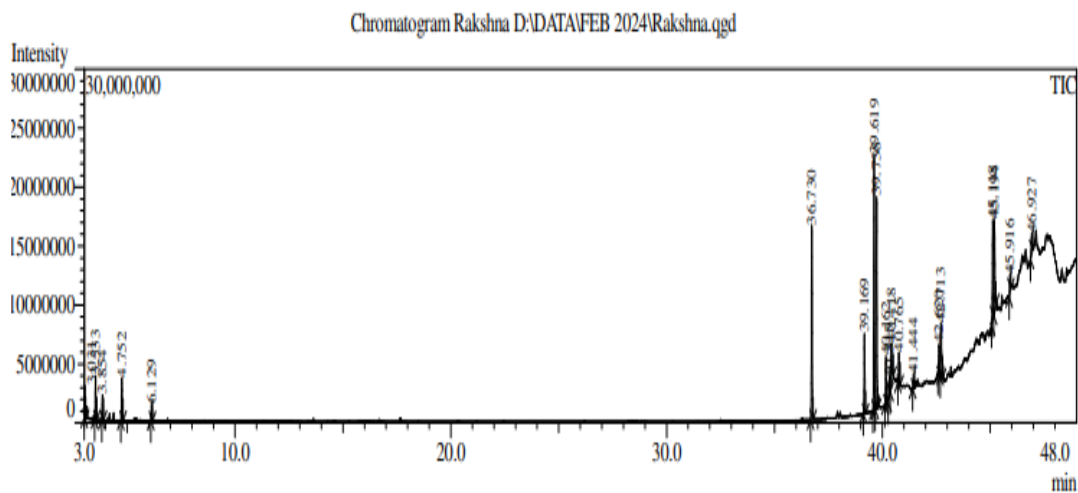


Fig 5.18 GCMS graph of produced biodiesel

The FAMES are key determinants of biodiesel composition. A detailed overview of the main FAMES, their retention times, and their peak areas is provided in the table

Table 5.7 Identification of Fatty Acid Methyl Esters (FAME)s

Component	Retention time(min)	Area	Nature of compound
Hexadecenoic acid methyl ester (Palmitic acid)	36.730	39625454	saturated
9,12-Octadecadienoic acid (linoleic acid)	39.619	51810207	unsaturated
9-Octadecenoic acid (oleic acid)	39.736	45576197	unsaturated
Methyl stearate (stearic acid)	40.162	9088498	saturated

In biodiesel production, FAME compounds are essential because they are the main components of biodiesel. When triglycerides undergo transesterification, they break down into glycerol and fatty acid methyl esters (FAMEs). These FAMEs are the actual biodiesel molecules. These FAMEs provide an alternative, renewable source of energy to traditional fossil fuels, such as diesel derived from petroleum. And can be used in existing diesel engines and infrastructure without modification, making it a convenient alternative fuel. Overall, these FAME compounds play a crucial role in biodiesel production, providing a sustainable and environmentally friendly alternative to conventional diesel fuel.

5.4.3 Properties of Biodiesel

Table 5.8 properties of biodiesel

S. No	Parameters	Results	Standard Biodiesel properies
1	Density	821.4 g/cc	860 - 900 g/cc
2	Viscosity	2.8896 mm ² /s	1.9 - 6.0 mm ² /s
3	Calorific value	2.21 MJ/kg	37.3 - 39.7 MJ/kg
4	Moisture Content	0.04%	≤ 0.05
5	Flash point by PMCC method (°C)	215 °C	> 130
6	Fire point by PMCC method (°C)	222 °C	> 170
7	Cloud Point °C	21 °C	≤ 0
8	Pour point °C	14 °C	≤ 0

Identification:

Moisture content is low, which is good for many applications. The viscosity is within the normal range of biodiesel, indicating good fluidity. Calorific value is unusually low for biodiesel. This may need to be further investigated or verified.

Density is Consistency with expected values of this parameter. The flash point (°C): indicates whether the vapor of the volatile material will ignite when it is mixed with air. By the obtained value we can conclude that the reaction mixture is stable and requires a large amount of heat to generate enough vapor to ignite. The fire point is

222°C, indicating that the mixture will become flammable at slightly higher temperatures than the flash point. cloud point (°C): indicates whether waxy crystals will form in the liquid. This is especially important for biodiesel production, as cloud points affect the low-temperature flow properties of the biofuel. pour point is the lowest point at which the liquid will flow or pour under given conditions. At 14°C, the biodiesel mixture will remain relatively fluid, which is ideal for practical use.

In summary Its low moisture content suggests minimal water presence, crucial for fuel stability. Furthermore, its viscosity, ensuring good fluidity and efficient engine performance. However, its low calorific value affects both energy content and engine efficiency. On the other hand, its density of 821.4 g/cc aligns with expectations. The flash point of 215°C and fire point of 222°C indicate stability during storage, requiring significant heat for ignition and minimizing fire hazards. Nevertheless, the cloud point at 21°C may impact low-temperature flow properties, while the pour point at 14°C suggests continued fluidity, improving practical use. In summary, while the biodiesel displays positive attributes, addressing the low calorific value and understanding the implications of the cloud point could optimize its performance across various applications and environments.

5.5 IC ENGINE TEST

5.5.1 For 0% (Pure Diesel Engine Performance Analysis)

Table 5.9 Biodiesel Characterization (0%)

ID	2.56	5.67	6.3	9.33
BP	0	1	1.8	2.53
2MEH	6.8	17	28.1	26.9
2THE	147.1	97.7	83.4	84
2BTH	10	16.6	23.4	27
SFC	0.85	0.52	0.37	0.32

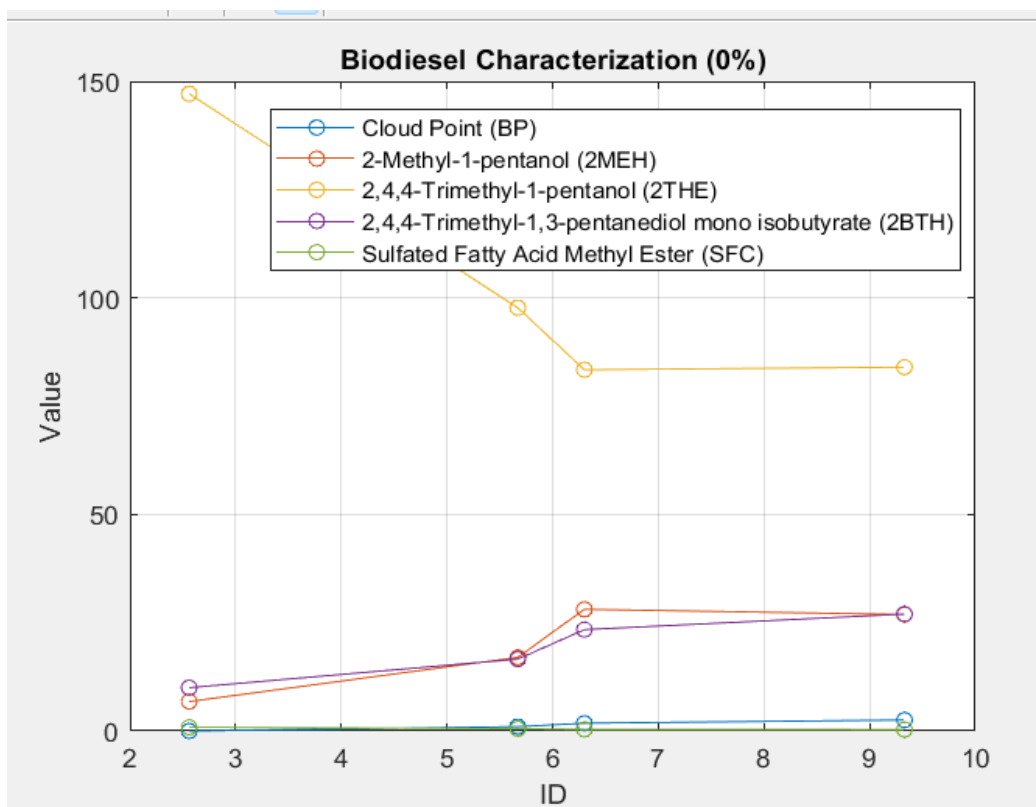


Fig 5.19 ID vs value graph for 0%

The provided data pertains to the characterization of a base fuel consisting of 0% biodiesel. Each parameter measured serves to delineate the properties and composition of this base fuel. The sample identifiers (ID) uniquely denote each analysed sample, aiding in tracking and referencing. Cloud Point (BP) values denote the temperature at which solid crystals begin to form within the fuel, indicating its low-temperature operability. Lower cloud point values signify superior low-temperature performance, as the fuel remains liquid at lower temperatures. Parameters such as 2-Methyl-1-pentanol (2MEH), 2,4,4-Trimethyl-1-pentanol (2THE), and 2,4,4-Trimethyl-1,3-pentanediol mono isobutyrate (2BTH) likely represent concentrations of specific compounds present in the base fuel, reflecting characteristics stemming from the fuel's production process or inherent properties. Additionally, Sulfated Fatty Acid Methyl Ester (SFC) values serve as indicators of the base fuel's quality, particularly in terms of acidity. Lower SFC values typically correlate with higher fuel quality, as heightened acidity levels can induce engine corrosion and related issues. The graph visually depicts these parameters against

the sample identifier, facilitating comparative analysis and identification of trends or anomalies within the base fuel's characterization data.

Table 5.10 Biodiesel Characterization (0%)

C	LOAD			
	0	3	6	9
CO	0.20	0.16	0.11	0.11
HC	47	35	19	20
CO ₂	1.60	2.00	2.10	3.70
O ₂	18.33	17.88	17.67	15.52
NOX	13	39	155	425
SOME DENSITY	4.1	9.8	19.1	27.8

The graph illustrates the relationship between engine load and various emission parameters, as well as exhaust gas density. Each line on the graph corresponds to a specific parameter, namely Hydrocarbons (HC), Carbon Monoxide (CO), Carbon Dioxide (CO₂), Oxygen (O₂), Nitrogen Oxides (NO_x), and exhaust gas density. By plotting these parameters against engine load, the graph offers valuable insights into how emissions and exhaust gas density fluctuate with changes in engine operation. For instance, variations in HC and CO levels may indicate incomplete combustion under certain load conditions, highlighting potential areas for combustion optimization. Similarly, changes in NO_x emissions can be observed, providing crucial information for emission control strategies. Moreover, tracking exhaust gas density alongside emission parameters offers additional context. Ultimately, this comprehensive visualization aids in understanding the intricate relationship between engine load, emissions, and combustion efficiency, facilitating the development of more environmentally friendly and efficient engine systems.

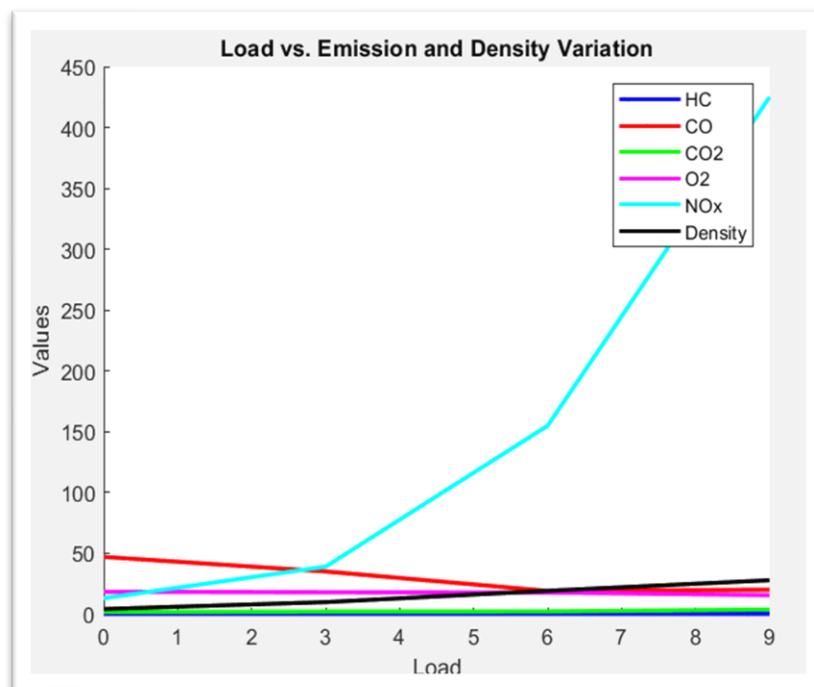


Fig 5.20 load vs Emission and Density Variation

5.5.2 The various parameters comparison between 20% and 40% load

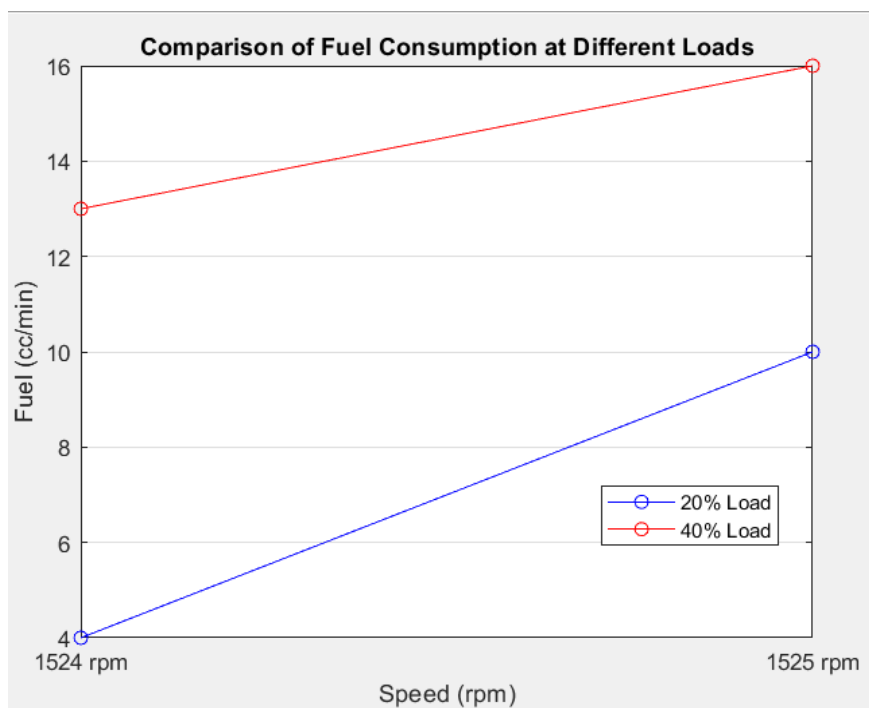


Fig 5.21 Comparison of Fuel consumption at Different Loads

The graph compares fuel consumption at two engine loads: 20% and 40%.

At both loads, as engine speed increases (moving right on the x-axis), fuel consumption generally increases.

This illustrates that higher engine speeds typically require more fuel to maintain power output, especially at higher loads.

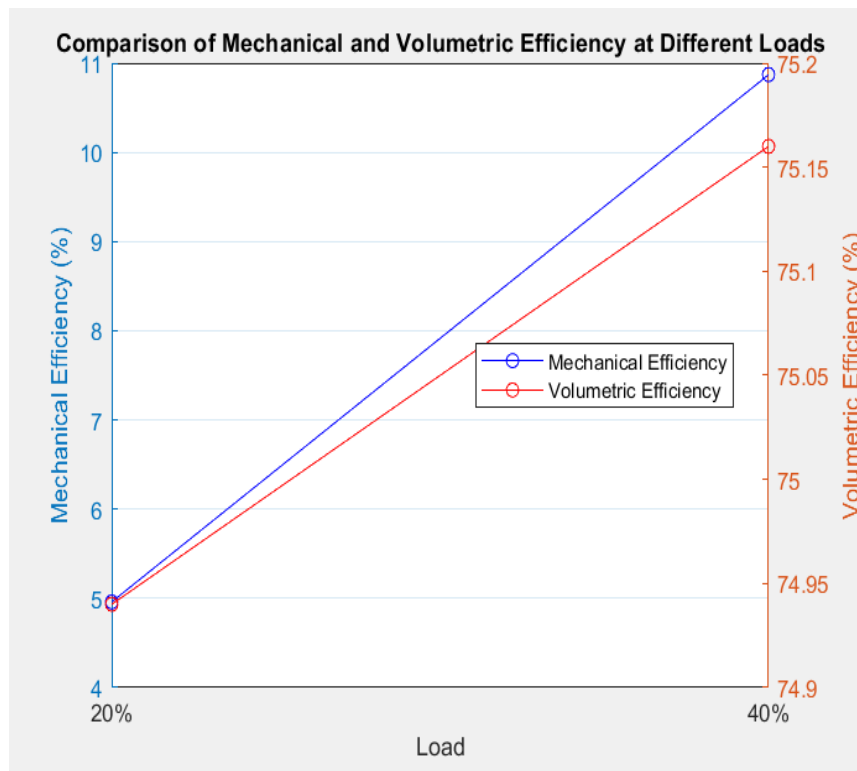


Fig 5.22 comparison of mechanical and volumetric efficiency at Different loads

This graph illustrates how mechanical and volumetric efficiency vary with engine load, specifically comparing 20% and 40% loads. Mechanical efficiency, representing the engine's ability to convert fuel energy into mechanical work, generally improves with higher loads due to optimized combustion. Volumetric efficiency, indicating the engine's air intake capability, also tends to increase at higher loads, reflecting improved breathing efficiency. Analyzing these efficiency metrics helps in understanding engine performance under different workloads, guiding optimization efforts for enhanced fuel economy and power output across various operating conditions.

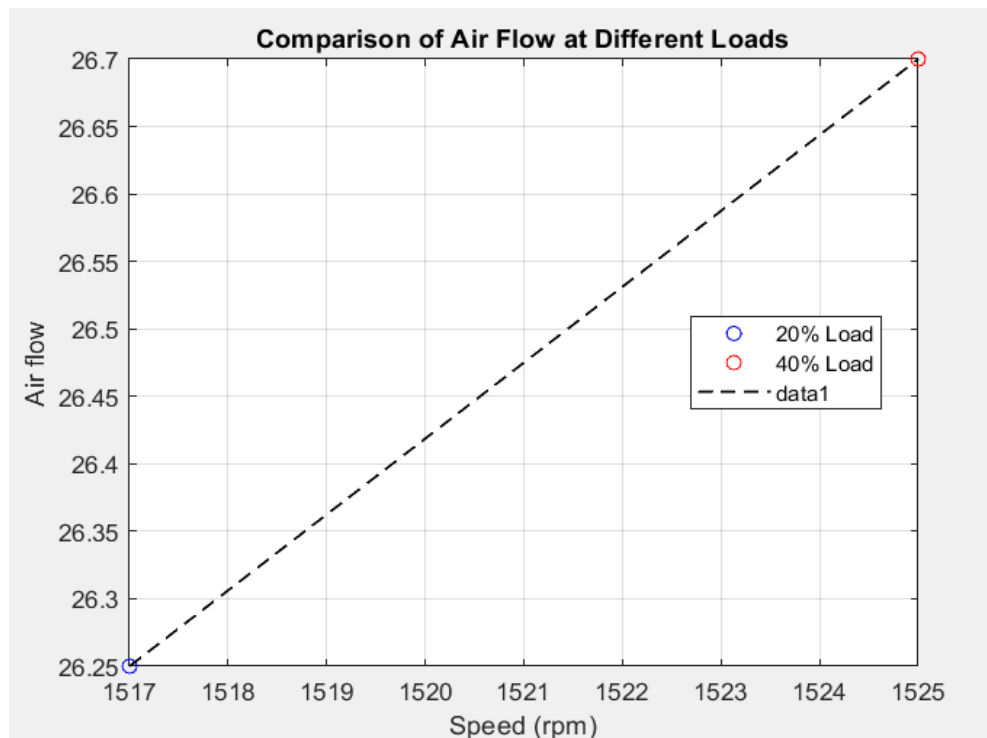


Fig 5.23 comparison of Air Flow at Different loads

This graph visually compares the air flow rates of an engine at 20% and 40% loads, providing valuable insights into its performance characteristics. Analyzing air flow at different loads is crucial for understanding how efficiently the engine draws in air for combustion under varying workloads. This comparison helps engineers identify potential inefficiencies or constraints in the engine's air intake system and guides optimization efforts to enhance overall performance. By visualizing the relationship between air flow and load, engineers can make informed decisions about engine tuning strategies to achieve desired performance outcomes, such as improved power delivery or fuel efficiency. Overall, this graph serves as a practical tool for evaluating engine performance and guiding optimization efforts to enhance efficiency and performance.

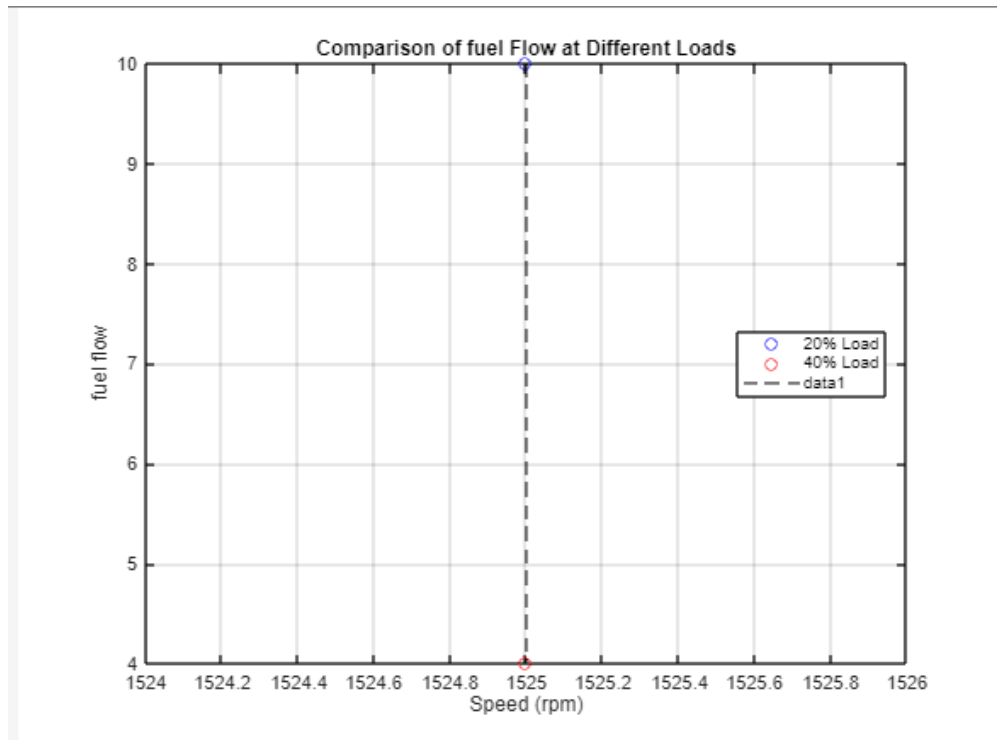


Fig 5.24 comparison of fuel Flow at Different loads

In internal combustion (IC) engine testing, the "load vs fuel flow" graph is a fundamental tool for understanding and optimizing engine performance. It's particularly crucial in dynamometer testing, where the engine is subjected to controlled loads to simulate real-world conditions without the need to operate the vehicle. Load in engine testing refers to the amount of resistance or work imposed on the engine by the dynamometer. This can be controlled by adjusting the dynamometer settings. Fuel flow indicates the rate at which fuel is being injected into the engine's cylinders. Fuel flow needs to be precisely controlled to maintain the desired air-fuel ratio for optimal combustion and engine performance.

In the context of an IC engine test, the "load vs fuel flow" graph visually represents how these parameters vary with changes in engine load. According to the graph load on the horizontal axis and both fuel flow on the vertical axes. As the load on the engine increases, typically both fuel flow rates also increase. This is because the engine requires more air and fuel to produce the additional power demanded by the load. The relationship between load and fuel flow rates may not be linear and can vary depending on factors such as engine design, operating conditions, and fuel type.

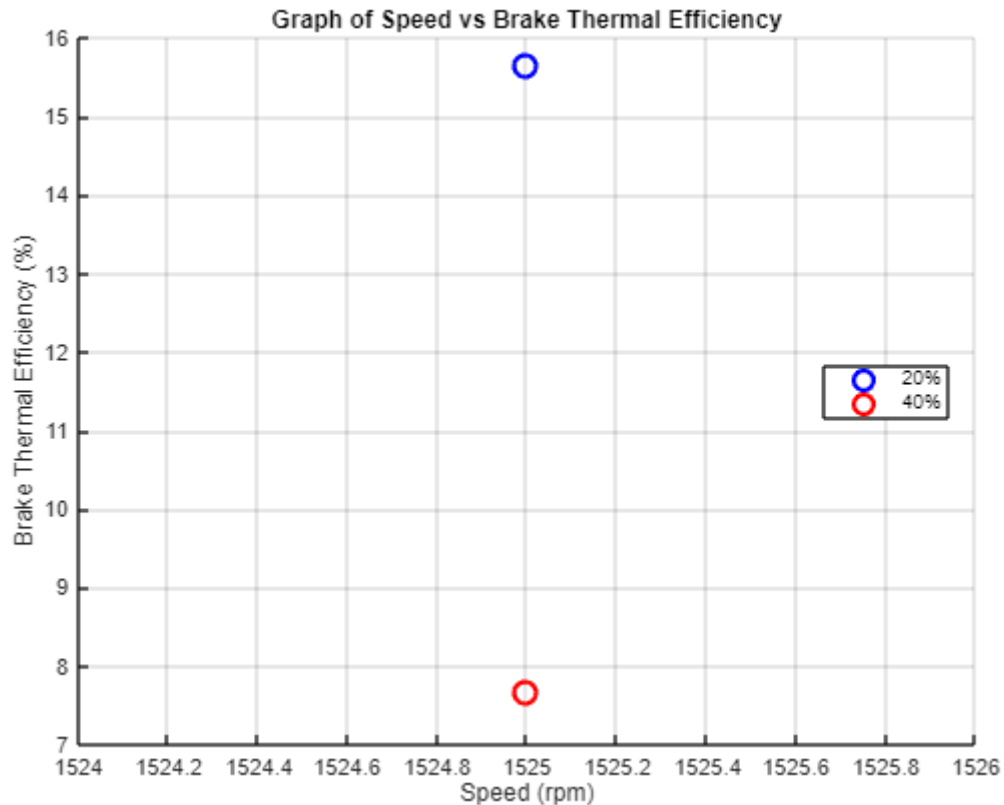


Fig 5.25 Graph of speed vs brake thermal efficiency

Speed vs. brake thermal efficiency refers to the relationship between the rotational speed of an engine and its brake thermal efficiency. Speed refers to the rotational speed of the engine's crankshaft, typically measured in revolutions per minute (rpm). It indicates how fast the engine is running. Brake thermal efficiency is a measure of how effectively an engine converts the chemical energy of fuel into mechanical energy at the output shaft. It is expressed as a percentage and represents the ratio of the useful work output to the total energy input from the fuel. Higher brake thermal efficiency indicates better fuel utilization and overall engine performance.

Typically, from the graph the relationship between speed and brake thermal efficiency may not be linear, and there may be an optimal speed range where the engine operates most efficiently.

20%

40%

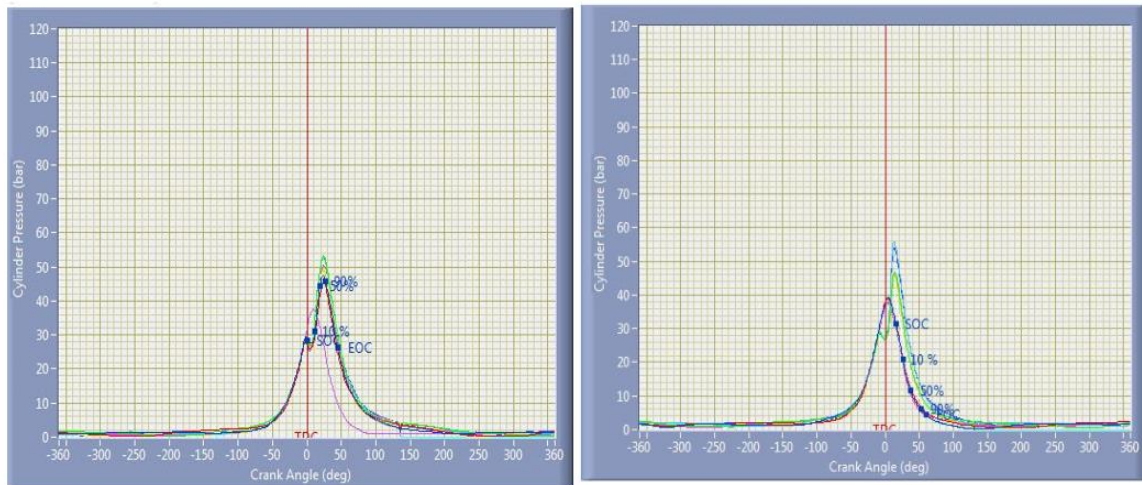


Fig 5.26 edx

The graph illustrates how the pressure within the combustion chamber (cylinder) changes over the engine's cycle, typically against the crankshaft angle or piston position for 20% and 40%. The graph depicts the four strokes of the engine cycle: intake, compression, power, and exhaust. Each stroke is associated with characteristic pressure changes within the cylinder. During the intake stroke, the pressure within the cylinder decreases as the piston moves downward, creating a vacuum that allows the air-fuel mixture to enter the cylinder. In the compression stroke, the pressure within the cylinder rises sharply as the piston compresses the air-fuel mixture. This pressure increase prepares the mixture for efficient combustion. The power stroke is characterized by a rapid increase in pressure as the ignited air-fuel mixture expands, pushing the piston downward and producing mechanical work. The pressure reaches its peak during this stroke

CHAPER 6

SUMMARY AND CONCLUSION

SUMMARY

The study investigates the feasibility of producing biodiesel from non-edible CPSO using a TiO_2/ZnO catalytic converter in a photocatalyst reactor. CPSO's promising physio chemical properties, rich in 9(12)-octadecadienoic and n(hexadecenoic) acids, make it suitable for biodiesel production. Extensive characterization of the TiO_2/ZnO nano-composite was conducted, revealing its composition, structure, and properties. Successful glycerol transesterification resulted in biodiesel suitable for various applications. The study demonstrates the viability of using TiO_2/ZnO catalysts for sustainable biodiesel production through photo-transesterification.

Additionally, the study includes comprehensive analyses of various parameters related to internal combustion (IC) engine performance under different loads. Visual representations, including load vs. emission and density variation graphs, offer valuable insights into combustion efficiency and emission parameters. Comparative analyses at different engine loads elucidate fuel consumption, mechanical and volumetric efficiency, air and fuel flow rates, and speed vs. brake thermal efficiency, contributing to understanding engine performance characteristics.

CONCLUSION

In conclusion, the study confirms the feasibility of producing biodiesel from CPSO using TiO_2/ZnO catalysts via photo-transesterification, while also providing insights into IC engine performance under varying loads. Characterization techniques offer detailed insights into the composition and chemical structure of biodiesel, affirming its suitability as a fuel source. Further research endeavours may focus on optimizing engine conditions, enhancing fuel efficiency, and exploring advanced combustion technologies to improve internal combustion engine sustainability and performance.

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ABSTRACT

Biodiesel, derived from renewable feedstocks such as silk cotton seed oil, presents an environmentally friendly and domestically produced alternative fuel with numerous benefits, including reduced greenhouse gas emissions, enhanced energy security, and support for sustainable agriculture. Traditional biodiesel production methods often involve chemical catalysts and heat in transesterification reactions. However, photo-transesterification offers an innovative approach utilizing light energy, particularly ultraviolet (UV) or visible light, to drive the reaction. Techniques such as FTIR (Fourier Transform Infrared Spectroscopy), GC-MS (Gas Chromatography-Mass Spectrometry), and IC engine studies are conducted. FTIR helps identify functional groups in biodiesel, GC-MS analyses its chemical composition, while IC engine studies evaluate its combustion properties and engine performance. These characterization studies play a crucial role in assessing biodiesel's suitability as a sustainable alternative fuel. The identification of compounds in biodiesel production is crucial for understanding its composition and yield. In our study, we confirmed the presence of glycerol through the detection of characteristic peaks in the FT-IR spectrum. Specifically, the OH group of glycerol was identified at approximately 3007.44 kg/m^2 , while the corresponding C-H peak appeared at 2853.17 kg/m^2 in the bottom layer. Moreover, the absence of an OH peak alongside the presence of ester (1647 kg/m^2) and C-H (2853.17-2853.44 kg/mol) peaks in the bottom layer suggests the prevalence of chain fatty ester compounds, which are indicative of biodiesel composition. To optimize the yield percentage, experiment with variations in molar ratio, catalyst weight percent, and reaction time. Currently, the highest yield of 81% was achieved at a molar ratio of 1:24, catalyst weight percent of 5%, and reaction time of 180 minutes. Adjusting these parameters slightly, along with optimizing temperature, and minimizing byproducts, could enhance yield further.

Keywords: photo transesterification, catalyst, impregnation, sustainable.

CHAPTER 1

INTRODUCTION

1.1 BIODIESEL

Biodiesel is a renewable and environmentally friendly alternative to traditional fossil fuels, especially petroleum-based diesel. This is a biofuel made from organic materials, usually vegetable oil, animal fat, or recycled cooking oil. The production of biodiesel is a process called transesterification, where this raw material is chemically processed to break down the triglyceride structure into biodiesel and glycerol. One of the major advantages of biofuel over conventional diesel fuel is its ability to reduce greenhouse gas emissions. Because biodiesel comes from a renewable resource, the carbon dioxide released during combustion is part of the natural carbon cycle. Instead, burning fossil fuels releases carbon dioxide that has been stored for millions of years, thereby exacerbating climate change. Biodiesel can be used in diesel engines without modification because it is convenient and suitable for transport infrastructure. There are also properties that improve fuel lubrication, thus extending the life of diesel engines.

The widespread use of biodiesel is driven by a variety of factors, including concerns about energy security, the desire to reduce dependence on fossil fuels, and the need to address environmental issues. Governments and corporations around the world are looking for ways to integrate biomass into their energy production in order to create a more sustainable and energy-efficient future. Although the impact is positive, challenges such as the supply of raw materials, production costs, and competition for raw materials for food production must still be overcome. Biodiesel production is an economical primary fuel source. Research and development continue to improve the efficiency and sustainability of biodiesel production, making it a key driver in the transition to clean and sustainable energy solutions. Biodiesel production is an environmentally friendly and sustainable process that involves converting renewable raw materials, such as vegetable oil, into environmentally friendly and sustainable products. Recycled animal fat, or cooking oil, is an alternative to conventional diesel fuel.

The most important way to improve environmental sustainability in biodiesel production is to diversify livestock feed and focus on non-food sources such as waste oil and algae. To reduce carbon emissions and encourage the switch to green fuels, governments continue to implement regulations and incentives to encourage the biofuels industry. The goal of technical research and development is to ensure the full life of the organism while researching new raw materials and increasing the efficiency of production. In addition, due to developments in engine technology, it is possible to blend other biodiesel with conventional diesel fuel. Eco-design technologies have revolutionized the landscape of bio-based manufacturing, incorporating sustainable and eco-friendly practices throughout the process. The key is the selection of raw materials, using non-food sources such as algae, jatropha, or cooking oil to reduce the pressure on food resources.

Biodiesel also provides energy security by diversifying fuel sources and relying on domestic raw materials. Since biodiesel is compatible with existing diesel engines and infrastructure, this will allow for better transferability and wider adoption. In addition, the lubricating properties of biodiesel extend the life of the engine, thus reducing maintenance costs.

The conversion process is key to biodiesel synthesis and requires the use of environmentally friendly catalysts such as enzymes or solid catalysts, thus making cleaning more efficient than traditional chemical machinery. Innovations such as supercritical water technology improve environmental efficiency by operating at low temperatures and pressures, saving energy, and reducing the overall ecological footprint. Biomass synthesis has become an environmentally friendly activity, optimizing the use of various raw materials and reducing waste, thereby encouraging biological sustainability. The focus on waste utilization, including the conversion of waste cooking oil and animal fats into biomass, demonstrates commitment to circular economy principles.

Energy-efficient processes, often incorporating renewable energy sources, help reduce the carbon footprint of biodiesel production. In addition, water recycling and biological plant life conservation practices. Life cycle analysis is a comprehensive tool that evaluates the entire biodiesel production process, from raw material cultivation to end-use disposal. By incorporating these environmentally friendly technologies, the

biodiesel industry is paving the way to an environmentally responsible and sustainable future in the alternative fuel sector. Alternative catalysts in biodiesel production represent a major leap towards more sustainable and efficient processes.

Biodiesel continues to be a significant player in the renewable fuels market, although its exact status may vary depending on factors such as government policies, technological advancements, and market demand. Biodiesel production and consumption have been steadily increasing over the years due to various factors such as concerns about climate change, energy security, and the desire to reduce reliance on fossil fuels. Many countries have implemented policies and incentives to promote the use of biodiesel as part of their efforts to reduce greenhouse gas emissions and meet renewable energy targets. These policies include blending mandates, tax incentives, subsidies, and renewable fuel standards.

Ongoing research and development efforts are focused on improving biodiesel production processes, increasing feedstock diversity, enhancing fuel properties, and reducing production costs. These advancements contribute to the continued growth and viability of biodiesel as a renewable fuel option. Despite its benefits, biodiesel faces several challenges, including competition with other biofuels such as ethanol, concerns about land use change and deforestation associated with certain feedstocks, and fluctuations in feedstock prices. The global biodiesel market is influenced by trends in energy markets, environmental regulations, agricultural policies, and trade dynamics. Emerging economies, in particular, are expected to play a significant role in driving future growth in biodiesel production and consumption.

1.2 RESEARCH OBJECTIVE

The objective of our project is to produce biodiesel by photocatalytic method using ZnO and TiO₂ catalyst. The chosen of these two catalysts is due to their photocatalytic performances. The use of photocatalytic transesterification method is due to their several potential advantages over traditional transesterification methods. This method utilizes light-activated catalysts to facilitate the transesterification reaction. Photocatalytic reactions can exhibit higher selectivity towards the desired product, leading to fewer undesirable by-products or impurities in the biodiesel. This can result in higher yields and improved product quality and also occurs under milder reaction conditions which leads to reduced energy consumption and lower operating costs.

However, it's essential to note that photocatalytic transesterification is still an emerging technology and may face challenges such as the development of efficient and stable photocatalysts, optimization of reaction conditions, and scalability for industrial-scale production. Additionally, the economic viability of photocatalytic transesterification compared to traditional methods will depend on factors such as catalyst cost, energy requirements, and process efficiency. Further research and development efforts are needed to fully realize the potential benefits of this approach for biodiesel production.

The use of silk cotton seed oil is due to their Abundant Availability .it is cultivated worldwide, and silk cotton seed oil is readily available as a byproduct of cotton production. This abundant supply makes cottonseed oil a potentially sustainable feedstock for biodiesel production. It typically has a high oil content, making it an efficient feedstock for biodiesel production. This means that a significant amount of biodiesel can be produced from a given quantity of cottonseed oil. By utilizing cottonseed oil for biodiesel production, waste from the cotton industry can be minimized, contributing to waste reduction and resource efficiency.

1.3 PROBLEM STATEMENT

Biodiesel production, a promising alternative to fossil fuels, faces a critical hurdle: maintaining optimal reaction temperatures during transesterification. Deviations from this ideal range can significantly impact the process, leading to lower biodiesel yield, unwanted byproducts, and increased energy consumption. This inefficiency necessitates the development of improved temperature control strategies. By achieving precise temperature control, we can unlock the full potential of biodiesel production, maximizing yield, minimizing waste, and optimizing energy usage, ultimately paving the way for a more sustainable and efficient biofuel industry

The problem statement for biodiesel production involves several key aspects, including the reversible nature of the transesterification reaction, challenges associated with commercial-scale batch processing, and the utilization of photocatalytic reactors with ZnO and TiO₂ catalysts. Additionally, issues related to glycerol formation present further complications.

Biodiesel production through transesterification involves a reversible reaction, which complicates the achievement of high yields and purity levels. This reversibility

necessitates the careful optimization of reaction conditions to Favor the production of biodiesel over the reactants, thus maximizing efficiency.

Commercial-scale biodiesel production often relies on batch processing due to its simplicity and flexibility. However, this approach poses challenges such as limited scalability, longer processing times, and difficulties in maintaining consistent reaction conditions, leading to variations in product quality and yield.

The utilization of photocatalytic reactors presents a promising alternative for biodiesel synthesis, with ZnO and TiO₂ emerging as catalysts due to their photocatalytic properties. However, challenges remain in optimizing reactor design, catalyst loading, and reaction parameters to achieve high conversion rates and selectivity while minimizing energy consumption and catalyst degradation.

Furthermore, glycerol formation during transesterification poses a significant problem, as it affects product purity and complicates downstream processing. Strategies to mitigate glycerol formation, such as catalyst modification or process optimization, are essential to enhance the overall efficiency and economic viability of biodiesel production processes.

The analysis of biodiesel production experiments reveals significant challenges in achieving optimal yield and maintaining temperature control. Despite variations in parameters such as molar ratio, catalyst weight percentage, and reaction time, the highest yield was not consistently attained across experimental conditions. For instance, while the yield peaked at 67% with a molar ratio of 1:24 and a catalyst weight percentage of 5%, it fluctuated in other scenarios, indicating a lack of consistency in achieving maximum yield. Additionally, the experiments did not explicitly address temperature control, which is crucial for maintaining reaction stability and maximizing yield. Without precise control over temperature, variations in reaction conditions may occur, potentially impacting yield and product quality. Therefore, the results underscore the challenges associated with optimizing yield and controlling temperature effectively in biodiesel production, highlighting the need for further research and refinement in these areas.

1.4 PHOTOCATALYTIC TRANSESTERIFICATION:

Photocatalysis represents an innovative biotechnological approach that integrates photocatalytic principles into traditional conversion methods. This method uses light energy to initiate the conversion reaction using a catalyst, usually an inorganic material such as titanium dioxide (TiO_2). Solar power absorbs photons from a light source (usually ultraviolet or visible light) and produces electron-electron pairs that participate in redox reactions. When producing biodiesel, this process controls the conversion of triglycerides (found in vegetable oils and animal fats) into biodiesel and glycerine. Photocatalytic distillation has many advantages, including simple reaction conditions, reduced energy consumption, and the potential of solar energy as a sustainable light source. This approach is expected to improve the efficiency of biodiesel synthesis and reduce the environmental impact associated with traditional conversion methods. Ongoing research in this area includes optimization of photocatalytic materials, reaction parameters, and scale-up to pave the way to greener and more sustainable biomass production.

Esterification plays a crucial role in the production of esters, organic compounds that find extensive applications across multiple industries. Catalysis accelerates the reaction between carboxylic acid and alcohol, producing ester and water as by-products. The general formula for the neutralization reaction is carboxylic acid + alcohol catalyst. A catalyst (usually sulfuric or hydrochloric acid) helps remove water molecules from the reactants to form the ester. Esters have a sweet odour and are easy to detect, making them useful in hunting and the arms control industry. Importantly, the esterification reaction is reversible.

Under certain conditions, the ester can be hydrolysed to the carboxylic acid and the parent alcohol. Transfection is the basic chemical process used to produce biodiesel, converting triglycerides in fats and oils into biodiesel (methyl fatty acids or ethyl esters) and glycerol. In the presence of a catalyst, the glycerol moiety of triglycerides is replaced by alcohol (mainly methanol and ethanol). Biodiesel and glycerine are the general formulas for transesterification. Enzymes (usually sodium or potassium hydroxide) aid in digestion by breaking down the complex structure of triglycerides into biomolecules and glycerol. This technology is important in the biodiesel industry because it produces high-quality biodiesel that can be used as a renewable and clean

alternative to conventional diesel fuel. Biodiesel is popular because it is compatible with a variety of raw materials, such as vegetable oils, animal fats, and recycled cooking oils, contributing to the efficiency and sustainability of biodiesel production. The by-product glycerine can be used for other purposes and adds value to the entire biodiesel production process. As biodiesel becomes increasingly popular as an environmentally friendly fuel source, research is underway to improve the efficiency and sustainability of the conversion process.

1.5 SILK COTTONSEED OIL (*ceiba pentandra* silk oil):

In biodiesel production, silk cotton seed oil extracted from *Ceiba pentandra* seeds offers several advantages. Firstly, it is a non-edible feedstock, thus avoiding competition with food crops and addressing concerns regarding food security. Additionally, silk cotton seed oil possesses favourable characteristics such as high lipid content and low free fatty acid levels, making it suitable for biodiesel production. Furthermore, *Ceiba pentandra* trees are fast-growing and resilient to harsh environmental conditions, contributing to sustainable feedstock cultivation. The use of silk cotton seed oil in biodiesel production promotes environmental sustainability by reducing greenhouse gas emissions and dependence on fossil fuels. However, challenges may arise in large-scale production, such as variability in seed oil composition and limited availability of *Ceiba pentandra* seeds in certain regions, necessitating further research and development to optimize its utilization in biodiesel production.

Silk cotton seed oil extracted from *Ceiba pentandra* seeds presents several advantages for biodiesel production. Firstly, *Ceiba pentandra* trees are widely distributed in tropical and subtropical regions, ensuring a sustainable and abundant source of feedstock. Additionally, silk cotton seeds contain a high oil content, typically ranging from 18% to 30%, making them valuable for oil extraction purposes. Importantly, as non-edible seeds, they alleviate concerns about competing with food crops for fuel production, promoting sustainable agricultural practices. Moreover, the low levels of free fatty acids in silk cotton seed oil reduce the need for extensive pre-processing steps, streamlining biodiesel conversion processes. Environmentally, biodiesel derived from silk cotton seed oil offers benefits such as reduced greenhouse gas emissions and decreased air pollution compared to conventional diesel fuel.

Furthermore, economically, its utilization can create opportunities for farmers, particularly in regions where *Ceiba pentandra* trees are cultivated, thus contributing to rural development and income generation.

However, several challenges accompany the use of silk cotton seed oil in biodiesel production. Firstly, the variability in oil composition due to factors like geographic location and tree genetics can impact biodiesel yield and quality, necessitating careful monitoring and optimization of production processes. Additionally, the availability of *Ceiba pentandra* seeds may be seasonal, and harvesting them can be labour-intensive, posing logistical challenges for consistent feedstock supply. Furthermore, despite its favourable characteristics, silk cotton seed oil may require specialized processing techniques to overcome challenges such as high viscosity and impurities. Competition with other uses, such as traditional medicine and fibre production, also exists, potentially affecting the availability of seeds for biodiesel production. Addressing these challenges will require further research and development to optimize production processes, improve extraction efficiency, and ensure sustainable practices throughout the supply chain.

1.6 CATALYST:

In the process of photocatalytic transesterification for biodiesel manufacture, zinc oxide (ZnO) and titanium dioxide (TiO₂) assume vital roles as catalysts, leveraging their unique attributes under light exposure. ZnO and TiO₂ serve as photocatalysts, initiating chemical reactions when exposed to light energy. Upon illumination with ultraviolet (UV) or visible light, both catalysts generate electron-hole pairs, thereby creating reactive species like superoxide radicals (O₂⁻) and hydroxyl radicals (-OH). These reactive species play a crucial role in catalysing the transesterification reaction by interacting with triglyceride molecules present in vegetable oils or animal fats. Through this process, triglycerides are converted into biodiesel and glycerol. ZnO and TiO₂ offer distinct advantages, including high surface area, stability, and cost-effectiveness, making them well-suited for catalysing the transesterification reaction under photocatalytic conditions. Additionally, their ability to generate reactive oxygen species under light illumination enhances the efficiency of the biodiesel production process. Thus, ZnO and TiO₂ play integral roles as photocatalysts in facilitating the

conversion of triglycerides into biodiesel, contributing to the advancement of sustainable and environmentally friendly fuel production technologies.

1.7 CHARACTERIZATION

Various characterization techniques are employed in biodiesel production and catalyst analysis to understand their properties and optimize production processes. For catalyst analysis, techniques such as Field Emission Scanning Electron Microscopy (FESEM) are utilized to examine catalyst surface morphology, providing insights into particle size, shape, and distribution, crucial for catalyst stability and activity assessment. Energy-Dispersive X-ray Spectroscopy (EDX) is employed to determine the elemental composition of catalyst materials, identifying active species and impurities essential for understanding catalyst performance. Additionally, Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) analyses assess catalyst surface area, pore size distribution, and volume, optimizing catalyst design for enhanced catalytic efficiency in biodiesel production.

OVAT (One Variable at a Time) experimentation is crucial in biodiesel production research. It involves adjusting one variable while keeping others constant to precisely assess its impact on yield, quality, and efficiency. By focusing on individual factors, researchers gain insights into process dynamics, simplify experimental design, and achieve cost and time efficiency. Overall, OVAT facilitates systematic exploration and optimization of biodiesel production processes.

On the other hand, for biodiesel analysis, Fourier Transform Infrared Spectroscopy (FTIR) is employed to analyse functional groups in biodiesel, confirming successful transesterification and providing insights into purity and composition. Gas Chromatography-Mass Spectrometry (GC-MS) determines the fatty acid methyl ester (FAME) profile of biodiesel, aiding in feedstock identification and assessing quality and properties crucial for engine performance. Additionally, Internal Combustion (IC) engine testing evaluates biodiesel performance under real-world conditions, assessing parameters like power output, fuel efficiency, and emissions characteristics. These combined characterization techniques play a vital role in understanding biodiesel properties, catalyst behaviour, and optimizing production processes for efficient and sustainable biodiesel production.

CHAPTER 2

LITERATURE SURVEY

Michael Rahul Soosai et al. (2022) have published a journal titled "Integrated global optimization and process modelling for biodiesel production from non-edible silk-cotton seed oil by microwave-assisted transesterification with heterogeneous calcium oxide catalyst." The creation of biodiesel, an environmentally benign and sustainable alternative to conventional diesel fuel, is the main topic of this study. Transesterification, a chemical procedure used to make biodiesel, involves reacting triglycerides (fats and oils) with an alcohol and a catalyst to create biodiesel and glycerine. In this investigation, microwave help is used throughout the transesterification process. Chemical processes can be accelerated by microwave heating, potentially increasing the yield of biodiesel. A heterogeneous calcium oxide catalyst is used to catalyse the transesterification reaction. Solid heterogeneous catalysts may be easily removed from the result and are appropriate for reuse because they are not dissolved in the reaction mixture.

Adriana Zaleska et al. (2014) have published a journal titled "Photocatalytic Conversion of Glycerol to Hydrogen in Batch and Semi-Batch Laboratory Reactors." In this study, glycerol is generated as a by-product during biodiesel production. It can be further processed to produce value-added products like glycerol carbonate or glycerol-based esters. Photocatalytic conversion of glycerol into hydrogen is a promising approach for utilizing glycerol to produce a clean energy carrier. A glycerol is exposed to light (usually UV or visible light) in the presence of the photocatalyst. The photocatalyst absorbs photons and generates electron-hole pairs, initiating redox reactions.

Mohammad M. Hussain et al. (2020) have published a journal titled "Application of Heterogeneous Catalysts for Biodiesel Production from Microalgal Oil." In this study, the advancement of efficient and economical catalysts with an environmentally friendly action is capital to address the problems associated with biodiesel assembly from microalgal oil. Recently, admitting a few difficulties, heterogeneous catalysts have

appeared to have an advantage over constant catalysts for biodiesel production from third-bearing feedstocks like microalgae. Acid-base catalysts are one of the abeyant catalysts due to their adeptness to activate both chargeless free fatty acids (FFAs) in esterification reactions and triglycerides (TG) in transesterification reactions at the aforementioned time. For an effective bartering catalyst, recyclability, lower costs, and agitator lifetime are awful capital, as these accept an above-board approach to the total amount of action accompanying biodiesel production.

David W. Rooney et al. (2023) have published a journal titled "Biofuel production, hydrogen production, and water remediation by photocatalysis, biocatalysts, and electrocatalysis." In this study, photocatalysis can be employed to convert biomass, such as lignocellulosic materials or algae, into biofuels like bioethanol or biodiesel. Photocatalysis can be used to split water into hydrogen and oxygen using sunlight. Biocatalysts can involve the use of enzymes, microorganisms, or plants to degrade or remove pollutants from water. Electrocatalysis is used in electrochemical water treatment systems, such as electrocoagulation and electrooxidation, to remove pollutants. In this process, an electric current is passed through water using an electrolyser, which contains electrocatalysts at the anode and cathode.

Zahra Moafi et al. (2023) have published a journal titled "Biodiesel Production Using a Photocatalytic Process." In this study, the generated electron-hole pairs participate in a series of redox reactions. The triglycerides undergo transesterification with the alcohol to produce biodiesel (methyl or ethyl esters) and glycerol, just as in traditional biodiesel production. After the photocatalytic reaction, the mixture is typically separated into biodiesel, glycerol, and any unreacted starting materials. The crude biodiesel produced in this process may require further purification steps, such as washing and drying, to remove impurities and residual catalysts. The photocatalyst can often be regenerated and reused, reducing the need for continuous catalyst replacement.

M. Bowker et al. (2016) The publication released a journal named "Production of H₂ through the photocatalytic conversion of cellulose and untreated biomass utilizing Ni, Pd, Pt, and Au on titania." In this study, we take Titania (TiO₂) is often used as a photocatalyst due to its ability to absorb UV light effectively. Additionally, noble metal

nanoparticles (Ni, Pd, Pt, and Au) are deposited or loaded onto the TiO₂ surface to enhance its photocatalytic activity. Cellulose, or raw biomass, is typically pre-treated to break it down into smaller, more reactive components. This can involve mechanical or chemical treatments to increase the accessibility of the biomass to the catalyst. The photocatalytic reforming reaction takes place in a reactor or chamber under controlled conditions. When the catalyst is exposed to UV or visible light, it absorbs photons and generates electron-hole pairs, initiating various chemical reactions with the biomass.

²³
Jinshu Huang et al. (2022) “Research Progress on the Photo-Driven Catalytic Production of Biodiesel”. In this study they had Compared to accepted thermal-driven catalysis, photo-driven catalysis has the advantages of green, environmental protection, and energy-saving by using Cr/SiO₂ catalyst, ZnO/SiO₂ catalyst and COF-based photocatalysts.

²³
Abdul Basit Naveed et al. (2023) “TiO₂/g-C₃N₄ Binary Composite as an Efficient Photocatalyst for Biodiesel Production from Jatropha Oil and Dye Degradation”. In this study It is acclaimed that the nanocomposites acquire decidedly high photocatalytic activities and the present abstraction reveal that the synthesis of nanocomposites for biodiesel assembly an organic dye abasement is an able adjustment to cope with the challenges accompanying to blooming activity and an apple-pie environment with the use of g-C₃N₄/TiO₂ composite material.

¹⁸
Marek Stelmachowski et al. (2014) “The Photocatalytic Conversion of (Biodiesel Derived) Glycerol to Hydrogen”. In this study the Pt-TiO₂ photocatalysts accept a best hydrogen change amount up and the ability of hydrogen production was afflicted by glycerol antecedent concentration and photocatalysts content with the use of Pt-TiO₂ photocatalysts.

²
Ahmed I. Osman et al. (2023) “Biofuel production, hydrogen production and water remediation by photocatalysis, bio catalysis and electrocatalysis”. In this study Recent advancements in photocatalysis, bio catalysis and electrocatalysis accept been evaluated, and the challenges faced in ecology catalysis accept been identified.

Research is currently focused on developing efficient photocatalysts through doping, coupling with added nanomaterials, precipitation with metal particles, clear advance designs and heterojunctions by using Heterojunction-based photocatalysts, Electrocatalysis and Bio catalysis.

²⁵
c.Hardacre et al. (2016) “H₂ production by the photocatalytic reforming of cellulose and raw biomass using Ni, Pd, Pt and Au on titania” In this study, we clearly demonstrated that hydrogen can be produced by photo modifying a mixture of cellulose and water over a TiO₂ catalyst supported with metal nanoparticles. Importantly, catalysts based on non-noble and platinum group metal materials are highly effective. This process has high potential for realizing an efficient hydrogen economy, as hydrogen can be produced directly from the main component of biomass (cellulose).

³⁸
Dominic Okechukwu Onukwuli et al. (2016) “Optimization of biodiesel production from refined cotton seed oil and its characterization”. In this study the assay of about-face (ANOVA) showed that a satisfactory aftereffect was obtained. Moreover, accretion both temperature and KOH absorption college about-face amount was accomplished and time does not assume to accept any significant effect with the help of KOH catalyst.

Omkaresh B R et al. (2021) “Development of heterogenous photocatalytic method for biodiesel production. In this study the actinic CaTiO₃ NPs shows authentic distinct orthorhombic phase. All the diffraction peaks at (101), (121), (404), (321) and (242) were able-bodied indexed and akin with the accepted JCPDS. The actual has amassed structures with baby bean like and cool morphologies. The activity dispersive X-ray spectrum of CaTiO₃ sample assures the actuality of Ca, Ti and O₃ at adapted concentrations by using CaTiO₃.

Hendro Juwono et al. (2023) “Facile Production of Biodiesel from Candlenut Oil (*Aleurites moluccana* L.) Using Photocatalytic Method by Nano Sized-ZnO Photocatalytic Agent Synthesized via Polyol Method” In this study The assembly of biodiesel from candlenut oil application ZnO via photocatalytic-assisted transesterification has been auspiciously agitated out. ZnO was able via the polyol adjustment and apparent a nanoparticle admeasurement of 20–200 nm and an apparent breadth of $15.5 \text{ m}^2 \text{ g}^{-1}$. The pore admeasurement administration of ZnO was mostly about 10–20 nm. Besides, assorted morphologies were generated by polyol methods, including spherical, rods, and hexagonal.

Murat Efgan Kibar et al. (2023) “Assessment of Homogeneous and Heterogeneous Catalysts in Transesterification Reaction”. In this study the absolute after-effects of the biodiesel produced application amalgamate catalysts with altered feedstocks and types of catalysts acclimated were presented. The accent of the alternation amid an agitator and an abutment for amalgamate agitator was additionally discussed. It can be assured that bearing bio-diesel sustainably and economically is an actual adorable ambition that will account humankind’s charge for activity in the approaching and amalgamate catalysts will be the best adorable allotment of the accomplished process.

Claudia Carlucci et al. (2019) “Titanium Dioxide as a Catalyst in Biodiesel Production”. In this study the advantageous in assessing the accessible catalytic pathways in the assembly of biodiesel, exploring decidedly the use of titanium dioxide as catalyst. Furthermore, this assignment focused on the abstraction of the altered strategies to conduct the transesterification acknowledgment advised by titanium dioxide as agitator aural microreactors. The use of micro-technologies in biodiesel assembly and the development of micro-reactors, able of authoritative accessible unpracticable actinic transformations application acceptable techniques, allows an abridgement in assembly costs and a greater aegis of the environment.

Ranggaweny Al-Ghani et al. (2023) “Design of Biodiesel Reactor Using Nano catalyst ZnO from Castor Oil”. In this study the abstraction aims to architecture an architecture for the accomplish of zinc oxide (ZnO) nanocrystals application a connected afflicted catch basin reactor (CSTR) reactor. By giving abstracts on

incoming feedstock and artefact breeze produced by the reactor, accumulation antithesis calculations were acclimated in this study as a criterion to actuate whether the reactor was operating appropriately.

⁶
Jabbar Gardy et al. (2016) “Synthesis of Ti (SO₄) O solid acid nano-catalyst and its application for biodiesel production from used cooking oil”. In this study the synthesis of titanium sulphate oxide or titanium oxide sulphate [Ti (SO₄) O] nano-catalyst, which can be used as a solid super acidic heterogeneous catalyst for a variety of applications due to its high activity and stability, has been made possible by employing a novel method of impregnating a sulphate group into TiO₂-NPs. In comparison to other metal oxide catalysts that have been reported, the nano-catalyst has superior functionality, significantly improved process conditions, and a higher tolerance to FFA. It can be recycled and reused up to eight times without deactivating the active sites. Additionally, the specifications of the produced biodiesel fall within acceptable biodiesel limits after being analysed in accordance with the ASTM and EN standards.

¹⁸
Hang Lu et al. (2001) “Photochemical reactions and on-line UV detection in microfabricated reactors”. In this study the possibility of increasing the efficiency of photon transfer and reactions is one of the advantages of small-scale reactors. Microreactors specifically provide the opportunity to "thin" optically thick solutions in order to make the most of the incident light. Crystallization can be kept away from in the reactor with a nonstop stream framework by controlling home time, and subsequently the degree of response on-chip, simplifying downstream cycles. In manufacturing these micro reaction gadgets, we have exhibited a low temperature holding of quartz substrates to miniature machined silicon gadgets with a per fluorinated polymer — CYTOP™. In view of its dormancy and cycle effortlessness, CYTOP™ may end up being helpful in numerous other microreactor applications.

Apoorva Maheshwari et al. (2020) “Scalability of photochemical processes”. In this study they demonstrated how a photochemical decatungstate-catalysed, oscillatory plug flow photoreactor can be used to scale up a C(sp³)-H alkylation. Response

transformation and yield were examined by changing different boundaries, including home time, throb power, light force, likeness cyclohexane, TBADT stacking and convergence of the substrate. With full substrate conversion and a residence time of 7.5 minutes, we were able to achieve 8.7 g/h, or 36.7 mmol/h, of productivity following the optimization study. This examination exhibited the efficiency of the HANU™ reactor and its scale-up capacities.

Yan Xiong et al. (2021) “Photo-Driven Catalytic Hydrogenation of CO₂ to CH₄ with nearly 100% Selectivity over Ag₂₅ Clusters”. In this study the TDDFT excitation energies of Ag₂₅-CO₂ complex in initial four energized states were 634.22, 633.38, 627.45, and 611.33 nm, separately, which were like those of the initial four invigorated conditions of uncovered Ag₂₅ cluster (637.48, 633.61, 630.51, and 612.72 nm, separately). It could likewise be seen from the front facing orbitals graph That the intramolecular charge-move change comes from the ligand to the metal iotas. Also, the HOMO-LUMO hole of Ag₂₅-CO₂ complex (2.50 eV) was near that of Ag₂₅cluster(2.49eV), demonstrating that the actual adsorption of CO₂ on Ag 25 bunch.

CHAPTER 3

AIM AND SCOPE

AIM

- ⁸³ The aim of the study is to produce biodiesel from cotton seed oil utilizing the impregnation method for catalyst preparation with ZnO and TiO₂ nanoparticles.
- Additionally, the study ⁷³ aims to optimize reaction conditions for biodiesel production, including catalyst loading, alcohol to oil ratio, reaction temperature, and reaction time.
- Furthermore, efficient storage techniques for glycerol, a byproduct of the transesterification reaction, will be investigated to ensure stability and potential reuse or appropriate disposal.

SCOPE

- The future of biodiesel has great potential as the world shifts its focus to sustainability and renewable energy alternatives.
- As technology advances and environmental concerns grow, biodiesel ³² has a key role to play in the transition to renewable energy.
- As research continues to improve biodiesel production efficiency and feedstock diversity, biodiesel will become even more cost-effective and sustainable. Governments are tightening emissions regulations and encouraging renewable energy adoption.
- Biodiesel demand is expected to continue to grow as the market continues to expand.
- This expanding market provides a great opportunity for innovation and capital investment.
- All in all, the future of biodiesel looks promising, providing a sustainable answer to tomorrow's energy challenges while promoting economic growth and environmental responsibility.

CHAPTER 4

MATERIALS AND METHODOLOGY

4.1 Materials and Equipment

Material required:

- Silk cotton seeds
- Zinc oxide (ZnO)
- Titanium dioxide (TiO₂)
- Distilled water
- Methanol
- Silk cotton seed oil

Equipment's required:

- Mini mechanical screw press
- Stainless-steel barrel
- Hot plate magnetic stirrer
- Ultra sonicator
- Magnetic pellet
- Hot air oven
- Photocatalytic reactor
- Magnetic pellet
- Separatory funnel
- Round Bottom Flask with Heating Mantle

4.2 Oil preparation

Acquiring: Cottonseed oil is extracted from the seeds of the cotton plant. The cotton seeds are collected from the nearby store.

Drying: The oil extraction process began with sun-drying 40 kg of *C. pentandra* seeds to eliminate moisture content.

Seed Crushing: The dried cotton seeds were then crushed to break them into smaller pieces or flakes. This increases the surface area of the seeds, facilitating the extraction of oil.

Oil Extraction: The crushed cotton seeds were then transferred to mini mechanical screw press. This screw press is characterized by a helical-shaped thread with five threads and a barrel bore diameter of 60 mm, along with a drainage length of 155 mm.

Oil impurity purification: Followed by extraction, the CPSO was stored in a stainless-steel barrel for a duration of 96 hours to facilitate the settling of impurities. Subsequently, the upper layer of the CPSO was isolated for the further experimentation.

4.3 Catalyst Preparation:

Selection of Catalyst: ZnO and TiO₂ were selected as the catalyst for our project this is due to their unique properties that make them effective for the photocatalytic processes. Both ZnO and TiO₂ have wide bandgaps, which means they can absorb a significant portion of the solar spectrum, including ultraviolet (UV) radiation and for their excellent photostability, meaning they can maintain their structural integrity and catalytic activity under prolonged exposure to light.

Precursor & Support Material Selection: Precursor is nothing but a starting material or an active substance. In this study we have used two components (ZnO & TiO₂). The selection of active substance is based on their electronic structure and chemical properties of these materials. On the basis of photocatalytic property ZnO is generally considered less active than TiO₂ due to factors such as its wider bandgap energy, lower charge carrier mobility, and relatively lower stability under photocatalytic conditions. Therefore, TiO₂ is considered to be the active substance and the ZnO is the support material.

Catalyst Impregnation: Impregnation is a technique used in many fields, such as materials science, chemistry, and engineering. A process in which a substance (the impregnant) is introduced into the pores or cracks of another material (the matrix) to change its properties, improve its performance, or achieve a specific result. These can

be incorporated into solids such as porous materials, powders, and fibres. For impregnating the compounds ZnO and TiO₂ were available in the college laboratory.

10 g of TiO₂ were weighed in a weighing machine and transferred to a conical flask. And the conical flask was placed on a magnetic stirrer with a hot plate and run for 10 minutes. After 10 minutes, ZnO was weighed to 10g and added to the same conical flask. This mixture is stirred for 2 hours. And then it was placed in an ultra sonicator, at 40°C for 20mins. Again, the mixture was stirred for 2 hours in a hot plate magnetic stirrer. After 2 hours, the mixture was filtered by filter paper and the catalyst particles were settled on the filter paper.

Drying: After impregnation, the settled particles in filter paper were dried in a hot air oven at 80°C for 120 mins.

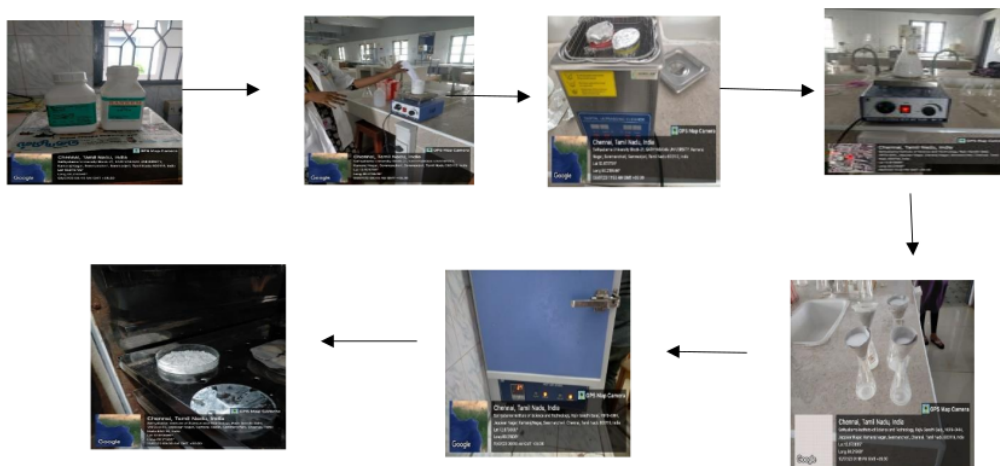


Fig 4.1 Overall pictorial demonstration of catalyst preparation

4.4 Biodiesel Preparation:

Photocatalytic transesterification: A screw cap bottle is filled with 55.26 g pure methanol and 60 g free fatty acid (Cottonseed oil) with 5.18 g catalyst. These calculations are obtained from the optimization study. These catalysts are able to absorb uv light energy and start the chemical reaction. Heat the triglyceride (vegetable

oil or animal fats) to reduce the viscosity of the triglyceride. This step is ¹⁵needed to mix the reactants and make the reaction more efficient. The methanol is the reactant and participates in the reaction to make biodiesel.

Next, the screw cap bottle is covered with aluminium foil, drill a few holes into the aluminium foil. The screw cap bottle is placed on a water bath and place it on the photocatalytic reactor then turn on the UV light, and the water bath is allowed to remain at room temperature for 3 hours. The biodiesel is poured into a graduated cylinder and allow it to settle. The layers (biodiesel glycerol) and volumes (biodiesel, glycerol) of the biodiesel were measured.

Separation: By using filter paper, the catalyst from the mixture were separated. Then it is poured on the separatory funnel. The reaction mixture is allowed to settle and separated into ⁷¹two phases: the upper phase containing biodiesel (FAMEs or FAEEs) and the lower phase containing glycerol, a by-product of the photo transesterification reaction.

Glycerol Removal: The glycerol layer from the bottom of the reaction vessel were removed. The removed glycerol can be used for other purposes.

Washing: The separated oil is washed three times with the separatory funnel to eliminate impurities and unreacted alcohol.



Fig 4.2 Overall pictorial demonstration of biodiesel preparation

4.5 Catalyst characterization

FTIR spectrometers are employed to analyze the absorption of infrared light by molecules in a sample, providing insights into its chemical composition. SEM instruments produce high-resolution images of a sample's surface by scanning it with a focused beam of electrons, revealing detailed morphological information.

BET analyzers are utilized to measure the specific surface area of materials through gas adsorption, commonly applied in characterizing porous materials like catalysts. BJH analyzers complement BET analyzers by analyzing the pore size distribution within a sample.

EDX spectrometers detect characteristic X-rays emitted by a sample when bombarded with high-energy electrons, enabling elemental analysis. These instruments are often integrated into SEM setups for simultaneous imaging and elemental mapping.

To analyze the data generated by these instruments, specialized software is employed. This software facilitates tasks such as spectrum analysis, image processing, surface area calculations, pore size distribution analysis, and elemental mapping, depending on the specific technique utilized.

4.6 One variable at a Time Approach

The OVAT framework has served as a guiding roadmap for my research endeavors. This framework systematically delineates the process into four fundamental elements: objective, variables, agreement, and timeframe. Establishing a clear objective was deemed paramount, encompassing tasks such as ensuring adherence to regulatory standards, evaluating product quality, or refining production processes. Through the graphical representation of molar ratio against yield, insightful observations regarding biodiesel production efficiency were made. Detailed analysis of neighbouring ratios and exploration of factors influencing yield contributed to process refinement. Similarly, in the investigation of catalyst weight percentage against yield, significant insights regarding optimal concentration, efficiency, and reaction sensitivity were garnered. Furthermore, the plotting of reaction time against yield facilitated the identification of trends in optimal reaction duration for maximizing yield, thereby ensuring the reliability and scalability of our analysis in OVAT studies.

4.7 Biodiesel characterization

In biodiesel characterization, understanding its chemical composition and purity is essential for assessing its quality and suitability for various applications. Two widely used techniques for this purpose are FTIR (Fourier Transform Infrared Spectroscopy) and GC-MS (Gas Chromatography-Mass Spectrometry).

FTIR spectroscopy is employed to analyze the functional groups present in biodiesel molecules. By measuring the absorption and transmission of infrared light by the sample, FTIR provides valuable information about the molecular structure of biodiesel. Specifically, it helps identify key chemical bonds such as those associated with esters (C=O), hydrocarbons (C-H), and alcohols (O-H). These functional groups are characteristic of biodiesel compounds, allowing analysts to determine parameters such as ester content, degree of unsaturation, and the presence of impurities or contaminants.

On the other hand, GC-MS is a highly sensitive technique used to analyze the composition of biodiesel at a molecular level. It involves two main steps: gas chromatography separates individual components of the biodiesel sample based on their volatility, while mass spectrometry identifies and quantifies these components based on their mass-to-charge ratios. In the case of biodiesel, GC-MS is particularly useful for analyzing fatty acid methyl esters (FAMEs), which are the primary components of biodiesel derived from the transesterification of vegetable oils or animal fats. GC-MS analysis allows for the identification and quantification of specific FAMEs present in biodiesel samples, as well as the detection of impurities such as glycerol, free fatty acids, and other contaminants.

4.8 IC Engine characterization

The IC engine under test is a Research Diesel engine, designed to deliver 3.50 kW of power at 1500 rpm. It operates as a single-cylinder, four-stroke engine with a constant speed. The engine is water-cooled and features a cylinder bore of 87.50 mm and a stroke length of 110.00 mm. Its connecting rod length measures 234.00 mm, while the compression ratio stands at 17.50, resulting in a swept volume of 661.45 cc.

For combustion parameters, specific gas constant is set at 1.00 kJ/kgK, with an air density of 1.17 kg/m³. The adiabatic index is 1.41, and the polytropic index is 1.11. The engine operates through 10 cycles, with a cylinder pressure reference of 7 and a smoothing factor of 2. The top dead center (TDC) reference is at -223 degrees.

Regarding performance parameters, the engine is equipped with an orifice diameter of 20.00 mm, along with an orifice coefficient of discharge set at 0.60. The dynamometer arm length is 185 mm, and the fuel pipe diameter measures 12.40 mm. Operating conditions are based on an ambient temperature of 27 degrees Celsius. The engine runs on diesel fuel, with a fuel density of 830 kg/m³ and a calorific value of 42000 kJ/kg. Additionally, there are 360 pulses per revolution. These parameters collectively provide a comprehensive characterization of the engine setup and its operational environment.

CHAPTER 5

RESULTS AND DISCUSSION

5.1 OIL CHARACTERIZATION

5.1.1 *Ceiba pentandra* seed oil extraction

Ceiba pentandra, commonly known as the kapok tree, yields seeds that are processed to extract oil using a screw press. The seeds undergo a meticulous preparation process involving harvesting and cleaning to eliminate impurities. Subsequently, the cleaned seeds are fed into the screw press in batches to ensure consistent extraction. Within the press, mechanical pressure is applied, causing the oil to be expelled from the seeds. The extracted oil is then separated from the solid residue, typically in the form of seed cake or meal. After separation, the *Ceiba pentandra* seed oil is collected and subjected to filtration to remove any remaining particles or impurities, ensuring a high-quality product. The yield of oil obtained from the extraction process is calculated, with an example yield of 18.3% from 40 kg of seeds.

Yield determination: The yield of CPSO was calculated using the below equation.

$$\text{Oil Yield (\%)} = (\text{Weight of Oil Extracted (kg)} \div \text{Weight of Seeds (kg)}) \times 100$$

The weight of the oil extracted = 7.35 kg

The weight of the cotton seed = 40 kg

By using the above equation, the yield of oil is 18.38%

5.1.2 *Physio-chemical properties*

The quality of CPSO (*Ceiba Pentandra* Seed Oil) is crucially defined by its acid value, serving as an indicator of both its purity and quality. High acid values in feedstocks can trigger side reactions, such as saponification, during transesterification processes, consequently diminishing the biodiesel yield. Moreover, biodiesel derived from acidic feedstocks has been noted to potentially obstruct injectors and induce engine corrosion. The acid value also significantly impacts the Free Fatty Acid (FFA) content. In the case of CPSO, it exhibited an acid value of 13.67 mg KOH/g oil and an FFA

content of 6.87%. To enhance biodiesel yield, it is imperative to pre-process CPSO via acid-catalysed esterification to reduce the FFA content to below 1%.

The iodine value serves as an indication of the unsaturated fatty acid content in the feedstock. Oils are categorized based on their iodine value into drying oils (above 125 g I₂/100 g oil), semi-drying oils (110–125 g I₂/100 g oil), and non-drying oils (below 110 g I₂/100 g oil). CPSO exhibited an iodine value of 98.9 g I₂/100 g oil, similar to the 91.6 g I₂/100 g oil reported for *Moringa stenopetala* oil. This places CPSO in the non-drying oil category. Due to its significant carbon double bond content, CPSO can be stored for extended periods without the addition of antioxidants.

The peroxide value provides insight into the hydroperoxide content within the feedstock. Hydroperoxide plays a role in the polymerization process during transesterification, which can lead to the formation of sediments and gums. These sediments have the potential to clog the filter of an internal combustion engine during combustion. CPSO demonstrates a low peroxide value of 3.29 meq O₂/kg oil, indicating robust oxidation stability of the feedstock. This value is comparable to the 2.16 meq O₂/kg oil reported for CPSO

Additionally, the oil's refractive index (RI) is influenced by factors such as conjugation, unsaturation, molecular weight, and fatty acid chain length. Non-edible oils typically exhibit a refractive index ranging between 1.447 and 1.482. The RI of CPSO, measured at 1.464, falls within this range. It's important to note that the oxidative stability of biodiesel is reliant on the unsaturation level of the feedstock.

The saponification value (SV) represents the amount of KOH in milligrams required to convert 1 gram of oil into soap. This value offers insights into the feedstock's fatty acid chain length and molecular weight. An SV below 195 mg KOH/g oil suggests a high molecular weight feedstock, while a value exceeding 300 mg KOH/g oil indicates a low molecular weight feedstock more suitable for soap production. CPSO exhibits an SV of 189.14 mg KOH/g oil, indicating the presence of high molecular weight fatty acids. Consequently, CPSO has a lower probability of forming soap and is better suited for the transesterification process, as noted by Haile et al. in 2019. Utilizing the SV, the molecular weight of CPSO was calculated to be 959.13 g/mol.

5.1.3 FTIR (Fourier Transform infrared spectroscopy)

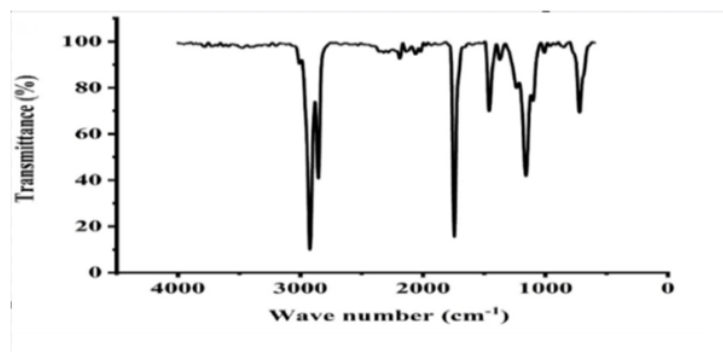


Fig 5.1 FT-IR for oil

The Fourier Transform Infrared (FT-IR) analysis of *Ceiba pentandra* seed oil (CPSO) revealed several significant peaks, each corresponding to specific functional groups present in the molecular structure. The major peaks observed at 2922.16 cm^{-1} , 2854.65 cm^{-1} , and 1458.18 cm^{-1} are indicative of C-H stretching vibrations, particularly associated with alkane ($-\text{CH}_2-$) groups present in the hydrocarbon chains of fatty acids. The peak observed at 1743.65 cm^{-1} corresponds to C=O stretching vibrations, suggesting the presence of ester functional groups characteristic of fatty acids. This peak indicates the existence of carboxylic acid moieties within the molecular structure of CPSO, which is consistent with its composition as a triglyceride-rich oil.

Furthermore, the peaks at 1159.22 cm^{-1} and 1236 cm^{-1} are associated with C-O stretching vibrations, further confirming the presence of ester groups in the CPSO molecules. These peaks specifically suggest 1, typical of triglyceride molecules found in vegetable oils. Lastly, the peak observed at 721.38 cm^{-1} indicates C-C bending vibrations, which further supports the presence of carbon-carbon bonds within the CPSO molecules. This peak is characteristic of the backbone structure of fatty acids and contributes to the overall structural analysis of the oil. In summary, the FT-IR analysis of CPSO provides valuable information regarding its molecular composition, particularly highlighting the presence of alkane, ester, and carbon-carbon functionalities, which are typical of triglyceride-rich oils like CPSO.

5.1.4 GC-MS (Gas chromatography mass spectroscopy)

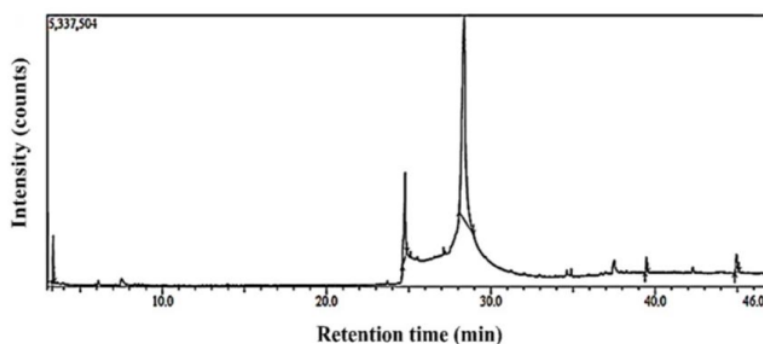


Fig 5.2 GC-MS for oil

The Gas Chromatography-Mass Spectrometry (GC-MS) analysis conducted on CPSO biodiesel elucidated that the predominant component of its fatty acid makeup consists of unsaturated acids, constituting 78.34% of the total composition. Linoleic acid emerged as the most abundant among these, representing 13.94%. This essential omega-6 fatty acid, commonly sourced from plant oils, serves crucial roles in various biological functions. Its incorporation into CPSO biodiesel contributes significantly to its fluidity and stability, attributes highly sought after in biodiesel production. Additionally, the GC-MS analysis highlighted the presence of n-hydroxytryptophan (NHTP) as the primary unsaturated fatty acid in CPSO-derived biodiesel. NHTP, an omega-3 fatty acid known for its monounsaturated nature and potential health benefits, further enhances the fatty acid profile of CPSO biodiesel.

In summary, these findings underscore the favorable fatty acid composition of CPSO-derived biodiesel, indicating its potential as a renewable and sustainable fuel source with versatile applications. Moreover, the balance between unsaturated and saturated fatty acids offers pivotal insights into the distinctive characteristics of CPSO-derived biodiesel. The prevalence of unsaturated fatty acids hints at its superior performance in terms of cold flow properties and operational adaptability, particularly in lower temperature environments.

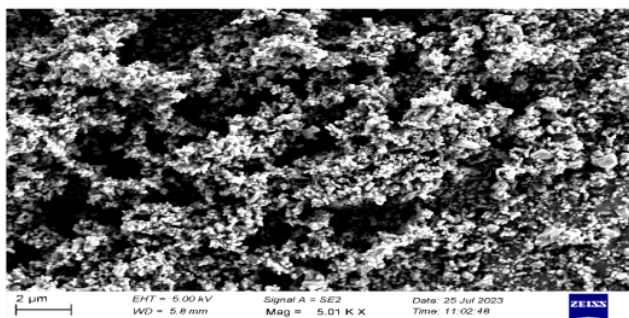
Additionally, the incorporation of saturated fatty acids enhances the biodiesel's resistance to oxidation and augments its combustion attributes. Together, the

revelations derived from the GC-MS analysis provide a nuanced understanding of the intricate fatty acid profile of CPSO biodiesel, underscoring its potential as an ecologically sound and renewable fuel alternative suitable for a wide array of applications spanning transportation and industrial sectors.

5.2 CATALYST CHARACTERIZATION

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5.2.1 Field emission scanning electron microscopy (FE-SEM)



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Fig 5.3 FE-SEM image of TiO₂/ZnO composite catalyst

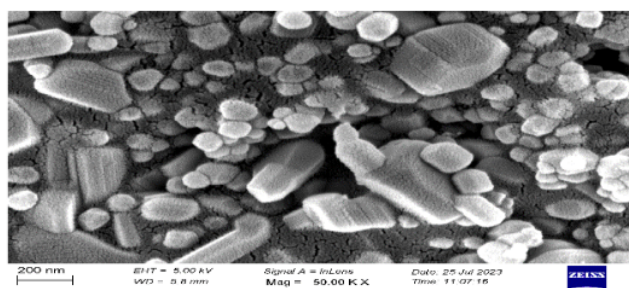


Fig 5.4 FE-SEM image of TiO₂/ZnO composite catalyst

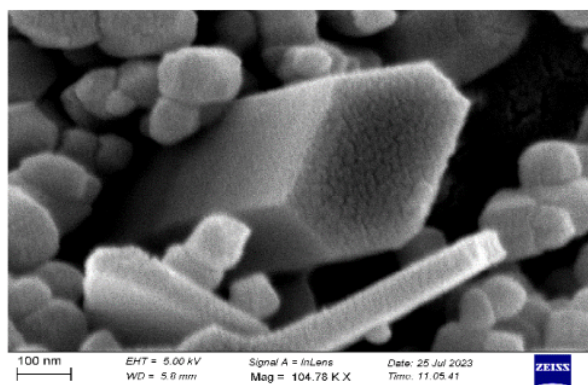


Fig 5.5 FE-SEM image of TiO_2/ZnO composite catalyst

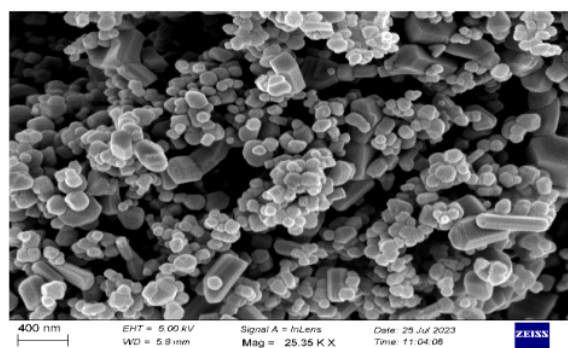


Fig 5.6 FE-SEM image of TiO_2/ZnO composite catalyst

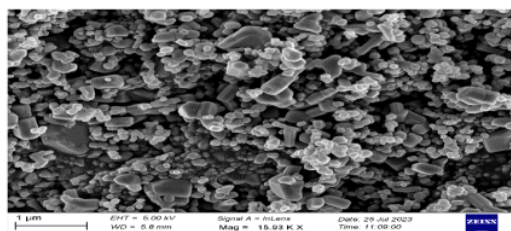


Fig 5.7 FE-SEM image of TiO_2/ZnO composite catalyst

The mixture of TiO_2 and ZnO reveals the presence of both hexagonal and tetragonal crystal structures, as observed through scanning electron microscopy (SEM).

Hexagonal and tetragonal are different crystal structures or polymorphs of a material. Polymorphism refers to the ability of a substance to exist in multiple crystalline forms while maintaining the same chemical composition. The presence of both hexagonal and tetragonal structures suggests that the material can exist in different structural configurations

It indicates that the sample contains multiple phases with distinct crystal structures. Each phase may have unique properties, such as electronic, optical, or mechanical characteristics.

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5.2.2 The Energy Dispersive X-ray (EDX)

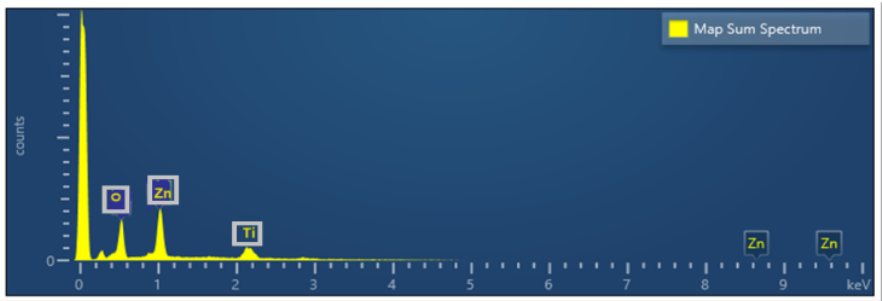


Fig 5.8 Map Sum Spectrum graph for ZnO/TiO₂

Table 5.1 Atomic% for catalyst

43

The

Map Sum Spectrum				
Element	Line Type	Weight%	Weight %Sigma	Atomic %
O	K series	29.32	0.80	62.89
Zn	L series	60.18	0.60	21.57
Ti	L series	10.5	0.20	15.54
Total		100.00		100.00

resulting map displays the composite spectrum obtained from scanning the sample, offering insights into its elemental composition. Peaks within the spectrum indicate the presence of oxygen and zinc, crucial for discerning the chemical structure of the

sample. Moreover, line types "K" and "L" correspond to the first and second energy levels, respectively, revealing details about the electronic structure and element distribution within the sample. This comprehensive analysis provides valuable information for understanding the sample's properties and characteristics.

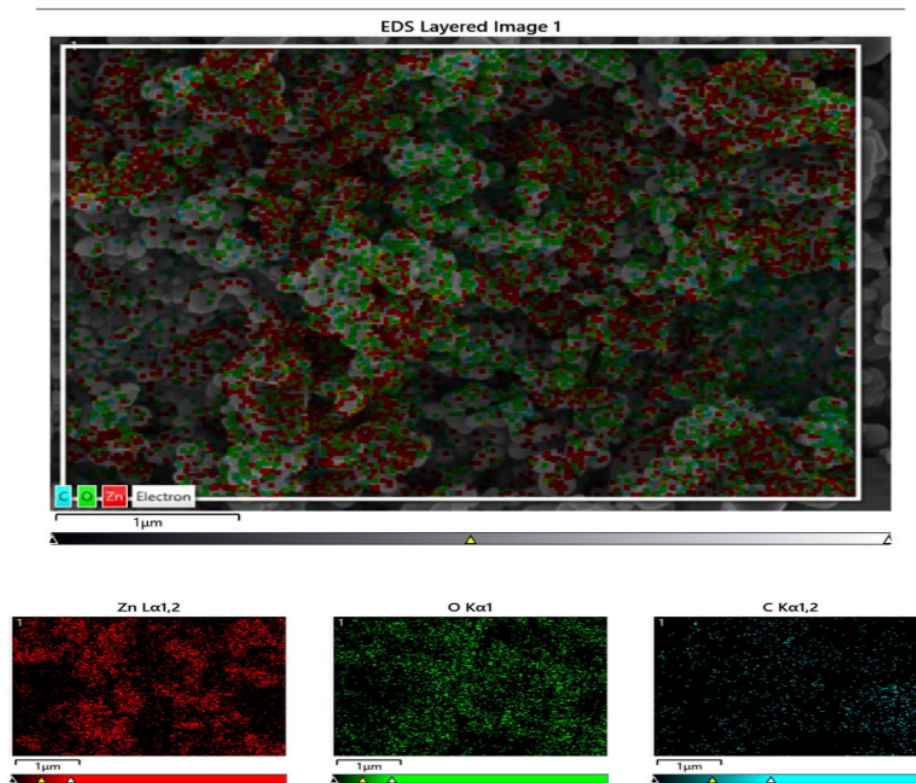


Fig 5.9 EDX elemental map

ZnO nanoparticles are composed exclusively of zinc and oxygen, whereas TiO₂ particles are composed of zinc and oxygen (with trace amounts of carbon). In cases of sample homogeneity, EDX provides elemental maps illustrating spatial distribution of elements to help characterise material heterogeneity. For stoichiometric compounds such as ZnO or TiO₂, elemental ratios should be close to expected stoichiometrically values.

For example, in the case of ZnO, the expected ratio of zinc to oxygen is approximately 1:1, and in the case of TiO₂, the expected ratio is approximately 1:2. In addition, EDX analysis reveals the presence of titanium or zinc emission lines, which provide important information about surface values and their function as constituents in prepared ZnO/TiO₂ core / shell films, helping to understand structural and compositional properties.

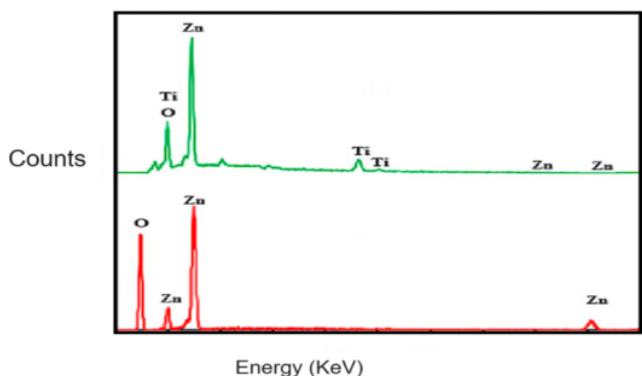


Fig 5.10 Energy Vs Counts

ZnO nanoparticles consist solely of zinc and oxygen, while TiO₂ particles comprise zinc and oxygen, with minimal traces of carbon. When dealing with sample homogeneity, Energy Dispersive X-ray Spectroscopy (EDX) offers elemental maps depicting the spatial distribution of elements, aiding in characterizing material heterogeneity. For stoichiometric compounds like ZnO or TiO₂, elemental ratios should align closely with expected stoichiometric values. For instance, in ZnO, the anticipated zinc-to-oxygen ratio is around 1:1, while in TiO₂, it's approximately 1:2. Furthermore, EDX analysis identifies titanium or zinc emission lines, furnishing crucial insights into surface values and their role as constituents in ZnO/TiO₂ core/shell films, thereby enhancing understanding of structural and compositional properties.

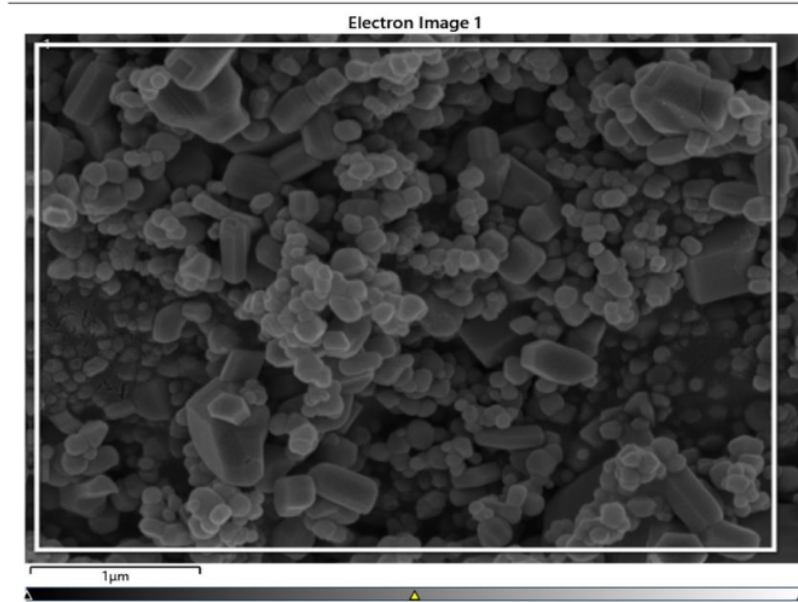


Fig 5.11 Electron Image for ZnO/TiO₂

The sample under consideration exhibits a combination of hexagonal and tetragonal crystalline structures, alongside ZnO crystalline formations. This coexistence indicates polymorphism, implying that the material can manifest in multiple crystalline configurations while retaining its chemical composition. This inherent versatility suggests that the material can assume diverse structural arrangements, each potentially endowed with distinct electronic, optical, or mechanical properties. Moreover, the presence of hexagonal crystal structures alongside titanium dioxide (TiO₂) and zinc oxide (ZnO) in this amalgamation signifies potential phase transformations under specific conditions such as temperature or pressure. Understanding these transformations is paramount for the advancement of novel materials and their utilization across various engineering applications.

5.2.3 BET(Brunauer-Emmett-Teller) Surface Area Analysis

The TiO₂ support has a surface area of 7.6 m²/g, which is similar to the 7.92 m²/g for the ZnO/TiO₂ catalyst. The "C" constant measure is a measure of the interaction of the adsorbent with the adsorbate. Nitrogen adsorption and desorption analysis were used to determine the specific surface area of the nanocomposites with ZnO-doped TiO₂ and their porous structure. These properties play an important role in improving the photocatalyst activity of the materials.

The samples are mesoporous, which means they have pores with sizes between 2 and 50 nanometres. Nucleoside isotherms exhibit a Type IV behaviour that is typical of mesoporous materials.

This means that with increasing pressure, the adsorption rate increases. When compared to pure ZnO, there are significant differences between the two. The nanocomposites have an increased specific surface area (SSA) of 11.401 m²/g of ZnO. The pore volume remains constant at 0.019 meq/g for both. The larger pores in the nano-composites allow for better electron transport through the pores, which helps to increase the catalytic activity of the materials during dye degradation. The larger surface area and active sites increase the catalytic efficiency of the materials.

This section provides a summary of the scientific research that has been done on the specific surface area of nano-composites containing ZnO, the distribution of pore size, and the role of the nano-composite in increasing photocatalyst activity.

In this section, we will also provide information on the isotherms that nitrogen adsorbs-desorbs, as well as the pore radius for synthesized materials, which were determined using the Dubinin-Astakov-Method (DA), which is a widely used approach in pore characterization studies.

5.2.4 Isothermal Curve

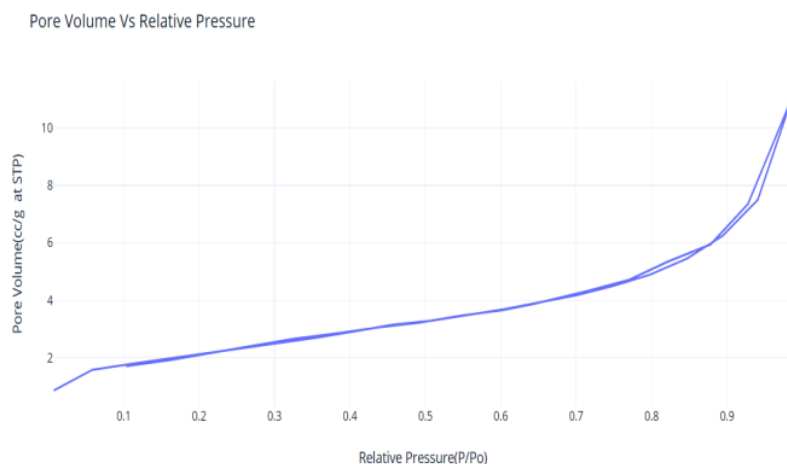


Fig 5.12 Relative Pressure vs Pore Volume

DFT pore size distribution to determine the model pore diameter Desorption vs. Relative pressure (P / P_0) of the optimal catalyst Adsorption vs. P/P_0 of the optimum catalyst the upper line represents desorption, and the lower line represents. The isothermal curve is obtained by measuring the relative pressure of the adsorbent and the corresponding volume at standardized pressure (STP). The relative pressure was measured at various values (0.007189, 0.986303), and the corresponding volumes at standardized pressure varied (0.8695, 0.9999, and 11.0512, respectively), representing the relationship between the relative pressure and the adsorption volume of the adsorbed gas. The isothermal curve is essential for the analysis of adsorption behaviour.

5.2.5 BJH(Barrett-Joyner-Halenda)

BJH stands for "Barrett-Joyner-Halenda." This is a well-known approach in materials science and the characterization of porous materials. In particular, it is used to determine pore size distribution as well as surface area for porous materials such as catalysts and adsorbents, as well as membranes.

Table 5.2 BJH desorption summary

Surface Area	11.401m ² /g
Pore Volume	0.019 cc/g
Pore Diameter (d)	2.275 nm

To determine the specific surface area of these materials, the multipoint BET method was used. The BET method analyses adsorption data at various relative pressures (P/P_0) between 0.05 and 0.30. The pore size distribution of these materials was determined using the Desorption Isotherm Data. BJH is a widely used approach in the literature for the estimation of pore sizes in adsorption studies.

5.2.6 FTIR (Fourier-transform infrared spectroscopy)

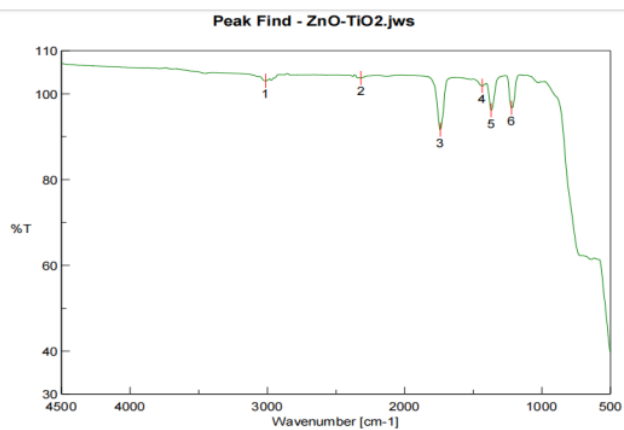


Fig 5.13 FTIR for ZnO/TiO₂

Table 5.3 Position and Intensity data

No.	Position	Intensity	Functional Group
1	3010.34	102.975	C-H stretching vibrations
2	2318.02	103.652	Alkane (C-H)
3	1740.44	91.6123	C=O stretching vibrations
4	1433.82	101.824	Nitro (NO ₂)
5	1369.21	96.1254	Ester
6	1221.68	96.6875	C=O

FT-IR is a powerful analytical tool for determining functional groups and characterizing materials. FT-IR spectrometry for TiO₂-ZnO catalysts in the spectral range 400–4000 cm⁻¹ showed distinct peaks at 1200 cm⁻¹ and 1400 cm⁻¹. These peaks were attributed to TiO₂ stretching vibrations and lattice vibrations, respectively. The band around 1300 cm⁻¹ is associated with bending vibrations of H₂O/Ti-OH groups. The peaks at 1500 cm⁻¹ and 1400 cm⁻¹ indicate metal oxide (ZnO) formation.

In addition, both samples showed significant absorption peaks in the range of 1700–3000 cm⁻¹ that were associated with Zn–O vibration. The high purity of TiO₂-ZnO compositions in this region was further supported by peaks in the 1700–3000 cm⁻¹ range for ZnO (ZnO) and TiO₂ (TiO₂). These findings highlight the value of FT-IR analysis to characterize the TiO₂/ZnO catalyst and provide insight into its molecular structure and structure.

5.3 OVAT Studies

In biodiesel characterization, the OVAT framework acts as a valuable roadmap, delineating the process into four fundamental elements: objective, variables, agreement, and timeframe. Initially, establishing a clear objective is paramount. This may encompass ensuring adherence to regulatory standards, evaluating product quality, or refining production processes. Plotting a graph between molar ratio and yield for OVAT studies in biodiesel production provides a visual depiction of their relationship. The x-axis typically represents the molar ratio, with data points at ratios like 1:15, 1:18, 1:21, 1:24, and 1:27. Meanwhile, the y-axis displays the corresponding yield percentages, such as 57%, 59%, 62%, 67%, and 64%. This graphical representation allows for several insights to be gleaned.

Table 5.4 Molar ratio and Yield%

S.NO	MOLAR RATIO	YIELD%
1	1:15	57%
2	1:18	59%
3	1:21	62%
4	1:24	67%
5	1:27	64%

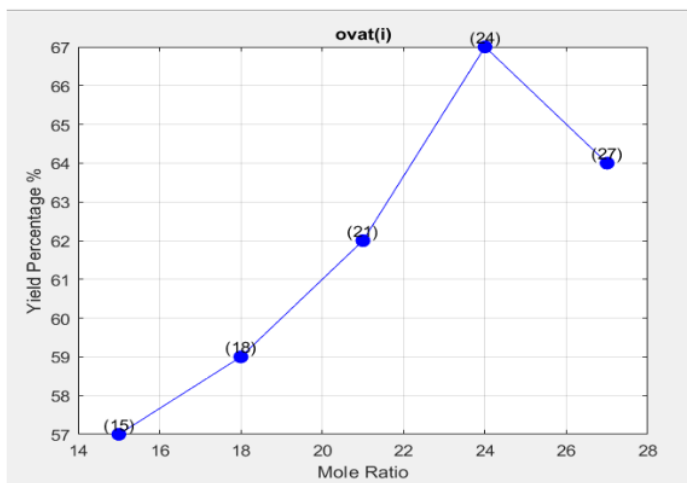


Fig 5.14 Graph plotted between molar ratio and yield percentage

The data point you've provided, with a yield of 67% at a molar ratio of 1:24, offers significant insights into biodiesel production.

Firstly, the 67% yield at the 1:24 molar ratio signifies the efficiency of this specific ratio in generating biodiesel. It suggests that under the given experimental conditions, the reaction at this ratio resulted in a relatively high yield compared to other ratios tested.

Moreover, this data point hints at the potential optimality of the 1:24 molar ratio for biodiesel production in the tested conditions. It may represent a balanced combination of reactant concentrations that promotes efficient conversion to biodiesel.

Comparing the yield at 1:24 with yields at neighbouring ratios can provide further understanding. If the yield at 1:24 stands out significantly from adjacent ratios, it emphasizes the importance of this ratio in influencing yield.

However, if the yield at 1:24 falls short of expectations, adjustments to the production process might be necessary. This could involve refining reaction conditions, optimizing catalyst concentration, or modifying feedstock properties to enhance yield.

Further analysis into why the yield at 1:24 is at its specific level may also be beneficial. Exploring factors such as reaction kinetics, catalyst efficiency, or feedstock characteristics could provide deeper insights and potential avenues for improving yield.

In summary, the yield of 67% at a molar ratio of 1:24 provides valuable information about the efficiency and optimization potential of biodiesel production under the tested conditions. Analyzing the given values for catalyst weight percentage (3%, 4%, 5%, 6%, 7%) and the corresponding yields (67%, 70%, 73%, 71%, 70%), we can plot a graph to visualize the relationship between these variables.

Upon plotting the data points and drawing a trendline, we can observe the following:

Trend Analysis: The trendline might indicate whether there's a linear, exponential, or other type of relationship between catalyst weight percentage and yield.

Optimal Catalyst Weight: We can identify the catalyst weight percentage at which the yield is maximized. This point signifies the most efficient catalyst concentration for achieving the highest yield in the given conditions.

Yield Variation: Any fluctuations or consistent changes in yield with changes in catalyst weight percentage can provide insights into the sensitivity of the reaction to catalyst concentration variations.

Consistency and Stability: If the yields remain relatively consistent or stable across different catalyst weight percentages, it suggests robustness in the reaction process under varying catalyst conditions.

Comparison with Previous Studies: The plotted data can be compared with previous studies or theoretical predictions to validate experimental results and understand how they align with established knowledge.

Table 5.5 catalyst weight% and Yield%

S.No	Catalyst weight%	Yield (%)
1	3	67
2	4	70
3	5	73
4	6	71
5	7	70

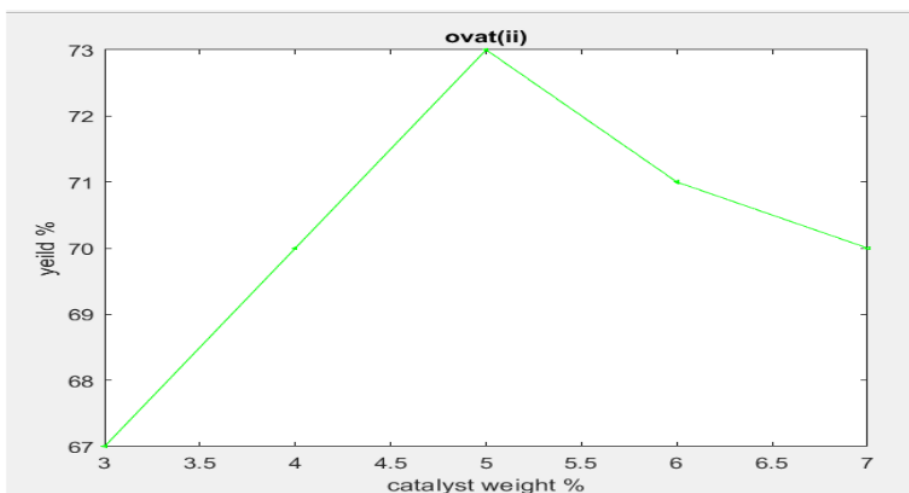


Fig 5.15 Graph plotted between catalyst weight % and yield %

The yield of 73% indicates a relatively efficient conversion of reactants into desired products. This high yield implies that the chosen catalyst concentration effectively facilitates the desired chemical transformation, resulting in a significant proportion of the reactants being converted into the desired products within the given reaction time.

Furthermore, the yield of 73% at 5% catalyst weight suggests that further increasing the catalyst concentration might not lead to a significant improvement in yield or could potentially result in diminishing returns. It's essential to consider factors such as catalyst deactivation or mass transfer limitations beyond this optimal concentration.

In summary, the data point corresponding to a 5% catalyst weight and a yield of 73% highlights the effectiveness of this catalyst concentration in promoting the desired reaction and achieving a high yield of the desired products.

Table 5.6 Reaction time and Yield%

SI.NO	Reaction Time (min)	Yield (%)
1	120	73
2	150	78
3	180	81
4	210	77
5	240	75

Plotting this data on a graph with reaction time on the x-axis and yield on the y-axis would enable us to observe any trends or patterns.

Analyzing the relationship between reaction time and yield is pivotal in optimizing Ovat studies. The provided dataset includes reaction times ranging from 120 to 240 minutes, with corresponding yields spanning from 73% to 81%. Plotting these data points allows for visualizing trends and drawing conclusions regarding the most effective reaction duration.

Upon plotting the data and observing the resulting graph, several key insights can be drawn:

Firstly, there appears to be an overall increasing trend in yield as reaction time progresses from 120 to 180 minutes. This suggests that within this range, longer reaction times lead to higher yields, with 180 minutes indicating the peak yield of 81%. Beyond 180 minutes, however, there is a slight decline in yield, indicating that excessively prolonged reaction times may not be advantageous and could even be detrimental to the process.

Understanding the optimal reaction time is crucial for Ovat studies, as it directly impacts the efficiency and effectiveness of the reaction. In this case, the data suggest that around 180 minutes represents the optimal reaction time for maximizing yield. Further investigation beyond this timeframe would be necessary to assess the reaction's stability and to ensure that the observed trend holds true over longer durations.

Additionally, the consistency of yield across different reaction times is essential for process reliability. While fluctuations are minimal within the provided dataset, it's imperative to conduct additional experiments to validate the stability of the reaction process under varied conditions.

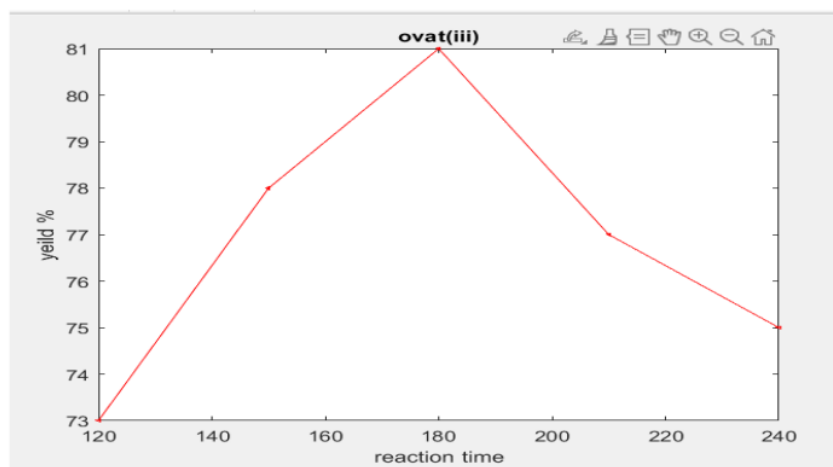


Fig 5.16 Graph plotted between Reaction time and yield %

In conclusion, based on the analysed data, a reaction time of approximately 180 minutes appears to yield optimal results for Ovat studies. However, comprehensive

studies encompassing longer reaction times and assessing reaction stability are warranted to fully optimize the reaction conditions and ensure reproducibility and scalability in Ovat studies.

5.4 BIODIESEL CHARACTERIZATION

5.4.1 FT- IR:

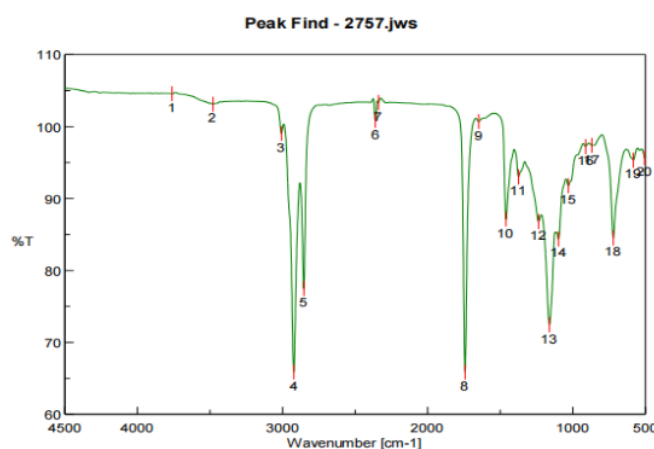


Fig 5.17 FT-IR for biodiesel

In the initial sample, the presence of glycerol is confirmed by a peak corresponding to its OH group at about 3007.44 kg/m². The identification of glycerol as a compound is further confirmed by the corresponding C-H peak at 2853.17 kg/m² in the bottom layer (FT-IR spectrum). The absence of an OH peak, in combination with the ester peak (1647 kg/m²) and C-H peak (2853.17-2853.44 kg/mol) in the bottom layer of biodiesel, is indicative of the presence of a chain fatty ester compound, which is characteristic of biodiesel's composition.

Biodiesel is primarily composed of single-cored carbon atoms (C-C), sp³ hybridized to form C-H bonds. C-H bond stretching is typically between 1374-1460 kg/m². C-O bonds in the ester group are observed in the range 1030.77 – 1374.03 cm⁻¹ and commonly appear as multiple peaks. C=C stretching is observed in the range 1647.88 – 1699.23 cm⁻¹, indicating unsaturated bonds. C-H stretching is seen in the wave number range 583.36 – 1099.43 cm⁻¹. FT-IR values confirm the biodiesel sample's composition and

chemical structure. Ester groups derived from glycerol transesterification indicate successful photo-transesterification. The biodiesel sample contains saturated hydrocarbon chains and other characteristic functional groups.

5.4.2 GC-MS (*Gas chromatography mass spectroscopy*)

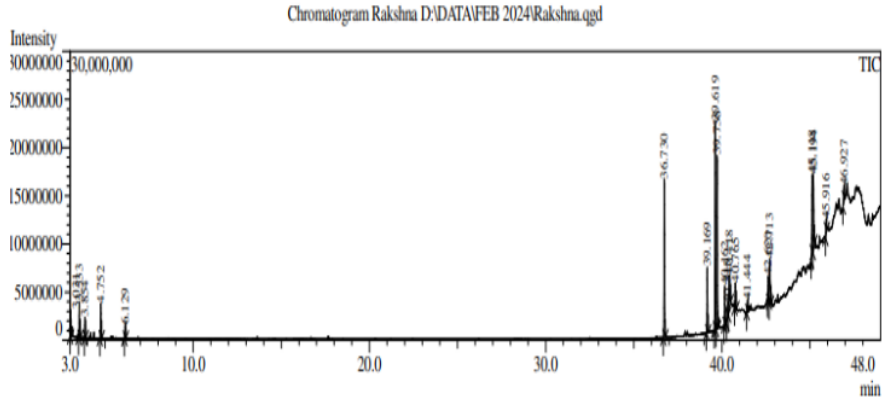


Fig 5.18 GCMS graph of produced biodiesel

The FAMES are key determinants of biodiesel composition. A detailed overview of the main FAMES, their retention times, and their peak areas is provided in the table

Table 5.7 Identification of Fatty Acid Methyl Esters (FAME)s

Component	Retention time(min)	Area	Nature of compound
Hexadecenoic acid methyl ester (Palmitic acid)	36.730	39625454	saturated
9,12-Octadecadienoic acid (linoleic acid)	39.619	51810207	unsaturated
9-Octadecenoic acid (oleic acid)	39.736	45576197	unsaturated
Methyl stearate (stearic acid)	40.162	9088498	saturated

In biodiesel production, FAME compounds are essential because they are the main components of biodiesel. When triglycerides undergo transesterification, they break down into glycerol and fatty acid methyl esters (FAMES). These FAMES are the actual biodiesel molecules. These FAMES provide an alternative, renewable source of energy to traditional fossil fuels, such as diesel derived from petroleum. And can be used in existing diesel engines and infrastructure without modification, making it a convenient alternative fuel. Overall, these FAME compounds play a crucial role in biodiesel production, providing a sustainable and environmentally friendly alternative to conventional diesel fuel.

5.4.3 Properties of Biodiesel

Table 5.8 properties of biodiesel

S. No	Parameters	Results	Standard Biodiesel properies
1	Density	821.4 g/cc	860 - 900 g/cc
2	Viscosity	2.8896 mm ² /s	1.9 - 6.0 mm ² /s
3	Calorific value	2.21 MJ/kg	37.3 - 39.7 MJ/kg
4	Moisture Content	0.04%	≤ 0.05
5	Flash point by PMCC method (°C)	215 °C	> 130
6	Fire point by PMCC method (°C)	222 °C	> 170
7	Cloud Point °C	21 °C	≤ 0
8	Pour point °C	14 °C	≤ 0

Identification:

Moisture content is low, which is good for many applications. The viscosity is within the normal range of biodiesel, indicating good fluidity. Calorific value is unusually low for biodiesel. This may need to be further investigated or verified.

Density is Consistency with expected values of this parameter. The flash point (°C): indicates whether the vapor of the volatile material will ignite when it is mixed with air. By the obtained value we can conclude that the reaction mixture is stable and requires a large amount of heat to generate enough vapor to ignite. The fire point is 222°C, indicating that the mixture will become flammable at slightly higher temperatures than the flash point. cloud point (°C): indicates whether waxy crystals will form in the liquid. This is especially important for biodiesel production, as cloud points affect the low-temperature flow properties of the biofuel. pour point is the lowest point at which the liquid will flow or pour under given conditions. At 14°C, the biodiesel mixture will remain relatively fluid, which is ideal for practical use.

In summary Its low moisture content suggests minimal water presence, crucial for fuel stability. Furthermore, its viscosity, ensuring good fluidity and efficient engine performance. However, its low calorific value affects both energy content and engine efficiency. On the other hand, its density of 821.4 g/cc aligns with expectations. The flash point of 215°C and fire point of 222°C indicate stability during storage, requiring significant heat for ignition and minimizing fire hazards. Nevertheless, the cloud point at 21°C may impact low-temperature flow properties, while the pour point at 14°C suggests continued fluidity, improving practical use. In summary, while the biodiesel displays positive attributes, addressing the low calorific value and understanding the implications of the cloud point could optimize its performance across various applications and environments.

5.5 IC ENGINE TEST

5.5.1 For 0% (Pure Diesel Engine Performance Analysis)

Table 5.9 Biodiesel Characterization (0%)

ID	2.56	5.67	6.3	9.33
BP	0	1	1.8	2.53
2MEH	6.8	17	28.1	26.9
2THE	147.1	97.7	83.4	84
2BTH	10	16.6	23.4	27
SFC	0.85	0.52	0.37	0.32

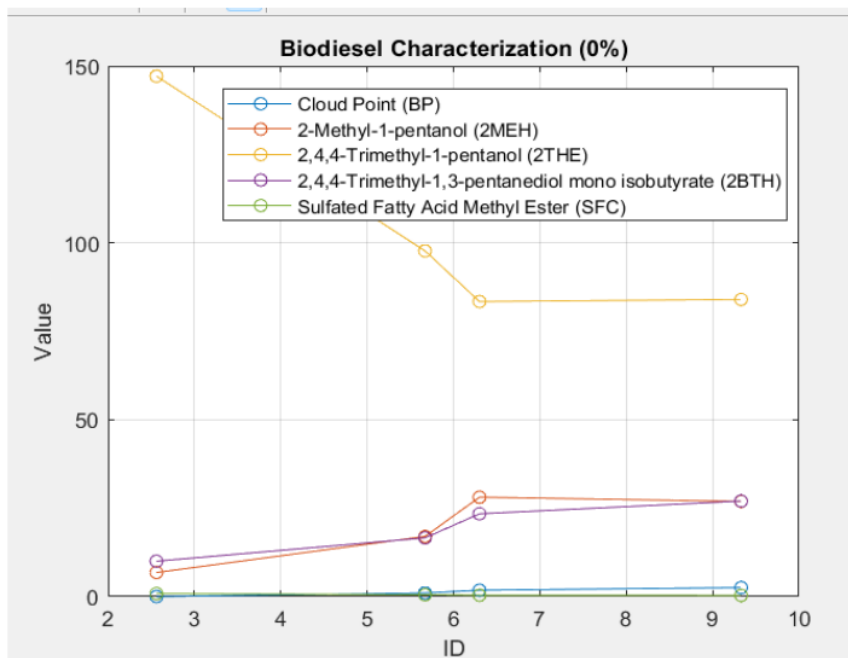


Fig 5.19 ID vs value graph for 0%

The provided data pertains to the characterization of a base fuel consisting of 0% biodiesel. Each parameter measured serves to delineate the properties and

composition of this base fuel. The sample identifiers (ID) uniquely denote each analysed sample, aiding in tracking and referencing. Cloud Point (BP) values denote the temperature at which solid crystals begin to form within the fuel, indicating its low-temperature operability. Lower cloud point values signify superior low-temperature performance, as the fuel remains liquid at lower temperatures. Parameters such as 2-Methyl-1-pentanol (2MEH), 2,4,4-Trimethyl-1-pentanol (2THE), and 2,4,4-Trimethyl-1,3-pentanediol mono isobutyrate (2BTH) likely represent concentrations of specific compounds present in the base fuel, reflecting characteristics stemming from the fuel's production process or inherent properties. Additionally, Sulfated Fatty Acid Methyl Ester (SFC) values serve as indicators of the base fuel's quality, particularly in terms of acidity. Lower SFC values typically correlate with higher fuel quality, as heightened acidity levels can induce engine corrosion and related issues. The graph visually depicts these parameters against the sample identifier, facilitating comparative analysis and identification of trends or anomalies within the base fuel's characterization data.

Table 5.10 Biodiesel Characterization (0%)

NAME OF THE ITEMS	LOAD			
	0	3	6	9
CO	0.20	0.16	0.11	0.11
HC	47	35	19	20
CO ₂	1.60	2.00	2.10	3.70
O ₂	18.33	17.88	17.67	15.52
NOX	13	39	155	425
SOME DENSITY	4.1	9.8	19.1	27.8

The graph illustrates the relationship between engine load and various emission parameters, as well as exhaust gas density. Each line on the graph corresponds to a specific parameter, namely Hydrocarbons (HC), Carbon Monoxide (CO), Carbon Dioxide (CO₂), Oxygen (O₂), Nitrogen Oxides (NO_x), and exhaust gas density. By

plotting these parameters against engine load, the graph offers valuable insights into how emissions and exhaust gas density fluctuate with changes in engine operation. For instance, variations in HC and CO levels may indicate incomplete combustion under certain load conditions, highlighting potential areas for combustion optimization. Similarly, changes in NO_x emissions can be observed, providing crucial information for emission control strategies. Moreover, tracking exhaust gas density alongside emission parameters offers additional context. Ultimately, this comprehensive visualization aids in understanding the intricate relationship between engine load, emissions, and combustion efficiency, facilitating the development of more environmentally friendly and efficient engine systems.

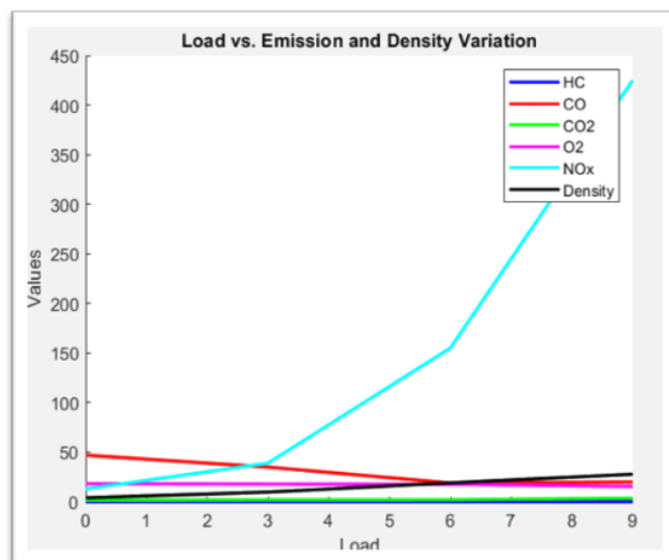


Fig 5.20 load vs Emission and Density Variation

5.5.2 The various parameters comparison between 20% and 40% load

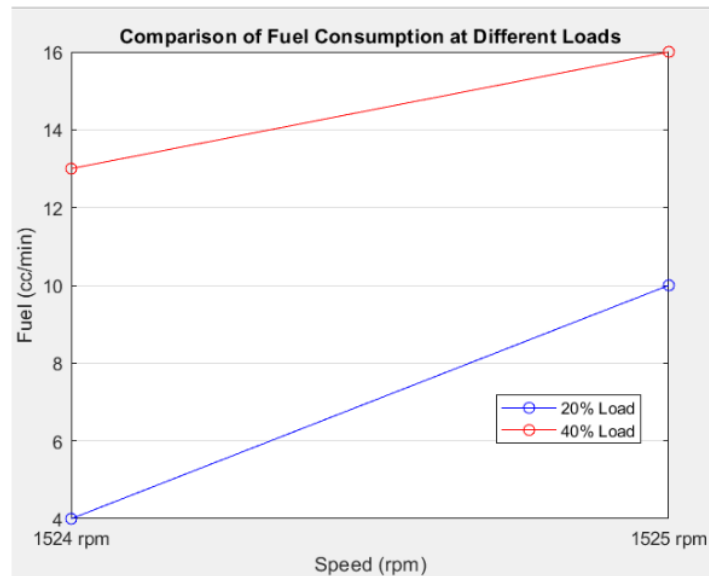


Fig 5.21 Comparison of Fuel consumption at Different Loads

The graph compares fuel consumption at two engine loads: 20% and 40%.

At both loads, as engine speed increases (moving right on the x-axis), fuel consumption generally increases.

This illustrates that higher engine speeds typically require more fuel to maintain power output, especially at higher loads.

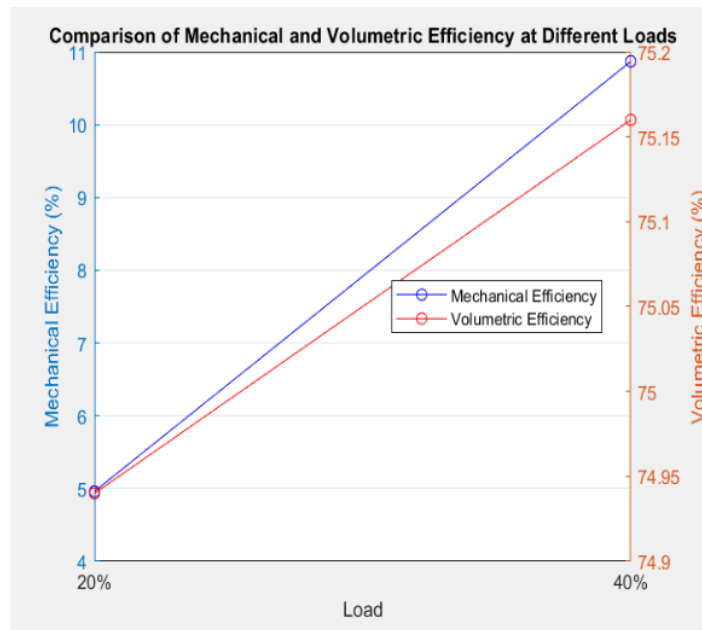


Fig 5.22 comparison of mechanical and volumetric efficiency at Different loads

This graph illustrates how mechanical and volumetric efficiency vary with engine load, specifically comparing 20% and 40% loads. Mechanical efficiency, representing the engine's ability to convert fuel energy into mechanical work, generally improves with higher loads due to optimized combustion. Volumetric efficiency, indicating the engine's air intake capability, also tends to increase at higher loads, reflecting improved breathing efficiency. Analyzing these efficiency metrics helps in understanding engine performance under different workloads, guiding optimization efforts for enhanced fuel economy and power output across various operating conditions.

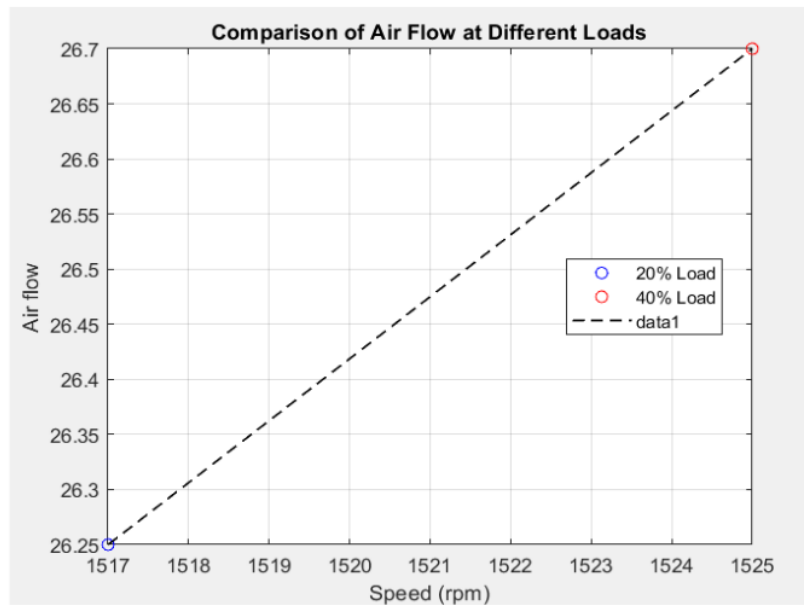


Fig 5.23 comparison of Air Flow at Different loads

This graph visually compares the air flow rates of an engine at 20% and 40% loads, providing valuable insights into its performance characteristics. Analyzing air flow at different loads is crucial for understanding how efficiently the engine draws in air for combustion under varying workloads. This comparison helps engineers identify potential inefficiencies or constraints in the engine's air intake system and guides optimization efforts to enhance overall performance. By visualizing the relationship between air flow and load, engineers can make informed decisions about engine tuning strategies to achieve desired performance outcomes, such as improved power delivery or fuel efficiency. Overall, this graph serves as a practical tool for evaluating engine performance and guiding optimization efforts to enhance efficiency and performance.

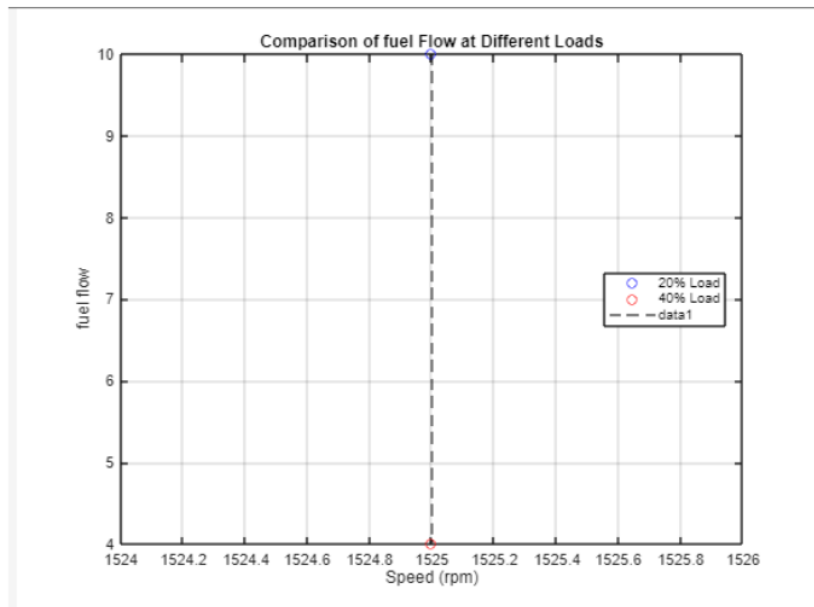


Fig 5.24 comparison of fuel Flow at Different loads

In internal combustion (IC) engine testing, the "load vs fuel flow" graph is a fundamental tool for understanding and optimizing engine performance. It's particularly crucial in dynamometer testing, where the engine is subjected to controlled loads to simulate real-world conditions without the need to operate the vehicle. Load in engine testing refers to the amount of resistance or work imposed on the engine by the dynamometer. This can be controlled by adjusting the dynamometer settings. Fuel flow indicates the rate at which fuel is being injected into the engine's cylinders. Fuel flow needs to be precisely controlled to maintain the desired air-fuel ratio for optimal combustion and engine performance.

In the context of an IC engine test, the "load vs fuel flow" graph visually represents how these parameters vary with changes in engine load. According to the graph load on the horizontal axis and both fuel flow on the vertical axes. As the load on the engine increases, typically both fuel flow rates also increase. This is because the engine requires more air and fuel to produce the additional power demanded by the load. The relationship between load and fuel flow rates may not be linear and can vary depending on factors such as engine design, operating conditions, and fuel type.

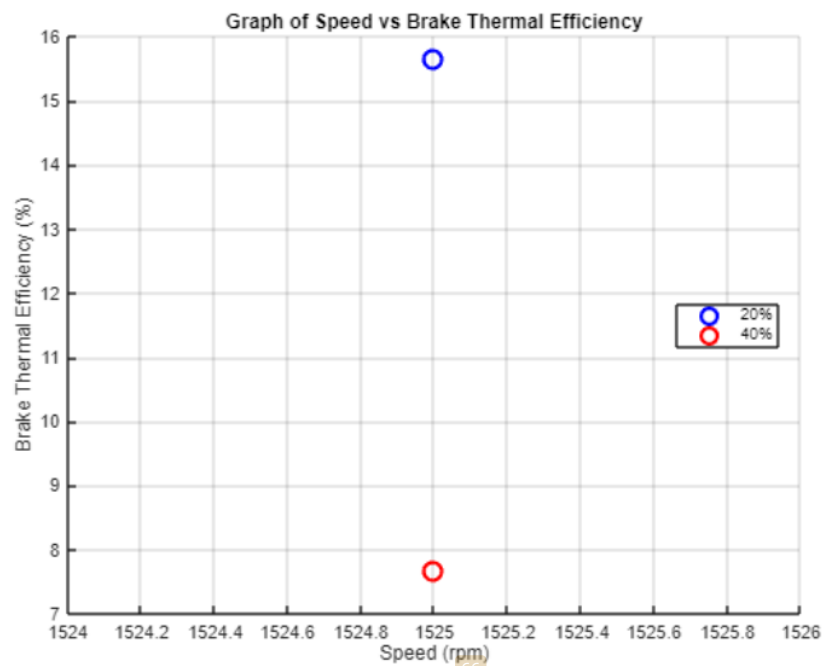


Fig 5.25 Graph of speed vs brake thermal efficiency

Speed vs. brake thermal efficiency refers to the relationship between the rotational speed of an engine and its brake thermal efficiency. Speed refers to the rotational speed of the engine's crankshaft, typically measured in revolutions per minute (rpm). It indicates how fast the engine is running. Brake thermal efficiency is a measure of how effectively an engine converts the chemical energy of fuel into mechanical energy at the output shaft. It is expressed as a percentage and represents the ratio of the useful work output to the total energy input from the fuel. Higher brake thermal efficiency indicates better fuel utilization and overall engine performance.

Typically, from the graph the relationship between speed and brake thermal efficiency may not be linear, and there may be an optimal speed range where the engine operates most efficiently.

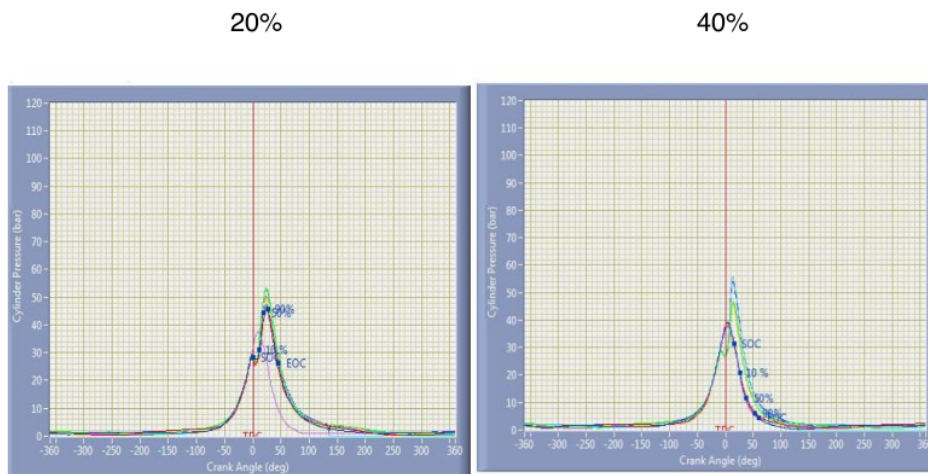


Fig 5.26 comparison of 20 and 40% load for the graph Crank angle vs Cylinder pressure

The graph illustrates how the pressure within the combustion chamber (cylinder) changes over the engine's cycle, typically against the crankshaft angle or piston position for 20% and 40%. The graph depicts the four strokes of the engine cycle: intake, compression, power, and exhaust. Each stroke is associated with characteristic pressure changes within the cylinder. During the intake stroke, the pressure within the cylinder decreases as the piston moves downward, creating a vacuum that allows the air-fuel mixture to enter the cylinder. In the compression stroke, the pressure within the cylinder rises sharply as the piston compresses the air-fuel mixture. This pressure increase prepares the mixture for efficient combustion. The power stroke is characterized by a rapid increase in pressure as the ignited air-fuel mixture expands, pushing the piston downward and producing mechanical work. The pressure reaches its peak during this stroke.

CHAPTER 6

SUMMARY AND CONCLUSION

SUMMARY

The study investigates the feasibility of producing biodiesel from non-edible CPSO using a TiO_2/ZnO catalytic converter in a photocatalyst reactor. CPSO's promising physio chemical properties, rich in 9(12)-octadecadienoic and n(hexadecenoic) acids, make it suitable for biodiesel production. Extensive characterization of the TiO_2/ZnO nano-composite was conducted, revealing its composition, structure, and properties. Successful glycerol transesterification resulted in biodiesel suitable for various applications. The study demonstrates the viability of using TiO_2/ZnO catalysts for sustainable biodiesel production through photo-transesterification.

Additionally, the study includes comprehensive analyses of various parameters related to internal combustion (IC) engine performance under different loads. Visual representations, including load vs. emission and density variation graphs, offer valuable insights into combustion efficiency and emission parameters. Comparative analyses at different engine loads elucidate fuel consumption, mechanical and volumetric efficiency, air and fuel flow rates, and speed vs. brake thermal efficiency, contributing to understanding engine performance characteristics.

CONCLUSION

In conclusion, the study confirms the feasibility of producing biodiesel from CPSO using TiO_2/ZnO catalysts via photo-transesterification, while also providing insights into IC engine performance under varying loads. Characterization techniques offer detailed insights into the composition and chemical structure of biodiesel, affirming its suitability as a fuel source. Further research endeavours may focus on optimizing engine conditions, enhancing fuel efficiency, and exploring advanced combustion technologies to improve internal combustion engine sustainability and performance.

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