Biodiesel Production using Waste Cooking Oil by Novel Heterogeneous Catalyst Synthesized from Waste Biomass

MS Thesis
by

Moeen Ali Rashad

CIIT/FA22-RNE-002/LHR

COMSATS University Islamabad
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by

Moeen Ali Rashad

CIIT/FA22-RNE-002/LHR

Department of Chemical Engineering

Faculty of Engineering

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Name	Registration Number
Moeen Ali Rashad	CIIT/FA22-RNE-002/LHR

Supervisory Committee

Supervisor	**Co-Supervisor**
Dr. Muhammad Haris Hamayun Assistant Professor Department of Chemical Engineering COMSATS University Islamabad (CUI) Lahore Campus	Dr. Murid Hussain Associate Professor Department of Chemical Engineering COMSATS University Islamabad (CUI) Lahore Campus

Certificate of Approval

This thesis titled

Biodiesel Production using Waste Cooking Oil by Novel Heterogeneous Catalyst Synthesized from Waste Biomass

By

Moeen Ali Rashad
CIIT/FA22-RNE-002/LHR

has been approved

for the Degree of MS in Energy and Environmental Engineering

at COMSATS University Islamabad, Lahore Campus

External Examiner: ______________________________

Supervisor: ______________________________

Dr. Muhammad Haris Hamayun

Department of Chemical Engineering

CUI, Lahore Campus

Head of Department: ______________________________

Dr. Muhammad Khaliq Majeed

Department of Chemical Engineering

CUI, Lahore Campus

Author's Declaration

I, Moeen Ali Rashad, CIIT/FA22-RNE-002/LHR, hereby declare that I have produced the work presented in this thesis, during the scheduled period of study. I also declare that I have not taken any material from any source except referred to wherever due to that amount of plagiarism is within an acceptable range. If a violation of HEC rules on research has occurred in this thesis, I shall be liable to punishable action under the plagiarism rules of HEC.

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Moeen Ali Rashad

CIIT/FA22-RNE-002/LHR

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It is certified that Moeen Ali Rashad, CIIT/FA22-RNE-002/LHR, has carried out all the work related to this thesis under my supervision at the Department of Chemical Engineering, COMSATS University Islamabad, Lahore Campus, and the work fulfills the requirements for the award of the degree of MS in Energy and Environmental Engineering.

Date: 29-05-2024

Supervisor:

Dr. Muhammad Haris Hamayun
Assistant Professor
Department of Chemical Engineering
COMSATS University Islamabad
Lahore Campus

Dedication

This thesis is dedicated to the most important people in my life – my parents. To my parents, I owe everything to you – your love, support, and encouragement have helped me succeed in my endeavors.

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I want to express my sincere gratitude to the Department of Chemical Engineering and Catalysis and Reaction Engineering Research Group for their support in the form of research experiences that have formed the backbone of this thesis. All the resources and the platform provided by the department have played a significant role in enhancing my performance.

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Moeen Ali Rashad

CIIT/FA22-RNE-002/LHR

Abstract

Biodiesel Production using Waste Cooking Oil by Novel Heterogeneous Catalyst Synthesized from Waste Biomass

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The growing need for renewable energy to conserve the environment has boosted research on biodiesel production from renewable resources. In this research work, the application of calcium oxide (CaO) obtained from the conversion of coconut shell (C-CCS) and coconut pith (C-CCP) as a catalyst in the transesterification process of waste cooking oil (WCO) to biodiesel is examined. The detailed catalyst characterization was analyzed using XRD, FTIR, and SEM, demonstrating that C-CCS showed higher crystallinity and well-defined carbon support structure than C-CCP. These catalysts were further examined under varying conditions of methanol-to-oil ratios, catalyst loadings, and reaction temperatures. The highest biodiesel yield of 92% was achieved was achieved using C-CCS at a methanol-to-oil ratio of 10:1, catalyst loading of 8 wt. %, and reaction temperature of 70 °C. Physical and chemical properties analyses showed that the produced biodiesel fulfilled the requirements of the set standard of ASTMD6751 with a density of 0 g/cc. The density ranged from 88 g/cc to 95 g/cc, applicable to the specificity of 4, the viscosity of 4.47 cSt at 40 °C while its flash point was 154 to 162 °C, the acid value was 0, saponification value of 49 mg NaOH/g oil and moisture content at 0.01%. C-CCS had comparatively superior properties to C-CCP because it has a higher surface area and pore size, which help enhancement in the catalytic activity. Besides focusing on biodiesel production with the help of biowaste-derived CaO catalysts, this study also underlines the possibility of utilizing waste materials as valuable resources for sustainable energy development. Directions for future research are also proposed in this dissertation. The research provides significant information on delivering a sustainable route map for biodiesel synthesis through cost-efficient and environmentally friendly catalysts.

Table of Contents

List of Figures

List of Tables

List of Abbreviations

ASTM	American Society for Testing and Materials
C-CCS	CaO Impregnated Waste Coconut Shell Catalyst
C-CCP	CaO Impregnated Waste Coconut Pith Catalyst
cSt	Centistokes
cm^{-1}	Wave Number in Infrared Spectroscopy
ECC	Eggshell Catalyst
EN	European Norms (used in reference to EN 14214)
FTIR	Fourier Transform Infrared Spectroscopy
GCMS	Gas Chromatography-Mass Spectrometry
g/cc	Grams per Cubic Centimeter
h	Hour
IR	Infrared
LCA	Life Cycle Assessment
min	Minute
NaOH	Sodium Hydroxide
SEM	Scanning Electron Microscopy
WCO	Waste Cooking Oil
XRD	X-ray Diffraction
°C	Degrees Celsius

Chapter 1

Introduction

1.1 Research Background

Biofuels stand out as an option in contrast to conventional fossil derivatives [1]. This increased interest primarily stems from global warming, increasing environmental pollution, and the depletion of fossil fuel reserves. Biodiesel, a notable biofuel, distinguishes itself due to its renewable and sustainable sources, non-toxic, and environmentally friendly properties [2]. The remarkable benefits of biodiesel are evident in its capacity to reduce carbon dioxide (CO_2) emissions and mitigate the release of harmful pollutants like particulate matter, aromatics, sulfur oxides (SO_x), and nitrogen oxides (NOx) [3]. Biodiesel utilization leads to considerable reductions, approximately 78% of net CO_2 emissions, 46.7% of CO emissions, 66.7% of particulate matter emissions, and 45.2% of unburned hydrocarbon emissions [4, 5].

Given the population growth rate, increased urbanization, and industrialization, the energy demand will keep soaring. Oil, coal, and renewable energy sources worldwide have also been progressing to accommodate this demand [6]. The primary fossil fuel, oil, is expected to remain the largest consumer in the global energy sector up to 2050. Renewable energy is identified as the fastest-growing energy resource, elicited in 2018, but is expected to rise to 114 EJ in 2040 and 161 EJ in 2050, as shown in Figure 1.1. While there have been increasing efforts to use renewable energy sources, fossil fuels primarily control the global energy reserve, and these are forecast to be depleted by 2050 if the current rate of depletion is to continue [2].

Conventional energy sources such as fossil fuels, which are most consumed in the energy sector, have impressive detrimental effects on climatic change by emitting carbon dioxide (CO_2) from inner combustion engines. The energy industry remains at the forefront of contributing nearly 35% of global CO_2 emissions. In 2018, global meat production and consumption stood at 3 billion metric tons and is expected to reach 43 billion metric tons [7]. Global energy consumption is expected to rise to 08 billion metric tons by 2050 to avert disastrous climate change. They include carbon dioxide, carbon monoxide, sulphur dioxide, and nitrogen oxides, which are recognized pollutants that adversely impact the environment and are perceived causes of global warming [8].

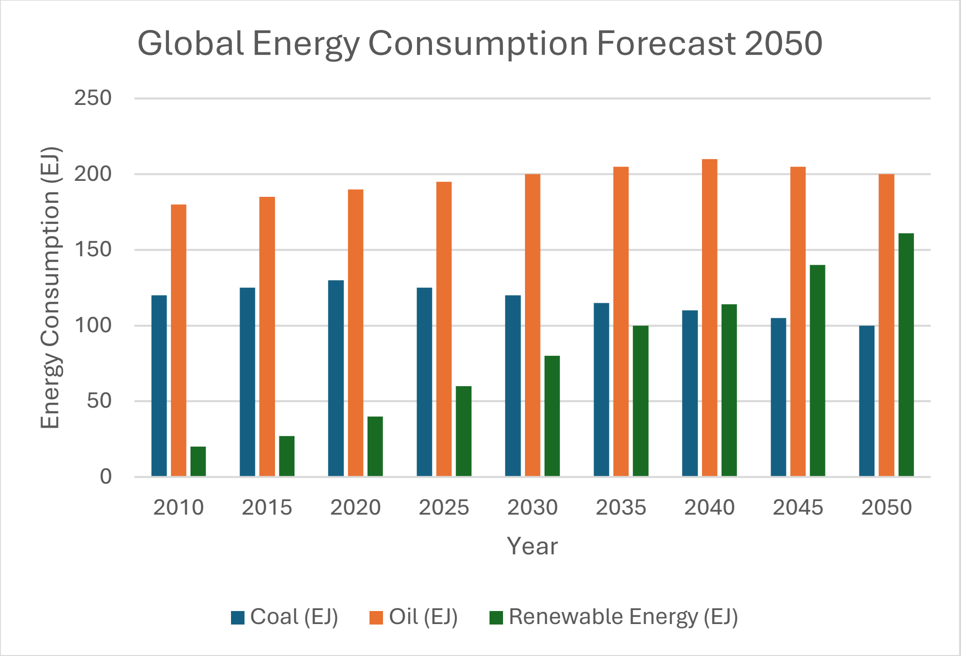

Figure 1.1 Global Energy Consumption Forecast 2050

As these challenges emerged, they have now played a vital role in developing and using renewable fuels. Among the renewable energy sources, biodiesel, bioethanol, and biogas have come under discussion as substitutes for ordinary non-renewable energy sources. Substantial human resources and capital investments have been committed to developing and utilizing these renewable fuels within the last few decades [9].

Biodiesel, or fatty acid methyl ester (FAME), is typically produced from feedstocks like edible and non-edible plant oils with or without a catalyst [10, 11]. Various feedstocks have been explored by researchers over time, including (1) first-generation edible oils from palm, sunflower, and soybean to (2) second-generation non-edible sources like neem oil, Jatropha curcas seed oil, castor oil, and waste cooking oil, too (3) third-generation biodiesel sources like microalgae oil [12]. Several biodiesel production methods exist: direct utilization, thermal cracking, pyrolysis oil dilution, micro-emulsion, and transesterification [13]. Among these methods, transesterification stands out as widely comprehensible and cost-effective [14, 15]. It involves a chemical reaction that requires fats or oils, alcohol, and a catalyst to yield FAME and glycerol.

Methanol is the preferred alcohol due to its cost-effectiveness, low molecular weight, and rapid reactivity [16]. Transesterification can be carried out using various types of catalysts. Homogeneous base catalysts bid distinct benefits with higher catalytic efficiency, shorter reaction times, and the availability of cost-effective raw materials [17].Conversely, homogeneous acid catalysts are utilized to deal with feedstocks with high free fatty acid (FFA) contents [18]. On the other hand, heterogeneous or solid catalysts have gained broader acceptance in biodiesel production due to their ease of separation, reusability, and superior corrosion resistance. While enzymatic transesterification holds promise for achieving high yields, it is constrained by its high cost and relatively slow reaction rate [19].

Table 1.1 Feedstock Types for Biodiesel Production with Potential Applications

Feedstock Type	**Benefits**	**Drawbacks**	**Examples**
First Generation	Straightforward process conversion available, abundantly	With food resources, low yield, limited cultivation area, less adaptability to harsh environments, competes	Oil, olive oil, sunflower oil, soybean oil, rice bran oil, mustard oil, palm oil, rapeseed oil, walnut oil, hazelnut oil, cashew nut oil, corn oil, radish oil, coconut
Second-generation	Production costs can be cultivated on non-arable land.	Resources, lower not interfere with food	Economically viable, low yield Jojoba
Third Generation	Food supply, rapid growth rate, and no competition with	Cultivation, expensive oil extraction, low lipid content for high energy requirement	Fish oil, poultry fat, chicken fat, Chlorella vulgaris algae, animal tallow
Fourth Generation	Growth rate, high energy output, efficient CO_2 absorption, limited	Cells, photobiological solar biodiesel	Biofuels, synthetic cells, photobiological solar biodiesel

	research, high initial investment, high lipid content, fast	electro, biofuels, synthetic

Using waste cooking oil as a feedstock and heterogeneous catalysts derived from biomass gives a promising approach [20, 21]. Solid biomass catalysts are both environmentally friendly and non-corrosive, helping to minimize effluent generation. Their predominantly biomass-based, readily available, and cost-effective composition improves their desirability [22]. The biodegradability of these catalysts also mitigates disposal concerns. This research centers on biodiesel production using heterogeneous catalysts derived from biomass. It will examine the catalysts' synthesis, preparation methods, performance characteristics, and comprehensive characterization. The primary objective is to provide valuable insights into biomass-derived heterogeneous catalysts, thereby contributing to future research in biodiesel processing while efficiently utilizing waste resources.

Biodiesel, one of the most potential renewable energy sources due to its biodegradable nature, has fewer carbon emissions and can reduce reliance on fossil fuels. A biofuel that exactly meets the definition of diesel fuel and for which the most common production method is the transesterification of cooking oils or animal fats with methanol or ethanol in the presence of a catalyst is biodiesel, and biodiesel production has input issues such as high cost of feedstock. To tackle these obstacles, WCO has been noted as a good candidate feedstock for biodiesel in the present study as it is cheap and eco-friendly. Nevertheless, achieving high biodiesel yields from WCO is only possible through effective catalysis since difficulties such as impurities and free fatty acids present in waste oils may extend the process [23].

1.2 Significance

1.2.1 Scientific Significance

The scientific importance of the current study lies in the fact that it has broadened the knowledge of different catalyst materials that can be used to produce sustainable energy. At the same time, it is concerned with the conservation of the environment. The use of waste biomass such as coconut shells, coconut pith, and waste eggshells to prepare heterogeneous catalysts for biodiesel synthesis is a recent advancement in the practice of biodiesel synthesis. This research will consider key questions on energy sustenance, waste biomass, and environmental depletion.

First and foremost, exploiting waste biomass in synthesizing catalysts is a key development as it closes different gaps in developing environmentally sustainable and cheap catalytic materials. Conventional biodiesel catalysts include alkalis and metal-sulfate-based compounds derived from sources such as fossils, which are scarce, expensive, and nonrenewable. In addition to contributing to developing an effective and cost-efficient solution for wastewater treatment, this study effectively repurposes waste coconut shells and pith to create activated carbon and incorporates calcium oxide obtained from waste eggshells to enhance the adsorption process. This approach aligns well with green chemistry and circular economy concepts by not wasting resources and disposing of unwanted byproducts.

Secondly, the contribution is made to the scientific knowledge of structural and surface characteristics of both new and modified catalysts based on waste biomass. Through studying the preparation of activated carbon from coconut shells and pith and the relationship between directly related factors such as surface area, porosity, and catalytic activity, this study can play an important role in the improvement of catalyst characteristics of the biodiesel production process. The findings in the synthesis and characterizations of these catalysts open avenues for other biomass-derived materials to develop a sustainable catalyst system.

Moreover, it responds to an important problem: the FFA content of WCO biodiesel has yet to be investigated. The biodiesel yields and fuel quality from WCO are negatively affected when high FFA content impedes the transesterification process. To apply WCO for biodiesel production, it is imperative that the heterogeneous catalyst sharpens reaction rates and lowers FFA content. This manuscript assesses the performance of the catalytic materials made in this experiment regarding MTO, the weight percentage of the catalyst, and the temperature at which the reaction is carried out. These could immensely benefit the growth of biodiesel production technologies since improvements could be made, making them versatile in terms of feedstock type.

Besides, due to the interdisciplinary coverage of this work, the findings are supported by the chemical engineering, material science, and environmental science fields, making this work more cohesive in its approach to solving global energy issues. Thus, the study contributes to increased scientific knowledge and cooperates with interdisciplinary solution approaches to provide sustainable energy systems. The findings can be used as a starting notion for subsequent research and development in the respective field and promote the usage of waste-based catalysts as a valuable resource on larger scales, thus supporting the worldwide shift to efficient and sustainable energy resources. Therefore, the scientific relevance of this research lies not only in advancing novel catalysts to enable biodiesel production but also in improving biodiesel manufacturing processes and sustainability for the biodiesel industry. This work contributed positively to renewable energy and the environment by using waste biomass to develop catalysts and augment catalytic properties.

1.2.2 Practical Significance

The synthesis of heterogeneous catalysts from waste biomass like coconut shells, coconut pith, and waste eggshells to overcome several realistic issues presents multiple advantages. As for its application, one of the most promising results of this research is the possibility of using waste cooking oil (WCO) for biodiesel production. Unfortunately, WCO is often thrown in the litter bin without proper disposal, causing environmental pollution and wasting natural resources. Besides, this research plays an important role in producing biodiesel from waste cooking oil and having the proper solution for utilizing waste oils.

In addition, using heterogeneous catalysts derived from biomass waste propositions performs even better than conventional heterogeneous catalysts. This is because conventional catalysts are relatively expensive; hence, the high cost may hinder the use of biodiesel. Since the research suggests using cheap and easily availed waste products such as coconut shells and eggshells, the cost of producing biodiesel is cut down. The economic advantage of biodiesel can positively influence the use of this energy source by making it more appealing than fossil energy in some sectors, such as the transport and manufacturing industries.

The two processes that involve using coconut shells and pith for synthesizing activated carbon and preparing calcium oxide (CaO) out of waste eggshells also present numerous social benefits in waste management. Coconut shells and pith are byproducts of coconut husk processing and are considered agricultural waste that can present disposal concerns. Likewise, the food industry necessitates that eggshells are best disposed of as waste or are merely left as waste by-products. Such conversion helps dispose of agricultural and food industry waste products but also converts these into valuable catalysts and adds economic value to waste materials. Moreover, the research considers technical issues related to the application of WCO in biodiesel production. WCO contains high concentrations of FFA, which could compete with methanol in the transesterification step and reduce biodiesel yields. This means that a more effective heterogeneous catalyst, which can lower the FFA content and, at the same time, increase the reaction rate, is important in practical biodiesel processing from WCO. This study reveals ways to enhance biodiesel yield and quality as well as the yielded catalyst by modifying the characteristics of the catalysts and the reaction conditions. The practical implications do not end here, as this research might have some optimal contributions toward environmental causes.

1.3 Thesis Organization

The present study comprises five chapters, which are as follows:

The first chapter gives a brief idea regarding the background and the aim of agenda-setting for the research. The core component is the research background section, which provides information about the necessity of developing sustainable energy sources and biodiesel as one of the options for mirroring global energy and ecological problems. The significance section explains the study's relevance in the scientific platform and its applicability to real-world problems touching on areas such as energy, waste, and the environment. Last of all, this section presents an introduction to the structure of the thesis to orient the reader towards the subsequent sections.

The second chapter is a literature review synthesizing the current literature on biodiesel production, catalyst development, variable metrics, and using waste materials with economic impact. Some areas considered include the type of raw materials used, the method used to produce biodiesel, known as transesterification, and the type of catalyst employed. Particular attention is taken to using waste cooking oil as the feedstock and production of heterogeneous catalysts from biomass. This review establishes the significance of the research problem and objectives by explaining how the present research can build upon the existing literature.

The third chapter provides information on the experiment's design and the methods used in the research. Materials and methods for producing activated carbon include waste cooking oil, coconut shells, coconut pith, and eggshells. In this study, the preparatory methods used in synthesizing the heterogenous catalysts involved the preparation of activated carbon and calcium oxide, and the preparation methods are outlined. The sections discussed in this chapter are the experimental procedures involved in the transesterification process together with the reaction parameters such as methanol to oil ratios, amount of catalyst used and reaction temperature of the process. Details on methods applied for characterizing the catalysts and for determining biodiesel yields, as well as quality, are well elucidated.

The fourth chapter is about results and discussion. It starts with the synthesized catalysts' physical and chemical characterization (FTIR, XRD, and SEM). The catalytic performance of the catalysts in the transesterification process is discussed in this study in terms of their efficiency in removing FFA and improving biodiesel yield. The findings are then discussed and compared with existing research to situate the research and showcase the offered contributions. The last section of this chapter outlines the biodiesel yield and the physicochemical characteristics of the optimized biodiesel produced.

The final chapter analyzes the conclusions of the conducted research and the potential practical application of the result. This section highlights the importance of research in enhancing sustainable energy generation from cost-effective catalysts. The chapter also discusses the limitations of the work, such as the size of the experiment or variation in the sample waste biomass. The suggestions for future research work are also given, and the prospects are explained, which indicate the potential for further investigation of catalysts based on waste materials and improvements in biodiesel production methods.

Chapter 2

Literature Review

2.1 Need for Alternative Feedstock

The receptiveness of global demand for energy and the necessity to mitigate greenhouse gas emissions provoked great interest in renewable energy sources. Compared to other fossil fuels, biodiesel is renewable, non-toxic, and can be biodegradable; thus, it is labelled as a promising fuel. Initially, biodiesel was made from waste cooking oils that were undergoing a transesterification stage. Nonetheless, some economic and environmental disadvantages are associated with cooking oils; thus, other sources, including the WCO, must be considered. Another hindrance common in the economic world is that cooking oils used for biodiesel production are expensive. The cooking oil prices are affected by various factors such as productivity in farming, the demand of the market, and the changes in the political system within the world market. For instance, the cost of the feedstock, such as soybean oil, palm oil, and rapeseed oil, which are usual feedstock for biodiesel, tends to vary in the international market [24]. These price variations can cut deep into the costs of producing biodiesel, making it generally unprofitable to venture into this line of production compared to fossil diesel product, which usually has stable price sets with doses of subsidy.

Also, cooking oils in biodiesel production directly threaten the food industry. This leads to a relationship between the biodiesel and cooking oil presumption: the higher the use of the former, the higher the use of the latter, which may cause an increase in food prices. Because the global population continues to fight for the availability of food and fuel, this only worsens it, especially in developing countries where many depend on affordable cooking oils. The food vs. fuel debate is one of the pressing economic factors that raises questions about the viability and ethical issues associated with utilizing cooking oils for biodiesel manufacturing [25].

It is equally impressive to note the environmental constraints that guard the use of waste cooking oils for biodiesel production. Growing oilseed crops on a commercial scale means that large quantities of land and water are used, with chemical inputs including fertilizers and pesticides. Increased cultivation of oilseeds also contributes to deforestation and destruction of the natural habitat in agricultural activities to expand produced agricultural land, such as unsustainable palm oil production contributed to the loss of tropical forests, resulting in the endangerment of several species and emissions of CO_2 due to changes in land-use systems. Additionally, oilseed crop farming involves intense agricultural practices that negatively impact the environment, such as destabilizing the topsoil, polluting water bodies, and even depleting freshwater sources. Feedstock cultivation's effects meaningfully necessitate waste cooking oil (WCO) as a feedstock in biodiesel production [26].

2.2 Homogeneous Catalysts

Homogeneous base catalysts have gained importance for industrial biodiesel production due to their delicate catalytic activity and good reaction kinetics [27]. It is crucial to recognize that the presence of FFA and water can significantly affect their efficacy. Additionally, issues with environmental sustainability and the potential for side reactions during saponification impede the extraction and purification processes, increasing the production of effluent and operating costs [28]. In cases involving feedstocks rich in FFA, such as pre-utilized cooking oil, non-edible plant oils, and animal fats, homogeneous acid catalysts like H_2SO_4, HCl, and H_3PO_4 have demonstrated more suitability [29, 30]. These acid catalysts resist FFA and water, expedite transesterification and esterification, and do not produce soap byproducts [31]. However, a significant drawback associated with them is their prolonged reaction times. Homogeneous acid catalysts are exceedingly acidic and reactive, which requires multiple phases for product purification in a homogenous system, resulting in considerable effluent production and increased operational costs, with the recycling process requiring supplementary processing stages [26].

2.3 Heterogeneous Catalysts

The use of heterogeneous catalysts in biodiesel production has been the focus of recent research. Unlike their homogeneous counterparts, heterogeneous catalysts occupy different physical phases to reactants and generally form active sites as they interact with reactants [24]. Heterogeneously catalyzed methanolysis is a three-phase system consisting of a solid heterogeneous catalyst and two immiscible liquid phases of methanol and oil [32, 33]. However, this form of catalysis demands higher temperatures and increased oil/alcohol ratios than homogeneous catalysis [34]. Heterogeneous catalyst recovery is streamlined, as they remain unaltered within the reaction mixture. Reusing these retrieved catalysts in succeeding procedures reduces the catalyst demand, minimizing costs [35]. Heterogeneous catalysis presents resistance to corrosion, ease of separation, and an extended catalyst lifespan [36].

Typical heterogeneous catalysts incorporating metals such as MgO, SrO, and CaO are the most reasonable for transesterification [37]. Readily available sources of calcium, like eggshells and bones, have been effectively harnessed for biodiesel production, contributing to waste reduction and cost-effectiveness [38, 39]. Catalysts for acid-catalyzed processes cover metal oxides, mixed oxides, and hydrotalcite. It is vital to recognize that a three-phase system can introduce diffusion challenges that could slow the reaction and reduce mass transfer efficiency [40]. Increasing the pore size and specific surface area is paramount in enhancing the efficiency of solid acid catalysts. At the same time, the hydrophobicity of external catalytic sites is also considered [41].

2.3.1 Heterogeneous Catalyst Derived from Waste Shells

The use of catalysts derived from organic waste materials, particularly those found in large quantities of calcium-rich substances such as animal bones and shells, has drawn interest [42, 43]. In calcination, calcium carbonate ($CaCO_3$) undergoes the transformation into calcium oxide (CaO), which acts as an exceptionally potent base catalyst in biodiesel production [44]. Traditionally, calcium carbonate extracted from limestone has been the primary source of CaO production. However, this method has considerable time and cost constraints [45]. Consequently, catalysts derived from organic waste have risen to prominence due to their non-toxic characteristics and ease of handling [29, 46]. The discarded waste shells, primarily comprising $CaCO_3$ (96-98%) with trace amounts of strontium carbonate ($SrCO_3$), magnesium carbonate ($MgCO_3$), calcium phosphate, organic matter, and water, are regarded as being of little value. They are often thrown into the waste [47]. The process of synthesizing catalysts from waste shells is cost-effective and not only contributes to waste reduction but also yields value-added products [7, 48]. A wide range of organic waste, from eggshells to animal bones, fish scales, and coral pieces, has been explored as prospective catalysts for biodiesel production [49, 50].

2.3.2 Activated Carbon as Catalyst Support

Activated carbon (AC) stands out due to its non-crystalline structure and porosity, sourced from carbon-rich materials like wood, agricultural residues, and waste biomass like coconut shells [51]. It supports catalysts applicable in systems addressing air and gas purification, wastewater treatment, and eliminating organic contaminants [25]. The considerable surface area of AC, resulting from its numerous pores, gives significant sites for the attachment of active metal particles. This plays a pivotal role in mass transfer and elevating the reaction rate in heterogeneous catalysis. Even though commercially available catalyst supports like alumina and silica have proven to be exorbitantly expensive, employing cost-effective AC for biodiesel production has emerged as a practical and economical alternative [6, 52]. Numerous investigations have ventured into using AC derived from biomass, often by impregnating specific active metals [23, 53]. Vinu Vadery et al. [54] demonstrated a significant increase in the catalytic activity of an AC-based catalyst through a chemical treatment involving a compound rich in potassium and calcium. In another study, Siow Hwa Teo et al. [55] identified that high concentrations of SiO2 and Fe_2O_3 serve as a cost-effective and highly efficient catalytic support compared to traditional options with feedstock from non-edible plants. These developments enhance the comprehensive efficiency of biodiesel production. Various ACs derived from biomass, including ashes from cocoa pods, coconut husks, and empty palm bunches, have been investigated for biodiesel production as solid base catalysts [56-58].

2.4 Synthesis of Heterogeneous Catalysts in Biodiesel Production

2.4.1 The Hydrothermal Method

The ability of hydrothermal synthesis to economically produce bulk metal catalysts with high purity and precise control over temperature and time parameters is well known [59]. Kassian T.T. Amesho et al. [60] investigated transforming crude Jatropha curcas oil into biodiesel using the hydrothermal method to generate calcium methoxide $Ca(OCH_3)_2$. The study examined the relationship between biodiesel yield and catalyst production factors like time, surface area, and morphology. After a fifth cycle under optimized experimental conditions, $Ca(OCH_3)_2$ displayed an impressive 86% FAME content and stood out as the most efficient heterogeneous catalyst for converting non-edible oils through transesterification. Maliyah et al. [61] employed a two-phase hydrothermal microwave-assisted approach for synthesizing mesoporous sulfonated TiO2 catalysts. Further analysis showed that the catalyst influenced a combination of Bronsted Lowry acid sites and favorable textural characteristics. Even after ten consecutive transesterification cycles, the catalyst showcased exceptional stability, requiring no additional mediations.

2.4.2 Carbonization Method

Rocha, Oliveira, and Franca [62] synthesized a solid base catalyst for biodiesel production by utilizing waste coffee grounds with KOH as an activator at 600 °C. The optimal transesterification conditions have a reaction temperature of 90 °C, a 3% catalyst weight loading, a 120-minute reaction time, and a 9:1 methanol-to-oil ratio, resulting in an impressive 91.57% biodiesel yield. The catalyst showed excellent reusability after regeneration. Fernández et al. [9] employed biochar derived from biomass through carbonization to be used as heterogeneous catalysts. Activated biochar achieved a 74% biodiesel conversion rate due to its increased specific surface area, remarkable catalytic effectiveness, and 93% conversion rate. Furthermore, Rathod et al. [63] developed an efficient catalyst from shrimp shells through partial carbonization with KF/CaO addition. The optimal conditions included carbonization at 450 °C, a 25% KF loading, and activation at 250 °C. This shrimp shell catalyst displayed 95% catalytic efficiency and environmental compatibility.

2.4.3 Calcination Method

Calcination breaks down into smaller components by heating them to high temperatures within an oxygen-free environment [64]. Depending on the biomass source, the precise calcination temperature, typically from 300 °C to 1000 °C, causes CO2 gas to be produced during the thermal treatment [65]. This temperature influences the surface morphology of the CaO catalyst, which primarily governs its surface area, thereby playing a pivotal role in determining the catalytic activity. Numerous studies have shown that calcination at lower temperatures, approximately 600 °C, yields uneven and aggregated structures due to the fusion of elemental components like Ca, Na, Mg, Si, and Sc [66-68]. In contrast, higher calcination temperatures, ranging from 700 °C to 900 °C, give rise to the formation of CaO with alterations in particle sizes [69]. Conversely, temperatures surpassing 950 °C disrupt catalytic activity by reducing pore volume and introducing micro-pores, diminishing the number of active sites on the catalyst's surface.

2.4.4 Wet Impregnation Method

For biodiesel production, Cholada Komintarachat and Sathaporn Chuepeng [64] investigated a novel solid heterogeneous catalyst (KCl/CaO) produced through a wet impregnation process. This catalyst underwent extensive testing for its efficiency in catalyzing the transesterification of used cooking oil. The researchers meticulously characterized the catalyst using FTIR, XRD, and SEM analysis. According to the results, the most favorable conditions are (1) a methanol-to-oil ratio of 12:1, (2) 3 wt. % catalyst loading, and (3) reaction time of 1.8 hours. Hanif et al. [65] introduced a heterogeneous catalyst prepared through wet impregnation of lithium onto TiO2. They designed a variety of Li/TiO2 catalysts with varying lithium concentrations (20%, 30%, and 40% by weight). Among these variations, the stimuli with 30% w/w lithium content, subjected to calcination at 600 °C, proved the most efficient, yielding an impressive Trans esterification rate of 98%. Table 2.1 below describes below the biodiesel production process using different kinds of feedstock through various synthesis techniques.

Table 2.1 Biodiesel Production Processes Using Various Waste Eggshells through Different Synthesis Methods

Material Source	**Synthesis Method**	**Feedstock**	**Production Process**	**Effect on Biodiesel Production**
Waste eggshell	Physical mixing	Waste cooking oil	The commercial calcium oxide (CaO) was blended with CaO derived from discarded eggshells, resulting in a hybrid catalyst.	Enhanced conversion efficiency Simplified catalyst separation - Reduced catalyst expenses

Waste eggshell	Physical mixing	Jatropha and Karanja oils	A slurry was formed by adding iodized water to a mixture of zinc oxide (ZnO) and CaO from waste eggshells, then drying in a furnace.	Achieved high conversion efficiency Improved quality and characteristics of biodiesel
Waste T. striatula shells	Physical mixing	Waste cooking oil	Barium (Ba) was homogeneously mixed with CaO derived from waste T. striatula shells, forming an effective catalyst blend.	Demonstrated high catalytic activity Enhanced conversion efficiency - Increased catalyst recyclability
Cocoa pod husks	Physical mixing	Soybean oil	Magnesium oxide (MgO) was incorporated with potash extracted from cocoa pod husks, creating a composite catalyst.	Increased biodiesel yields Enhanced product purity and integrity Improved engine performance of the biodiesel
Cuttlebone	Physical mixing	Waste palm olein oil	Strontium (Sr) was amalgamated with CaO obtained from cuttlebone, leading to the development of a novel catalyst.	Improved conversion efficiency - Enhanced surface area and thermal stability
Waste oyster shells	Calcination	Waste cooking oil	The waste oyster shell-derived CaO underwent	Reduced reaction time and temperature

			calcination at high temperatures, resulting in a more efficient catalyst.	Enhanced biodiesel yield and quality Lower activation energy
Waste chicken eggshell	Calcination and physical mixing	Waste cooking oil	The eggshell-derived CaO was combined with magnesium oxide (MgO) and subjected to high-temperature calcination, resulting in a robust catalyst.	Attained high biodiesel yield Achieved catalyst reusability for multiple cycles Reduced reaction time and temperature
Waste mussel shell	Calcination	Waste palm oil	The waste mussel shells were subjected to high-temperature calcination, producing a highly active CaO catalyst.	Increased CaO content and surface area Enhanced product yield, activity, and reusability
Waste cockleshell	Calcination	Waste palm oil	The waste cockleshells were calcined at elevated temperatures, resulting in a highly concentrated CaO catalyst.	Achieved higher CaO concentration and surface area Improved product yield and catalytic activity - Enhanced catalyst reusability

Waste scallop shell	Calcination	Waste palm oil	The waste scallop shells were subjected to high-temperature calcination, developing a potent CaO catalyst.	Enhanced CaO content and surface area Improved biodiesel yield and catalytic activity Increased catalyst recyclability
Coconut husk	Calcination	Jatropha oil	Coconut waste underwent calcination, resulting in the formation of a catalytically active material.	Achieved high biodiesel yield Reduced reaction time
Waste clamshell (Meretrix)	Calcination-hydration-dehydration	Refined palm olein cooking oil	The clamshells were subjected to a series of processes, including calcination, hydration, and dehydration, resulting in an efficient catalyst.	Formation of strong basic sites for enhanced catalytic activity Improved surface characteristics and conversion efficiency
Waste eggshells	Calcination-hydration-dehydration	Waste frying oil	The eggshells were subjected to calcination, hydration, and dehydration processes, forming	Increased catalytic activity and conversion efficiency Enhanced surface area and

			an active CaO catalyst.	basic strength - Improved reusability
Waste shells	Calcination-hydration-dehydration	Palm olein oil	The shells underwent a series of thermal and chemical treatments, forming an effective CaO catalyst.	Achieved improved FAME yield and fuel quality Demonstrated high catalyst reusability
M. lyrata shells	Calcination-hydration-dehydration	Crude Karanja oil	The shells were subjected to a series of treatments, developing a highly active catalyst.	Attained higher catalytic activity and surface area Demonstrated improved reusability and FAME yield
Waste animal bones	Wet Impregnation	Jatropha oil	Waste animal bone powder underwent calcination and impregnation, resulting in the formation of a potent catalyst.	Improved catalytic activity and FAME yield Enhanced catalyst reusability.
Waste chicken eggshell	Wet Impregnation	Palm oil off-grade	CaO derived from waste chicken eggshells was impregnated with magnetite (Fe_3O_4), forming a durable catalyst.	Achieved high catalytic activity and mechanical strength Demonstrated extended catalyst reusability

Waste eggshell	Bifunctional	Waste palm oil	The waste eggshells were subjected to a complex synthesis process involving calcination and blending, forming a multifunctional catalyst.	Attained high FAME yield Achieved improved surface characteristics and crystalline size Demonstrated high biodiesel yield
Waste Angel Wing Shells	Bifunctional	Palm fatty acid distillate	The waste angel wing shells underwent calcination and sulfation, resulting in the formation of a multifunctional catalyst.	Attained high biodiesel yield and selectivity Demonstrated increased surface area and pore volume Showed extended catalyst reusability.
Waste chicken eggshell	Co-precipitation	Waste frying oil	The waste chicken eggshell powder was co-precipitated with other compounds, forming an efficient catalyst.	- Achieved stability and high biodiesel yield - Demonstrated extended catalyst reusability

2.5 Different Feedstock for Biodiesel Production Using Heterogeneous Catalysts

The production of biodiesel relies on various feedstocks, and the selection of the feedstocks significantly influences the yield and overall quality of the final product guided by factors like availability, cost, quantity, and inherent physical and chemical characteristics [66]. Among the available options for biodiesel production, plant oils hold significant potential, both in the edible and non-edible categories [8, 12]. These oils are particularly suitable for biodiesel production because of their rich triglyceride content. Edible plant oils commonly used in biodiesel production include coconut, sunflower, soybean, rapeseed, palm, canola, peanut, olive, and mustard [8]. Due to their low FFA and water content levels, these edible feedstocks are primarily favored for first-generation biodiesel production. However, these feedstocks are generally more expensive to cultivate, and using them presents questions about the "food vs. fuel" trade-off.

2.5.1 Non-edible Oils

Non-edible plant oils are appealing alternatives for biodiesel production, primarily because they are unsuitable for human consumption due to their potentially harmful attributes [67]. These non-edible oils have enhanced combustion properties, reduced sulfur and aromatic content, and superior biodegradability. However, they often carry higher concentrations of free fatty acids (FFA), a factor that can vary based on the specific plant species and surrounding environmental conditions [68]. The list of non-edible oilseed crops comprises Jatropha curcas, castor bean seed, Karanja, rubber seed plant, Mahua, neem, tobacco seed, and rice bran [69]. According to a study by Remi et al., [70] biodiesel production using Jatropha oil, employing TiO2/CaO as the base catalyst and SO_4/TiO_2 as the acid catalyst, was carried out. These catalysts were used to lower the FFA concentration in oil with a three-wt. % catalyst, a 1:15 oil-to-methanol ratio, and a reaction duration of 90 minutes, the catalyst significantly reduced the FFA concentration within the oil from 1.22% to 0.59%. It yielded a production rate of 79.68% biodiesel.

2.5.2 Waste Cooking Oil

Shahabaldin Rezania et al. [71] investigated $LaPO_4$ as a heterogeneous catalyst for converting highly acidic waste cooking oil (WCO) into biodiesel and utilizing a 2.5 wt.

% $LaPO_4$ catalyst at a reaction temperature of 90 °C with a 5:1 methanol-to-oil molar ratio, the researchers synthesized 91% of FAME in just 120 minutes. In a distinct investigation, K. Cholapandian et al. [72] inspected the conversion of WCO to biodiesel using CaO. Various experiments were conducted on the organisms, and they used response surface methodology (RSM) to evaluate the impact of the various parameters. This has led to an outstanding biodiesel conversion of 97%. While using 9 concentrations of the catalyst, the percentage was 16. 6 wt%. According to their study, the catalyst can be reused for up to five cycles. This supports its use as a catalyst in the synthesis of biodiesel since it is economical and efficient. Hsiao et al. [73] described the reaction of WCO to biodiesel using a base-catalyzed method. For this, they studied various proportions and concentrations of the reactants to achieve the best biodiesel yield possible. The most effective ratio and concentration for using zinc-doped CaO as a catalyst in base-catalyzed WCO transesterification processes are 12:1. Work stress was reported significantly higher with 24% of participants indicating that it affected their work up to 50% while 5% asserted that their work efficacy was impacted by up to 1%. This also seeks to justify examining the quality and performance of the biodiesel product through the comparison of the density, flash point, and calorific value of the produced biodiesel and biodiesel derived from the processing of cooking oil residues.

2.6 Transesterification Process

The transesterification process plays a central role in the biodiesel process since it is a key step in the conversion of the triglycerides in the feedstock oils and fats to FAME and glycerol. It is essentially a saponification reaction or the reaction between oil or fats and an alcohol, mainly methanol, in the presence of catalysts. The outcome of this process rests greatly on few factors: the type of catalyst to be used, the ratio between the volume of alcohol and the volume of oil to be transesterified, the temperature at which the process is conducted, how long the transesterification process is to take and quality of feedstock [72].

In recent developments, the focus has shifted more towards utilizing heterogeneous catalysts such as CaO and AC because of some of the benefits associated with using these catalysts over homogeneous catalysts. Heterogeneous catalysts are more advantageous to the environment and are viewpoints as they can easily be separated from the reaction mixture and recycled. Hitherto, CaO obtained from waste materials, including eggshells, has been recommended for high basicity and catalytic properties. [11] have discussed a technique of using waste shells for the preparation of CaO as a catalyst for biodiesel manufacturing since it helped in reducing the costs of production as well as the waste. Calcium oxide also increases the conversion of triglycerides due to its high surface area and strong basic sites, thus making the process efficient at lower temperatures and pressures.

The applied CaO/CAC also shows promising results in enhancing the catalytic conversion efficiency due to AC's high surface area and porosity. The addition of CaO achieves alkali metal promotion, and AC promotes the dispersion of active sites while increasing the mass transfer of the reactants, increasing the reaction rate. [21] observed through their experiments that the implementation of the AC-supported CaO catalyst enhanced the efficiency of the transesterification process and biodiesel production significantly. The structure of the activated carbon enhances the accessibility of triglycerides and methanol to the active sites of CaO and provides more favorable conditions for reactions to occur.

2.6.1 Reaction Rates and Determination

The reaction between the triglyceride feedstock and the alcohol is known to influence the biodiesel production process, and thus, its rate is an important factor. It depends on the nature of the catalyst used, the volume ratio of methanol to oil, its temperature, and the volume of the catalyst used. Quantitative studies, as pointed out by [19] necessitate the determination of the concentration of reactants and products to establish the reaction's kinetics. These studies have indicated that the interest rate is proportional to the fraction of triglycerides in the reaction mixture, which is characteristic of heterogeneous catalysis, as depicted by CaO.

2.6.2 Effect of Molar Ratio and Other Parameters

There is also the concentration of alcohol in the reactor mass, which is an influential factor in transesterification. Theoretically, this is because a 1 molar ratio of methanol to triglycerides is adequate for the reaction to take place fully; amounts more than that are used to optimize the reaction towards completion. When more methanol is introduced in the reaction, the reaction moves toward producing methyl esters, achieving a higher reaction rate and yield. For instance, [60] reported that by increasing the molar ratio from 3:1 to 6:1, the enhancement in the biodiesel yield when employing CaO as the catalyst is significant and yields more than 90% as a product. However, beyond that optimal molar ratio, including more of the metal becomes less effective and can cause issues in extracting methanol from the product mix, which adds to costs.

2.6.3 Effect of Catalyst Loading

Another important aspect that weighs on the transesterification process is the amount of catalyst applied. This paper showed that having enough catalysts in the reaction ensures enough active sites for the reaction to occur, so there is a higher rate of producing biodiesel. Nonetheless, using larger amounts of catalyst may also create problems of mixing due to increased viscosity of reaction mass in the reaction vessel. The use of higher catalyst loading causes poor mixing of the reactants since the catalyst forms an external phase and reduces the active site's options, as pointed out by [63] for the CaO–AC catalyst where an optimum of about 5% (wt/wt of oil) gives the best result. However, high catalyst loading may lead to some complications in the catalyst's separation and recycling, which is not sound from the point of view of green chemistry [7].

2.6.4 Temperature and Reaction Time

The duration of the reaction, together with the temperature, also influences the efficiency of the transesterification process. In general, heat increases the reaction rate because it supplies activation energy for the reaction. Nevertheless, temperatures can be raised to take longer, leading to undesirable byproducts and wastage of energy, compromising the process's economy. Research carried out by [43] has identified that temperature and catalytic effect of CaO degradation favored an optimal temperature range of 60-70 °C for transesterification reactions. Other factors, such as reaction time, must also be well regulated so that the triglycerides are fully converted into the end product without excessive energy consumption. Longer reaction times result in secondary reactions that decrease the quality of the biodiesel produced; this is because the reactants have enough time to react further, producing undesired products.

2.6.5 Purity of Feedstock

In transesterification, feedstock quality plays a significant role since it determines the quality of the end products. Intrusion in the form of water and free fatty acids (FFA) in waste cooking oil (WCO) may influence the reaction by promoting saponification and lowering biodiesel yield. The pre-treatment process, like removing water content from the WCO and reducing FFA contents through acid esterification, is required to improve the fuel feed-stock quality and augment the transesterification efficiency. [69] observed that the WCO FFA content has to be pre-lowered to below 1%; this enhances the efficiency of CaO catalytic to yield better biodiesel production.

2.7 Kinetics of Biodiesel Production

The kinetics of biodiesel production are rather comprehensive, and it is a challenge to develop appropriate and comprehensive models for its description. This has made the kinetic experiments for biodiesel production few, with more and less concentration in both experimentation and simulation. Due to the presence of large molecular structures in the complex composition of biomass in biodiesel, previous studies have tried to model the best-known kinetics that characterize the overall rate of biodiesel production, involving the rate constants of the reactants transforming into diglycerides, monoglycerides, and the ester product. In the previous studies, the researchers worked on the kinetics of transesterification using base catalysts. These kinetic models are also synthesized selectively according to three consecutive reversible reactions wherein each step contains a different rate constant value established by [52].

Consequently, when the temperature of the reaction increases, the rate of the reaction also increases. Thus, it is crucial to maintain the reaction temperature below the boiling point of the alcohol used: These values of Tc were determined to be 65 °C for methanol and 78 °C. When the reaction is done at or above the boiling point of alcohol, one is forced to apply pressure on the reacting mixture to maintain the alcohol in its liquid. Among the methyl ester compounds, as per the kinetic analysis, the maximum number of carbon atoms was observed in the compound called methyl butanoate, which had the carbon atom chain of four carbon atoms connected with the methyl ester unit [13]. Like all the methyl esters of the above studies, methyl butanoate crank train performances were observed, and it cannot accurately simulate the crank train performance of biodiesel, which contains extensive chains of carbon molecules of 16-18. The number of reacting species is high under biodiesel containing several long hydrocarbon chains, and as such, the development of a kinetic model for biodiesel requires careful and detailed work.

The reaction of biodiesel from waste oil through a kinetic mechanism in the use of a carbon-based solid catalyst was investigated by [7] and noted that the reaction rate was second order. Additionally, [44] investigated the transesterification of tricaprylin (TCP) and methanol on a solid base heterogeneous catalyst, studying the reaction kinetics in the temperature range of 60-120 °C with a 15:1 ratio. Comparing the given molecular weight and the value obtained, the molar ratio of the precipitation reaction and the dissolving reaction is 1, 1 and respectively. [3] have investigated the reaction profile for biodiesel production from sunflower oil using the adequately dispersed CaO catalyst, where the reaction kinetics was found to be first order.

Some factors that elicit the non-universality of the kinetics are feedstock composition, the catalyst's efficiency, and reaction parameters, including temperature and pressure. Much attention is paid to the catalysts, particularly as it relates to factors influencing reaction rates and efficiency. For instance, when studying base-catalyzed reactions, the type and quantity of base influence the reaction rate and the demands of a catalyst.

Another factor that was found to be having a critical role was the temperature at which the chemical reaction occurs. One of the major benefits arises from the fact that the reaction temperature is maintained slightly below the solvent's boiling point; therefore, there is no need for pressure systems in the reaction. However, as was earlier noted, a combination of high temperature and low concentration of alcohol can slow down the rate of reaction, hence the strict control of conditions for the effectiveness of transesterification. Additionally, concerning the reaction rate of FAME in synthesizing biodiesel, the rates are not fixed for all fatty acids with methyl esters. The structure of these materials implies that they influence their reactivity as well as the rate of transesterification. Consequently, molecules present in biodiesel are much larger than methyl butanoate, and those larger types of molecules have more complex kinematics [3].

Kinetic studies also require more attention to establish the right kinetic models that will be viable in addressing the various feedstocks and catalysts to be used in the biodiesel production process. I also anticipate that enhancing the computational model and the experimental procedures will play a core role in accomplishing this work. Thus, it can be concluded that the kinetics of biodiesel production is indeed an extensive area with many approaches that can be taken into consideration when looking for ways how to improve the rate of biodiesel production. Nonetheless, biodiesel production still faces some factors and would need more research to emerge as a standard fuel in the global market. Improved and accurate kinetic parameters, ideal reaction conditions, and finding new catalytic systems will be critical for developing biodiesel production and further utilization of green energy [71].

2.8 Environmental and Economic Impacts

2.8.1 Sustainability and Waste Management

Using waste materials as feedstocks for biodiesel production also has environmental advantages in terms of sustainability and waste management benefits. This research work on biodiesel production using waste cooking oil (WCO), coconut shells, and eggshells is an excellent illustration of this circular economy, where waste products are reused. In the process, there is a reduction in pollution and the consumption of valuable commodities. This activity is advantageous in numerous ways in terms of its impact on the environment, and the effects of practicing the activities above and beyond this extreme are stunning in terms of waste, resources, and greenhouse gas saving.

Management of wastes remains a significant environmental nuisance, and the recycling of waste products for biodiesel production satisfies this vice appropriately. Used cooking oil, which is usually left to fen on its own after cooking, can adversely affect the environment in case of improper disposal. It also becomes a menace because it can block the sewage system, affect water bodies, and is implicated in the formation of soils. Through trans-esterification of WCO to biodiesel, they are, however, neutralized. The findings of [51] established that incorporating the use of WCO in biodiesel production helps in the management of waste oil through subsequent disposal while at the same time improving its value. Likewise, using coconut shells and piths as sources of activated carbon minimized the amount of agricultural residues that can cause undesirable effects such as generating pollutants and burning or decomposing where they release greenhouse gases.

Resource efficiency aims to optimize such resources in a manner that will less harm the environment and optimize the returns for resource inputs. A vision to create value-added products, namely activated carbon from coconut shell waste material and its application in the biodiesel industry. This gives purpose to gutters and minimizes the use of fresh raw materials. In a manner proceeding, eggshells, calcium carbonate-containing material, can be used to produce calcium oxide (CaO), an excellent catalyst for the transesterification process. [33] Synthesized the CaO from eggshells, which indicated that the material had high catalytic activity and could be used as a substitute for commercial catalysts.

Thus, biodiesel derived from waste received less carbon emission compared to both fossil fuels and biodiesel derived from waste cooking oils. A comparative analysis of the biodiesel life cycle from WCO proves that its production and use have a lower contribution to greenhouse emissions, as depicted by [36] Waste materials curtail the emission of methane and other greenhouse gases that would be emitted. Moreover, low-energy catalysts like CaO and activated carbon used in transesterification lower the temperature and pressure required for the process, thus cutting down the usage of energy and consequent emissions.

2.8.2 Circular Economy and Sustainability

It was integrated with the circular economy model, which avoids or reduces consumerism for products, recycling, and regeneration of materials. The utilization of waste products in the manufacture of biodiesel can be described as one such model in action. Apart from resolving waste disposal problems, utilizing and incorporating WCO and agricultural residues greatly offers biodiesel feedstock as a renewable source. This approach aligns with sustainable development aims, specifically those identified for furthering in the United Nations Sustainable Development Goals; this is in vision the twelfth goal aimed at promoting sustainable consumption and production patterns. To sum up, there are numerous advantages of using waste materials for biodiesel production in environmental concerns. This practice brings an element of sustainability to waste management, improves resource utilization, cuts the emission of greenhouse gases, and fosters the execute circle economy [31].

2.8.3 Economic Viability

The raw material costs of biodiesel production and, more specifically, the use of waste-derived catalysts are also essential factors in the feasibility of the process and its implementation. Some waste-derived catalysts that require categorization include calcium oxide obtained from eggshells and activated carbon derived from coconut shells; another benefit of the waste-derived catalyst in the economic sense is the low costs of raw material needed to produce the catalysts. Conventional catalysts in this type of process include sodium hydroxide and potassium hydroxide, which, despite being readily available in the market, are relatively expensive due to production and purification costs. While some raw materials, such as clay, can be readily obtained and have no problem with disposal, waste materials, such as eggshells and coconut shells, are easily available but are cumbersome to dispose of. Biodiesel production can benefit extensively from using waste as raw materials to prepare catalysts, drastically reducing expenses. According to [36] in their study, the cost of producing CaO from eggshells is significantly lower than the cost of commercial CaO, thus implying the possibility of using eggshells as a cheaper alternative in the production of CaO.

Another important aspect of the evaluation is the costs of processing and preparing catalysts from waste-derived feedstocks. As much as the collection, cleaning, and processing of waste materials present certain costs, they are also expensive compared to the costs incurred in producing conventional catalysts. The calcination of eggshells to get calcium oxide involves heating the shells to very high temperatures, which is a much-uncomplicated process and does not call for colossal expenses. Likewise, preparing activated carbon through the carbonization of coconut shells may involve thermal or chemical treatment, the latter of which can be accomplished through less costly and friendlier environment procedures [33].

2.8.4 Catalyst Performance and Longevity

Efficient catalysts increase the rates of reaction and biodiesel production, which, in turn, leads to shorter periods of production and lower expenses. In this regard, some studies employed waste derived CaO and activated carbon to do so. A recent study by [22] showed that the CaO – AC catalysts proved to be catalytically active and durable hence reducing the quantity of the catalyst to be used while at the same time increasing the length of its usability. This factor makes their reusability even more appealing to economically savvy chemists. Compared to heterogeneous catalysts, homogenous catalysts require neutralization. They are used up after the reaction process. At the same time, heterogeneous catalysts, such as CaO-AC, can be recycled several times, reducing the cost of a heterogeneous catalyst in the long run.

One more characteristic of the transesterification process that impacts its economic feasibility is the amount of energy needed for the process. These selective catalysts can work in relatively lower temperatures and pressures, which means a company can reduce the cost of energy. Some other types of CaO-AC catalysts, as claimed by Kansedo and others in their research, are that biodiesel with high yield can be made by employing moderate temperatures—about 60 to 70 °C, lower than those required by other catalysts in biodiesel production. This reduces energy consumption with operationally related costs and improves the environmental friendliness of processes and products with two-fold economic and environmental advantages.

2.8.5 Comparison with Traditional Catalysts

Several factors emerge into consideration when analyzing the cost differences brought by the utilization of waste-derived catalysts compared to normal catalysts. Conventional catalysts, on the other hand, do work initially, but they come with certain drawbacks based on their manufacturing, environmental usage, and removal. For instance, homogeneous catalysts must undergo neutralization and separation from the product formation, which adds costs in processing and environmental considerations. As for the heterogeneous catalysts derived from waste materials, the additional costs associated with using such catalysts can be eliminated, thereby enhancing their advantage. The fact that waste-derived catalysts are made from lower raw materials and the ability to separate and reuse waste catalysts make the whole process cheaper [31].

2.9 Gap Analysis

Although the literature review covered a wide range of heterogeneous catalysts for biodiesel production using various feedstocks, there exists a noticeable research gap concerning the specific use of eggshell sourced CaO as a catalyst in the transesterification of waste cooking oil into biodiesel. The literature review covers a variety of biomass-derived catalysts; however, it does not concentrate on activated carbon derived from waste coconut shells as a support material for CaO. This presents an opportunity for research to explore the creation of activated carbon from waste coconut shells and pith with its potential as catalyst support. Various studies have been reviewed with different catalyst systems with various active and support material combinations. Including CaO as metal oxide enhances the catalytic properties and efficiency of the catalyst systems, influencing the activated carbon characteristics such as surface area, porosity, and adsorption properties. The literature review has also outlined various biodiesel production feedstocks enclosing edible and non-edible waste cooking oil. This research will examine the challenges and prospects linked to waste cooking oil having high FFA content and how the proposed catalyst can effectively address these challenges. Although some studies have optimized reaction conditions for biodiesel production, further research is required to maximize biodiesel yield and quality by varying methanol-to-oil ratios, catalyst loadings, and reaction temperatures. The literature review also addressed catalyst stability but did not delve deeply into reusability. A critical research gap is determining the long-term stability and reusability of eggshell sourced CaO supported on activated carbon and the potential for regeneration.

2.10 Problem Statement

The growing issues of waste management and environmental degradation have highlighted the urgent need for sustainable waste resource utilization for energy production. It is essentially a solution to several interconnected challenges and opportunities. The development of biodiesel from waste materials like waste cooking oil, coconut shells, and eggshells will address the issues of waste disposal and, at the same time, implement the production of efficient and renewable energy. This research focuses on the major idea of not relying on fossil fuels, which is a step towards the effective ability to shift towards cleaner energy. This approach of using waste-derived feedstocks and waste biomass means that it's in line with the fight to curb carbon footprint and the negative effects of fossil fuels on the environment. The use of waste eggshell biomass as one of the materials incorporated in the catalyst demonstrates environmental stability. Waste materials such as coconut shells and pith, often disposed of, will be transformed into activated carbon that can offer a dual benefit: two broad functions: (1) to manage waste and (2) to serve as an efficient support for the catalyst. The use of non-edible feedstocks to produce biodiesel plays an ethical consideration in denying the need to use agricultural land for food production to meet the demands of fuel production.

2.11 Research Objectives

- To develop eco-friendly and cost-efficient catalysts from waste biomass, utilizing eggshells for calcium oxide supported by activated carbon.

- To investigate the production of activated carbon from waste coconut shells and pith, analyzing the catalyst’s structural and surface properties, surface area, and porosity.
- To evaluate catalytic efficiency in transesterification, focusing on properties and optimal combinations of methanol-to-oil ratios, catalyst loadings, and reaction temperatures, and assessing its role in reducing free fatty acid content, enhancing reaction rates, and improving biodiesel yield.

Chapter 3

Materials and Methods

3.1 Catalyst Preparation

Waste coconut shells, coconut pith, and eggshells are the primary materials used in catalyst preparation for this study. These materials were purchased locally; coconut shells and pith were purchased from a fruit seller in Lahore, and the eggshells from the COMSATS Lahore Cafeteria. Due to the availability of these waste products, they help cut down the cost of producing the catalysts and solve waste disposal problems since they are eco-friendly.

3.1.1 Producing CaO from Eggshells (Calcination)

Waste Eggshells were initially washed in distilled water to remove soil particles and other organic materials like membrane scrap and impurities. Sterilization is important to avoid cross-contamination during the process of calcination. The washed eggshells were then dried in a laboratory drying oven at a temperature of 110 °C for 12 h. This drying step ensures that no moisture is left before proceeding to the calcination process.

Once the eggshells were dry, they were crushed using a mortar and pestle to a fine consistency. Manual crushing, in this case, helps reduce the particle size, making it easier for the calcination to occur due to the increased area-to-volume ratio. The powdered eggshells were then subjected to sieve analysis, where a 100-micron sieve was used to collect a uniformly fine powder. The fine powder form enhances the efficiency of the calcination process, allowing for a more uniform conversion of calcium carbonate ($CaCO_3$) to calcium oxide (CaO).

The fine eggshell powder was then heated in a muffle furnace at 700 °C for 4 hours. This high-temperature treatment, known as calcination, decomposes the calcium carbonate in the eggshells into calcium oxide (CaO) and carbon dioxide (CO_2). The calcination process not only produces CaO, which is a highly effective catalyst, but also ensures the material is free from organic impurities and moisture. The resultant CaO, as presented in Figure 3.1, was collected and stored in a desiccator to prevent rehydration and contamination before use.

Figure 3.1 Calcined Eggshells at 600 °C after Sieve Analysis

3.1.2 Producing Activated Carbon from Waste Coconut Shells and Pith (Carbonization)

The coconut shells and pith were first thoroughly washed with distilled water to remove any surface impurities, such as dirt, oils, and organic matter, as presented in Figure 3.2. This step is important for ensuring that the raw materials do not have any contamination, which may compromise the subsequent steps.

Figure 3.2 Coconut Shells and Pith after Pre-Treatment

Samples were rinsed with distilled water to remove external contaminants, and then, through the process of drying, materials were left in an oven at 110 °C for 24 hours to minimize moisture content. One of the most important processes is the removal of moisture, as it helps increase the efficiency of the carbonization process and minimizes the chances of specific reactions while heating.

Coconut shells and pith were dried separately and then ground into a fine powder with the help of a ball mill. The ball mill serves the purpose of grinding the material to get a relatively smaller particle size and more surface area so that it increases reactivity and adsorption properties. After grinding the samples, the powders were analyzed by sieve analysis, where a 100-sieve mesh was used to obtain a fine and homogeneous sample. The pictures of the shell and piths are presented in Figure 3.3 and Figure 3.4, respectively.

Figure 3.3 Coconut Pith after Sieve Analysis at 100 Microns

Figure 3.4 Coconut Shells after Sieve Analysis at 100 Microns

The powdered coconut shells and pith used in this study were chemically pre-treated using 5M of potassium hydroxide (KOH) with a mechanical stirrer with KOH solution at room temperature for 3 hours, presented in Figure 3.5. This step aims to add potassium ions that are activators in the receptor type. These ions play the crucial role of opening pores within this carbon structure, thereby boosting the activated carbon's surface area and porosity. The higher porosity of the material will improve the efficiency of its work as an adsorbent and as catalyst support.

Figure 3.5 Mechanical Pre-treatment of C-CCS with 5M KOH

Following the pre-treatment, the KOH solution was further collected by vacuum filtration. This method effectively separates the solid phase from the liquid phase without needing additional tools or equipment. After the treatment, the powders were dried at a temperature of 90 °C for six hours to get rid of any moisture in the pre-treatment solution, which is presented in Figure 3.6.

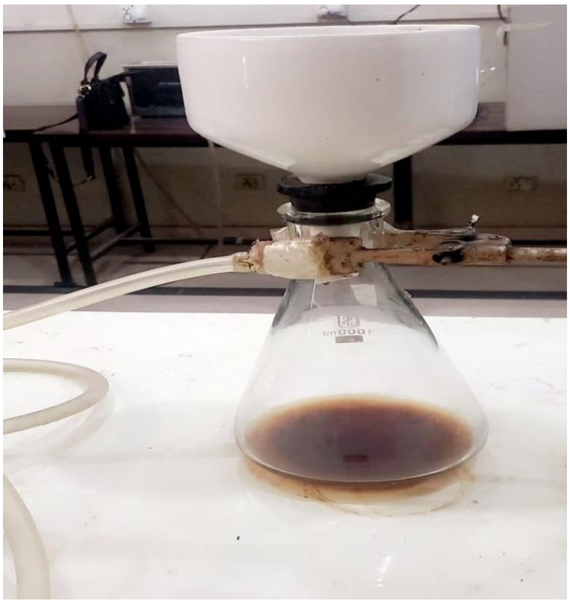

Figure 3.6 Vacuum Filtration of KOH Treated Coconut Shell Solution

The pre-treated powders were placed in a tube furnace for the carbonization process. Carbonization occurred at 700 °C for 2 hours under a nitrogen (N_2) atmosphere. The nitrogen flow rate was maintained at 70 ml/min to create an inert environment, preventing oxidation of the carbon during heating, presented in Figure 3.7. The high temperature facilitates the thermal decomposition of organic materials, converting them into a stable carbon structure. This step was performed separately for the coconut shells and pith to produce activated carbon from each source.

Figure 3.7 Carbonization of Coconut Shell and Pith carried out at 400 C

3.1.3 Wet Impregnation and Catalyst Synthesis (Calcination)

The activated carbon obtained from coconut shells and pith carbonization underwent a post-treatment process. This involved treating the activated carbon with 0.1N nitric acid. This acid treatment aims to remove residual alkaline impurities and introduce functional groups that enhance the material's adsorption properties. Nitric acid was then added to this treated carbon, and the two were stirred for one hour. After this, the samples were washed several times with distilled water to wash off any remaining acid and other unwanted materials, presented in Figure 3.8.

The second experimental procedure involved the wet impregnation method applied to the nitric acid-treated activated carbon. The CaO synthesized from the eggshells was added to the activated carbon during this process. The mixture was stirred for 2 hours to ensure that the CaO was dispersed evenly in the activated carbon and properly impregnated onto it. This method is very important in preparing the composited catalyst because it ensures that CaO is uniformly anchored on the surface of the activated carbon, thereby improving the catalytic capability of the activated carbon.

Figure 3.8 Calcination of Samples after Wet Impregnation of Cao with C-CCS and C-CCP

Once the impregnation process was complete, the resulting slurry was centrifugated at 5000 rpm for 15 minutes to facilitate segregation of the solid catalyst from the liquid phase. Sedimentation also aids in the effective accumulation of the solid phase; hence, after the process of centrifugation, they undergo other processes. The solid catalyst was then calcined at 6000 K for two hours to eliminate occluded vehemence. This final calcination helps constantly enhance the structure of the catalyst, its mechanical strength, and its catalytic character. The calcined product was marred by milling to fine particles to make the particle size uniform and labelled as C-CCS (CaO- impregnated Coconut Shell) and C-CCP (CaO- impregnated Coconut Pith) catalysts.

3.2 Catalyst Characterization

3.2.1 X-ray Diffraction (XRD)

This work uses XRD to identify synthesized catalysts' crystal structure and phase distribution. This technique includes illuminating the catalyst sample with the X-ray beams and determining the intensity and phase of the diffracting beam. These simple patterns give crystallographers rich information about the test sample's CRA structure.

3.2.2 Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR is employed to characterize the catalysts to know which functional groups and chemical bonds are contained in the catalysts. FTIR requests a point of IR light over the sample and a measurement of the extent to which frequencies of different wavelengths are absorbed, which correspond to various functional groups.

3.2.3 Scanning Electron Microscopy (SEM)

SEM analysis is performed to study the surface texture and employ particle size analysis instruments to determine the size of the catalysts. SEM is a type of microscopy that entails irradiating the sample surface with a fine beam of electrons and then detecting secondary electrons emitted from the sample surface to create an image of this surface.

3.3 Transesterification Process

3.3.1 Feedstock

The feedback selected in this research is waste cooking oil (WCO) collected from the COMSATS Lahore Cafeteria. This selection exhibits the use of an available material that is cheap and whose supply reduces the environmental problems associated with waste collection. WCO is obtained from several cooking processes that take place in the operation of the cafeteria, and the system is, therefore, exposed to various contaminants such as food residues, moisture, and FFA. However, the WCO has to go through several pre-treatment processes to make it fit for use in a transesterification process that is used to prepare biodiesel.

3.3.2 WCO Pre-treatment

Some of the necessary steps required to ensure efficient biodiesel production from WCO include the following: the WCO often contains some impurities that need to be separated, the FFA content of the WCO should be reduced, and moisture should be reduced. The primary objective of this step is to further purify the WCO to a level that is fit for use in transesterification, where FFA content is expected to be below 2%.

Because WCO is used in cooking, it undergoes the Maillard reaction, whereby moisture is adsorbed from food items, and the reaction that hydrolyzes triglycerides into FFAs takes place. Low temperatures reduce the oil's ability to flow and make it very acidic. FAA content and their moisture significantly affect biodiesel production, which contributes to soap formation during the transesterification process, leading to a low yield.

Based on the characteristics of the WCO to assess if it is fit for biodiesel production, some properties are quantified, including FFA content and AV. It is important to note that the AV and FFA content is determined through titration. The amount of WCO is dissolved in 10 ml of 2-propanol and titrated with the standard potassium hydroxide (KOH) solution containing 1.0 N of concentration with phenolphthalein as the endpoint indicator. The titration is repeated three times to increase the level of accuracy, the results are summed, and the average value is used further. The FFA content and AV are computed using the following equations:

$$\text{FFA (\%)} = (V \times M \times 56.1W)\ \text{FFA (\%)} = (\text{WV} \times \text{M} \times 56.1)$$

$$\text{AV (mgKOH/g)} = (V \times M \times 56.1W)\ \text{AV (mgKOH/g)} = (\text{WV} \times \text{M} \times 56.1)$$

Where:

VV = Volume of KOH solution used in titration (ml)

MM = Molarity of KOH solution

56.156.1 = Molecular weight of KOH

WW = Weight of oil sample (g)

Step 1: Filtration

The WCO is then filtered to conform to remove solid particles. This is achieved through the use of a fine mesh filter or, better still, the use of filter paper to remove any particles that may contaminate the oil and hence reduce the rate of chemical reactions in the next stages.

Step 2: Moisture Removal

The water quantity in the WCO is reduced by heating the oil before use. The oil is heated until its temperature is above 100 °C, with most temperatures rising to between 10 °C above the boiling point for aeration time of two hours. This ensures that all forms of moisture in the oil are eliminated from it through evaporation.

Step 3: Acid Value (AV) and FFA Content Determination

In order to determine the initial acid value and FFA content of the WCO, the following titration process is carried out:

- Sample Preparation: The filtered and dried sample of the WCO was weighed, and a quantity of 10 grams was taken in a clean and dried flask.

- Solvent Addition: Measured 2-propanol is then introduced into the flask containing the oil sample, and the volume added is 10 millilitres. The mixture is stirred until the oil is fully dissolved. Either at room temperature or with gentle heating, the mixture is blended until all the oil has melted into the mixture.

- Indicator Addition: Then, three droppers of phenolphthalein indicator solution are introduced to the titration mixture. While Kaolin was observed to undergo a physical change at a PH of about 8, Phenolphthalein alters color at this PH, showing the endpoint of the titration since there were no bubbles noted in the second sample that was added with the phenolphthalein.

- Titration: It is titrated with 0.1 M KOH solution. This is done by slowly dropping the NaOH solution and simultaneously stirring the mixtures. This is the endpoint, as it took 30 seconds for the mixture to turn to be non-pink in color.

- Volume Recording: The quantity of NaOH solution used to get to the endpoint is noted. This process is carried out three times to avoid ambiguity and confusion between the different impressions from the ones taken in the first stage.

The expression that is used to determine the AV is:

$$AV = \frac{(V_{\text{NaOH}} \times N_{\text{NaOH}} \times 56.1)}{W_{\text{sample}}}$$

A 10 g sample of waste cooking oil, with approximately 0.0473 mL of a 0.1M KOH solution, will reach the endpoint of the titration and achieve an Acid Value (AV) of 2.65 mg KOH/g.

To convert the acid value to FFA percentage:

$$FFA(\%) = \frac{2.65\ \text{mgNaOH/g}}{1.99} = \mathbf{1.33}\%\%$$

Therefore, the determination of the FFA content of waste cooking oil ranges from about 1. 33%. This is within the acceptable level of FFA content when it comes to biodiesel production through the trans-esterification process, which allows up to 2% FFA content.

3.3.3 Experimental Setup

The transesterification was conducted to yield biodiesel from waste cooking oil (WCO). The setup included a system used for refluxation, allowing fine-tuning of the reaction atmosphere. They included a 500 ml three-neck round bass concurrently equipped with a gas inlet tube, a condenser, and a thermometer. A water bath was used to regulate the reaction temperature. For the preparation of a suitable homogeneous solution in the flask, an overhead stirrer was provided, and the stirring speed was maintained constantly at 600 rpm. This also includes a condenser, which helps condense any vapour and return it to the reaction mixture, hence avoiding losing the reactants. Each experiment was carefully designed to vary one of the key parameters: Methanol to oil ratio, use of catalyst, and the reaction temperature. This systematic procedure used for the experiment provided a platform for assessing the impact of each parameter on the yield of the biodiesel.

The factors under consideration were:

- Methanol to oil ratio: 8:1; 10:1
- Catalyst loading: 6 wt.% and 8 wt.%

- Reaction temperature: 60 °C and 65 °C.

These factors were selected concerning pre-studies and reports within literature reviews, showing their strong influence on transesterification. The corresponding representation is provided in Figure 3.9.

Figure 3.9 Separating Funnel Separation of Optimized Biodiesel Yielding 92%

3.4 Process Optimization

The influence of the reaction parameters in the transesterification process becomes vital when pursuing the enhancement in yield and quality of biodiesel. This includes an orderly manner to test out factors such as the proportion of methanol to oil, the amount of the catalyst required, and the reaction temperature. With these factors under control, one can study the impact that each has on the transesterification process and the interaction between parameters.

3.4.1 Varying Methanol to Oil Ratio

Methanol and oil are two significant components in the transesterification process, and the reaction rate constant is directly proportionality to the methanol to oil ratio. To investigate the effect of varying methanol to oil ratios on biodiesel yield, a series of experiments were conducted using methanol to oil ratios of 8:1 and 10:1 using catalysts, C-CCS and C-CCP.

In each of the experiments, as mentioned above, 5 grams of filtered and dried waste cooking oil (WCO) was added to a three-neck flask. Methanol was added to the flask in the specified ratios of 8:1. Next, making sure that the range of methanol was different for each trial, we obtained the percentages of 10:1. As for a choice of a certain type of catalyst, it was C-CCS or C-CCP, they were added to the reaction mixture in quantity prepared. This product was kept in a water bath for the desired temperature of 60 °C and 65 °C and then stirred well for the reaction. The layering of the two phases was then done by separating the funnel; this was after measuring the reaction's completion and waiting for the reaction mixture to cool down. At last, the biodiesel accumulated in the upper layer container was accurately measured to assess the experimental yield.

3.4.2 Varying Catalyst Loading

Another important factor that can impact transesterification is the use of catalysts and the amount of them used. A catalyst helps in the reaction process; its concentration decides the rate of reaction taking place and the overall conversion efficiency. To investigate the influence of the catalyst concentration, experiments were done with C-CCS at wt.% and 8 wt. % and C-CCP at 6 wt.% and 8 wt.%. These experiments involved using WCO; therefore, 5 grams of the WCO that had been filtered and dried were put in the three-necked flask. Methanol was then added in a consistent ratio. The catalyst was supported by adding them in set loadings of 6 wt% and 8 wt.%. To set the desired temperature of the reaction mixture easily, the flask was heated using hot plates to 60 °C or 65 °C, depending on the reaction, and stirred for homogeneity. In the end, as far as the reaction process was concerned, the solution was cooled, and the various layers were separated.

3.4.3 Varying Reaction Temperature

The temperature of the reaction plays an essential role in the determination of the rate constants of the ester exchange. Higher temperatures favor increases in the rate of the reaction, although they have some adverse effects on the production of undesired products. The catalysts were further tested at 60 °C and 65 °C using both C-CCS and C-CCP to determine the impact of the reaction temperatures. In these experiments, 5 grams of the WCO were weighed and transferred to the three-necked flask. As per the requirements set by the experiment, the reaction mixture was then heated at 60 °C or 65 °C, with vigorous stirring to provide homogeneity. The mixture was then left to cool, and the two layers were separated. The formed biodiesel was also identified in the top layer, and its yield was quantified.

3.4.4 Experimental Procedure

In these experiments, 5 grams of filtered and dried waste cooking oil (WCO) was weighed on a Caesarbi Analytical Balance Model 50-600 and placed into a three-necked flask. Methanol was added in specified ratios. The catalytic amount was kept constant depending on the experimental plan. The reaction mixture was then heated to 60 and 65 °C, and aliquots were stirred evenly throughout the reaction. When the reaction has occurred or taken its full course, the mixture is also allowed to cool, and the layer separation is made using a centrifuge when needed. After this experiment, the biodiesel was left in the separating funnel to be separated from other components of the mixture. Cordia was taken at the top layer of the conical flask, and its weight was weighed to determine its yield. These steps helped sustain the control of the experiments' conditions to find out biodiesel yield variation by methanol to oil ratio, catalyst, and temperature.

Table 3.1 Biodiesel Production Process Optimization

Experiment	**Catalyst Type**	**Methanol to Oil Ratio**	**Catalyst Loading (wt %)**	**Reaction Temperature (°C)**
1	C-CCS	8:01	6	60
2	C-CCS	8:01	6	65
3	C-CCS	8:01	8	60
4	C-CCS	8:01	8	65
5	C-CCS	10:01	6	60
6	C-CCS	10:01	6	65
7	C-CCS	10:01	8	60
8	C-CCS	10:01	8	65
9	C-CCP	8:01	6	60
10	C-CCP	8:01	6	65
11	C-CCP	8:01	8	60
12	C-CCP	8:01	8	65
13	C-CCP	10:01	6	60
14	C-CCP	10:01	6	65
15	C-CCP	10:01	8	60
16	C-CCP	10:01	8	65

Chapter 4

Results and Discussion

4.1 Catalyst Characterization

4.1.1 XRD Analysis

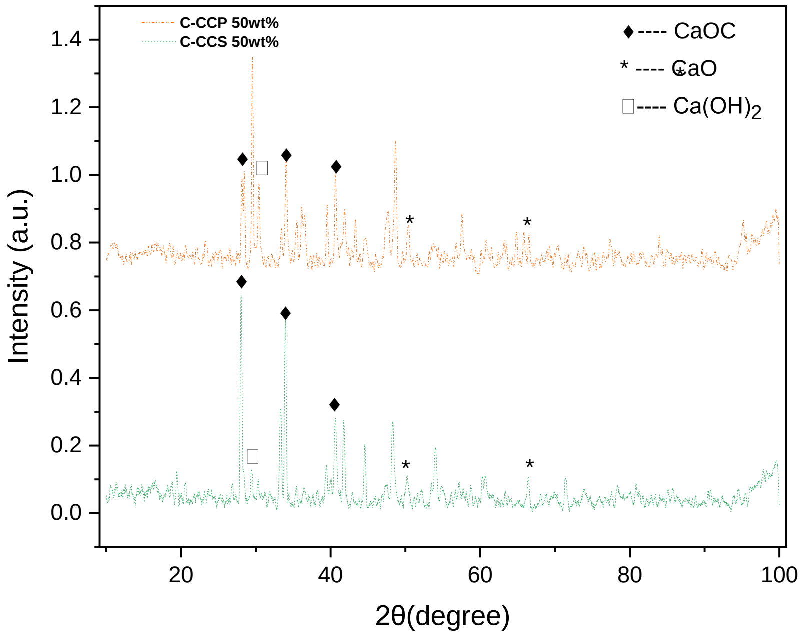

Figure 4.1 XRD Analysis of C-CCS and C-CCP Catalysts:

X-ray diffraction (XRD) X-ray diffraction analysis was used to reveal key structural properties of the samples of the prepared catalysts from coconut shell (C-CCS) and coconut pith (C-CCP). In the patterns of the samples, as shown in Figure 4.1, some peaks could be associated with characteristics of calcium oxide (CaO) for both samples and activated carbon for the second sample, which showed noticeable differences in the intensity and sharpness of the peaks. The peak in XRD patterns of both the activated samples corresponds to crystalline phases of CaO and activated carbon. However, the degree of output augmentation and the peak's steepness vary significantly. The XRD pattern of C-CCP is depicted in a red line for comparison with that of C-CCS, shown in the red line.

The major peaks of the activated carbon analyzed with an X-ray diffractometer are presented at 2θ ≈ 26. In the literature, the diffraction peak located at 2θ = 5° is indexed to the (hkl) plane of the diffraction pattern, identified with the interlayer distance of graphitic carbon d=17.7 Å°, indicating that the modified host H1 has retained the graphitic carbon structure. In contrast, the host H2 shows a more ordered structure with the interlayer distance of d = 21.0 Å3°, following the (100) plane of graphitic carbon. The CaOC peaks were observed at 2θ ≈ 36. The crystal data indicated that the Bragg angle of the (111) plane is 2° for the sample and 2θ ≈ 36 Δθ for the 4° in the (200) plane, 2θ ≈ 53. It then calculates 8° for the (220) plane and 2θ ≈ 64. When using the (311) plane, the angle 2θ equals 2°, and when using the corresponding angle ≈ 67°. In the case of macromolecules, low-angle X-ray diffraction patterns are observed for the (222) plane.

In the present study, for the C-CCP sample (red line), it was observed that the XRD pattern showed high noise and less peak intensity compared to C-CCS. This indicates that C-CCP may be less crystalline in composition, structure, or formation. The appearance of a large hump or broad peak near 26 evidenced the existence of activated carbon. 5° and a slighter peak at approximately 43°. Kronauer Stages III and IV positions at 2θ ≈ 36° were attributed to the formation of CaO. 2°, 37. 4°, 53. 8°, and 64. 2°, which suggests the existence of crystalline CaO, though the diffraction peaks' intensity appeared smaller than in.

However, compared with P-CCS, the XRD pattern of the C-CCS sample (black line) demonstrated sharper and more distinguishable diffraction peaks due to the higher crystallinity of the latter. In the activated carbon, the main diffraction peak appears at the 2θ ≈ 26. 5° and 43. 3° were more distinct, indicating a highly ordered or developed structure for graphitic carbon in the coconut shell-derived carbon. Besides, the CaO peaks have been observed approximately at 2θ ≈ 36, which can be attributed to the presence of this oxide in the material after heat treatment. 2°, 37°, 53.8°, 64.2°, and 67. 3° were more intense and this might be due to a better crystallinity of this sample or to the presence of a higher amount of CaO in the sample.

Comparing the two samples, in the analysis of the samples by XRD, it can be seen that C-CCS, derived from coconut shell, has more crystalline CaO than C-CCP, derived from coconut pith. This would imply that the C-CCS sample has either a more crystalline CaO phase or is composed of bigger crystallites and better dispersion than the reference sample. These results reveal the amorphous nature of the materials present in the C-CCP sample and confirm that the noise intensity is high compared with the C-CCS sample. Similarly, each sample possesses a typical activated carbon pattern, yet these patterns have sharper and more intense builds in the C-CCS sample, likely due to lower noise levels.

The prepared CaO/activated carbon derived from the coconut shell (C-CCS) shows enhanced peak intensity and higher peak sharpness, suggesting that the CaO sample may possess higher crystallinity or better dispersion of its particles. The peaks corresponding to activated carbon are also sharp and clear, indicating a highly ordered and developed graphitic structure. On the other hand, the CaO/activated carbon derived from coconut pith (C-CCP) The XRD pattern exhibits less crystallinity and considerably weaker diffraction peaks, suggesting a more amorphous nature of CaO or smaller particle size of CaO. There is less contrast to the structure of C-CCP's activated carbon than C-CCS' carbon pellets. As seen from this analysis of the data, it can be stated that even though both the catalysts comprise similar features, the XRD patterns and possibly the distribution of CaO on AC are dissimilar between the C-CCS and that of the C-H2O samples in which C-CCS display a relatively higher crystallinity, which can be associated with the more well-defined carbon support.

4.1.2 FTIR Analysis

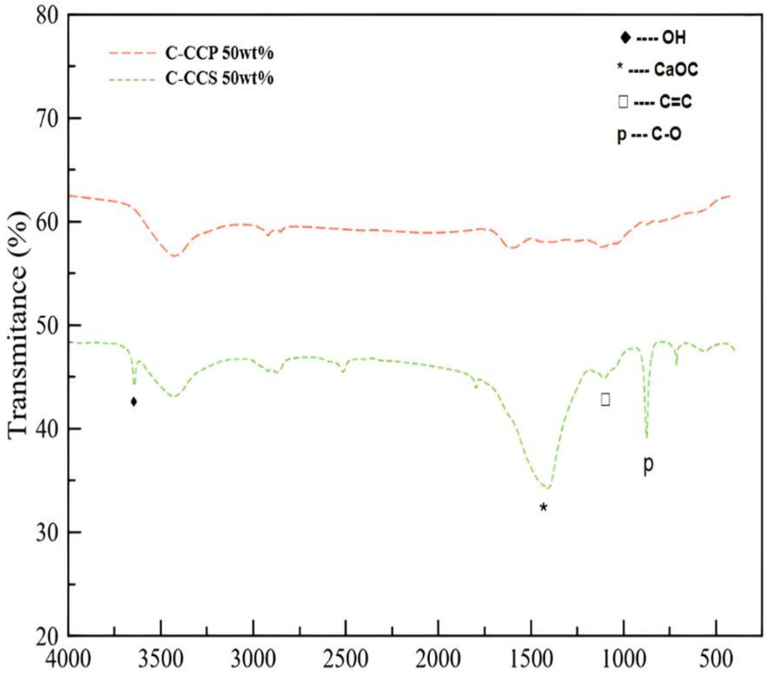

Figure 4.2 FTIR Analysis of Catalysts; C-CCS and C-CCP

FTIR (Fourier Transform Infrared Spectroscopy) analysis provided detailed insights into the structural properties of the biochar, its activated form, and the final optimized catalyst, 20Ca/AC-650. Figure 4.2 illustrates the FTIR spectra of these samples, revealing significant functional groups and chemical bonds on the catalyst surfaces.

The C-CCP (red spectrum) and C-CCS (green spectrum) samples exhibit several peaks indicative of various functional groups. The spectra reveal a complex pattern of absorption bands, which is characteristic of activated carbon materials due to the presence of multiple surface functional groups. A broad peak around 3450 cm^{-1} is observed in all characterized samples, indicating the presence of -OH groups on the catalyst surface. These hydroxyl groups could be attributed to the surface of the activated carbon or adsorbed water. Sharp peaks near 1470 cm^{-1}, 873 cm^{-1}, and 714 cm^{-1} are associated with asymmetric stretching vibrations that signify the presence of $CaCO_3$. This is likely due to the incomplete carbonization of CaO, indicating that some $CaCO_3$ remained in the catalyst structure. A peak at 3644 cm^{-1} is also attributed to the structural hydroxyl groups in $Ca(OH)_2$, as revealed by literature.

Broad peaks around 3300-3500 cm^{-1} suggest O-H stretching vibrations, indicating hydroxyl groups or adsorbed water on the activated carbon. Peaks around 2850-2950 cm^{-1} correspond to C-H stretching vibrations, indicative of aliphatic hydrocarbon chains. Sharp peaks near 1600 cm^{-1} are likely due to C=C stretching vibrations, typical in aromatic rings, indicating the presence of aromatic hydrocarbon molecules in the biochar. Peaks around 1000-1300 cm^{-1} are associated with C-O stretching vibrations in alcohols, ethers, or carboxylic acids.

The observed functional groups and their corresponding peaks align well with known activated carbon and calcium oxide (CaO) characteristics. For activated carbon, broad O-H stretching bands around 3300-3500 cm^{-1} are due to surface hydroxyl groups. C-H stretching bands in the range of 2850-2950 cm^{-1} are common, indicating the presence of aliphatic groups. Aromatic C=C stretching vibrations near 1600 cm^{-1} are expected due to the aromatic nature of carbon structures. Calcium oxide (CaO) typically does not show strong IR absorption because it is ionic and IR inactive in the region where organic functional groups absorb. However, interactions with the carbon surface might alter the spectral profile slightly.

O-H, C-H, and C=C stretching vibrations align well with the expected surface functional groups on activated carbon. The comparison between C-CCP and C-CCS shows that both spectra are similar but not identical, suggesting that the precursor material (shell vs. pith) influences the surface chemistry of the activated carbon. The FTIR spectra obtained are consistent with the expected functional groups on activated carbon. The presence of these functional groups is critical as they can influence the catalytic properties of the material. The peaks identified in the spectrum are consistent with other studies of activated carbon and suggest that the synthesis procedure probably yielded the desired material.

Additionally, using FFT smoothing shows that C-CCS green, derived from coconut shell, provides sharper and more obvious diffusion peaks compared to the red spectrum of C-CCP of coconut pith. This means that the playing time of C-CCS has sharper features, and the corresponding peaks are narrower, implying a higher degree of crystallinity. The well-organized characteristics of the functional groups and the increased peaks of the carbon spectra in the C-CCS spectrum suggest that it can have better catalytic activity. In this regard, it can be observed that the use of the

CaO/activated carbon catalyst from the coconut shell (C-CCS) exhibits a higher catalytic activity compared to the catalyst from the coconut pith (C-CCP), recommending the use of C-CCS for applications that demand higher activity of the catalyst.

4.1.3 SEM Analysis

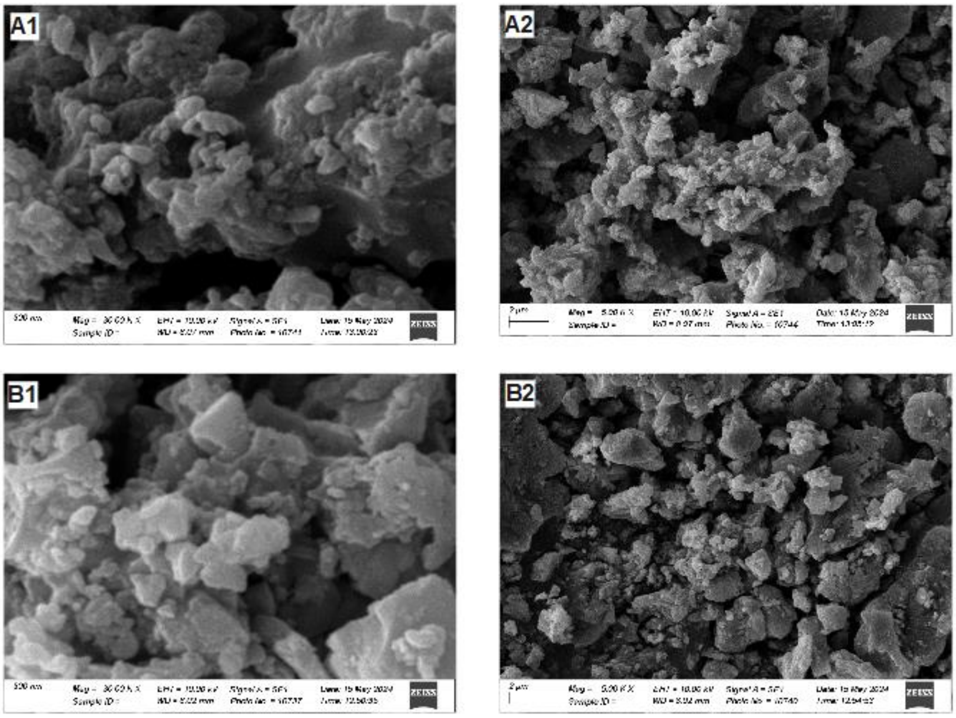

Figure 4.3 SEM Analysis of C-CCS (A1, AB) and C-CCP (B1, B2)

The analysis of C-CCS (A1 and A2) and C-CCP (B1 and B2) catalysts using the SEM maps displays the dissimilarity of their structure; it is established that the morphology of C-CCS is superior to that of C-CCP.

The SEM image of the C-CCS catalyst reveals that it has a hierarchical structure with round clusters of different sizes in a matrix-like form. The overall morphology of the surface seems rather featureless. However, one notices the presence of small pits, which point to a highly porous structure that is ideally suitable for use in a catalyst. They seem to consist of channels resembling a network of channels that can transport the reactants and the products as they undergo catalytic reactions. Also, the morphology of the C-CCS catalyst presented here seems to show a uniform distribution of the surface area, indicating that the active sites are uniformly distributed throughout the mass of the catalyst material. This uniformity is necessary in that it assists in maintaining a constant rate of catalytic reactivity throughout the entire catalyst-bearing substrate.

On the other hand, it is possible to see that the SEM image of the C-CCP catalyst presents a lower distinction than the Pd-CCP/Al2O3 sample with a configuration formed by irregular clusters and a non-uniform morphology causing a rougher aspect to the surface. The physiognomic outlines reveal roughness, and the pores and channels seem to be less distinguished than those in C-CCS's structure. There was a significant difference in the arrangement of the clusters on the surface of the C-CCP catalyst. The clusters in the C-CCP catalyst are not as organized as those in the commercial catalyst, indicating that they are randomly placed, and the morphology of the C-CCP catalyst appears to be a bit less structured than that of the commercial catalyst. Such a structure deviation could result in differing active sites and slow down mass movement during catalysis.

From SEM images of C-CCS and C-CCP, it can be deduced that the observed changes are due to the differences in precursors (coconut shell and coconut pith) and the formation process. The carbon nanomaterial produced from the coconut shell possibly possesses a more ordered structure with a larger surface area and pore sizes than the carbon derived from coconut pith. Furthermore, it is possible that due to the differences observed in the carbonization and activation processes of the two materials, there also exists a disparity in the structure of the catalysts. To summarize, the SEM analysis supports the conclusion that C-CCS has a much superior structure to the other catalysts, characterized by ordered and well-dispensed porosity and active sites. This structural advantage ought to further boost the catalytic efficiency of C-CCS over that of C-CCP, which should make the former even more suitable across the range of catalytic uses.

4.2 Biodiesel Fuel Characterization

4.2.1 Biodiesel Yield

Yield in biodiesel production is an important factor when considering efforts in the transesterification process that results in biodiesel from used cooking oil. It is the measure of the biodiesel yield following the reaction between feedstock oil and a biodiesel blend. Various elements in biodiesel production include the type of catalyst, methanol and oil ratio, amount of catalyst used, and the temperature to ensure that the oil has maximum biodiesel conversion.

The experiments used were the central composite design (CCD) to study the influence of these factors on biodiesel yield in a well-planned manner. In the experiment, the type of catalyst used was either C-CCS or C-CCP, and the amount of methanol per oil, loading of catalyst, and reaction temperature were modified based on the CCD matrix. Every single run of the experiment generated a biodiesel yield, which indicated that the specific effect of each variable was on biodiesel yield efficiency. Table 4.1 enlists the scheme used in the experiment and the resulting biodiesel production as a percentage for each trial:

Table 4.1 Experimental Design and the Corresponding Biodiesel Yields

Experiment	**Catalyst Type**	**Methanol to Oil Ratio**	**Catalyst Loading (wt %)**	**Reaction Temperature (°C)**	**Biodiesel Yield (%)***
1	C-CCS	8:01	6	60	80.25
2	C-CCS	8:01	6	65	82.15
3	C-CCS	8:01	8	60	81.48
4	C-CCS	8:01	8	65	83.72
5	C-CCS	10:01	6	60	85.36
6	C-CCS	10:01	6	65	88.19
7	C-CCS	10:01	8	60	87.89
8	**C-CCS**	**10:01**	**8**	**65**	**92.48**
9	C-CCP	8:01	6	60	77.25
10	C-CCP	8:01	6	65	79.63
11	C-CCP	8:01	8	60	79.48
12	C-CCP	8:01	8	65	82.72
13	C-CCP	10:01	6	60	80.91
14	C-CCP	10:01	6	65	85.19
15	C-CCP	10:01	8	60	84.92
16	**C-CCP**	**10:01**	**8**	**65**	**88.78**

*Biodiesel Yield (%) =Mass of Feedstock Oil Used/Mass of Biodiesel Produced ×100

4.2.2 Effect of Methanol to Oil Ratio

Methanol to oil is one of the process factors for transesterification and greatly controls biodiesel yield. Methanol also plays a double role in the reaction – it is both a reactant and a solvent, and the proportion of methanol to the oil affects the extent of conversion of the triglycerides into biodiesel. In our experiments, we investigated, as presented in Figure 4.4, the effects of varying the methanol-to-oil ratio at two levels: In syntheses of furans and pyrans with 8:1 and 10:1 selectivity, both C-CCS and C-CCP catalysts have been employed.

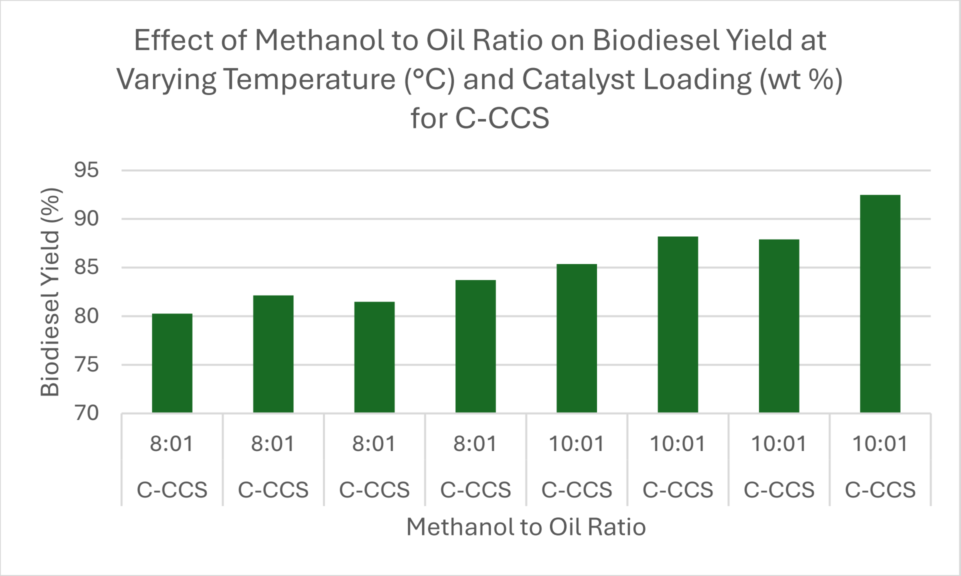

Figure 4.4 Effect of Methanol to Oil Ratio on Biodiesel Yield

In the experiments, a fixed quantity of about fifty milliliters of filtered and dried waste cooking oil (WCO) sample weighing about 5g was used in a three-neck flask. Methanol was added in the specified ratios 8:1. The mean values of VIF, TSS, RSD, and SSE for each trial must have been less than 10:1. The Required amount of the chosen catalyst, C-CCS or C-CCP, was added to the mixture, with temperature and stirring rate set to their levels as indicated above to enable proper mixing and proceeding of the reaction. After that, the reaction was allowed to cool, and the last step of separating the organic layers from the aqueous medium was completed. A centrifuge was used. The biodiesel yield was measured with the top layer, and the density and kinematic viscosity were measured at different temperatures, as shown in the following subsections.

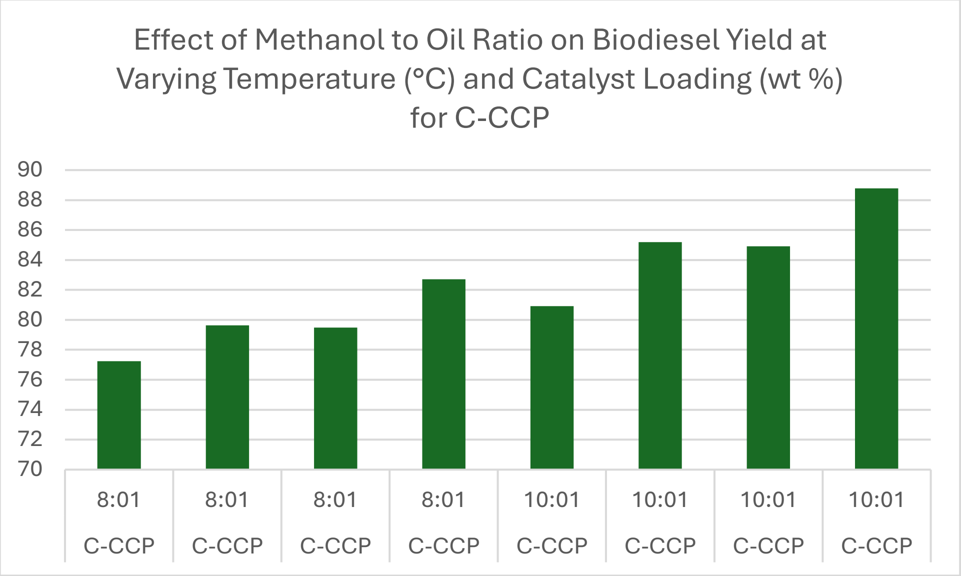

Figure 4.5 Effect of Methanol to Oil Ratio on Biodiesel Yield

The Methanol-to-oil ratio used in generating biodiesel was also found to have derived a clear relationship with biodiesel production. From a general viewpoint, the authors discovered that increasing the methanol-to-oil ratio improves the biodiesel yield. This can be attributed to the high concentration of methanol molecules used for the transesterification to overreach the triglycerides, resulting in very high biodiesel production. The data is presented in Figure 4.5. At methanol to oil ratio of 8:1. This was so, despite using the same feedstock: At 1:1, biodiesel yield was a bit lower than the one obtained in the 10:1 ratio. A possible reason for the effect, as mentioned above, is that triglycerides may not react completely with methanol due to the absence of a sufficient quantity of the reagent. For instance, when using a C-CCS catalyst, the yield rose from 80 per cent to 84 per cent within a short span, according to research done by Rafiei. 25% at 8: Retention of two comparable rats, one for the control group and the other for the experimental group, is important at a ratio of 1:85. 36% at 10: At 60°C, it was established that the reaction occurred in one ratio and from 82—15% to 88.19% at 65 °C. A similar trend was also observed when using a C-CCP catalyst, whereby yields were received at 77.25% to 80.90% at 60°C.

Higher ratios of methanol to oil are beneficial for biodiesel production in terms of yield; however, higher concentrations of methanol have a disadvantage in soap formation and increased usage of methanol, thus translating to higher costs. Hence, diametrically, the parameters have to be managed well to be cost-efficient while at the same time minimizing unwanted reactions. The results suggest that the 10:1 ratio is preferable in terms of biodiesel yield without having a hurdle, as the yield according to the maximum amount of biodiesel production reached 92. A direct experiment was completed to achieve this conversion using a fresh C-CCS catalyst at 65 °C, and the obtained result was 48%.

4.2.3 Effect of Catalyst Loading

The loading of the catalyst is also an important factor in the transesterifications since it directly impacts the rate of the process and the amount of biodiesel produced. The catalyst works to make the reaction sites more effective for converting the triglycerides to biodiesel, and the concentration of the catalyst plays an important role in the high efficiency of the process. Our study examined the effects of catalyst loadings at two levels: acetic acid of concentration six wt% and eight wt%, employing both C-CCS and C-CCP catalysts, presented in Figure 4.6.

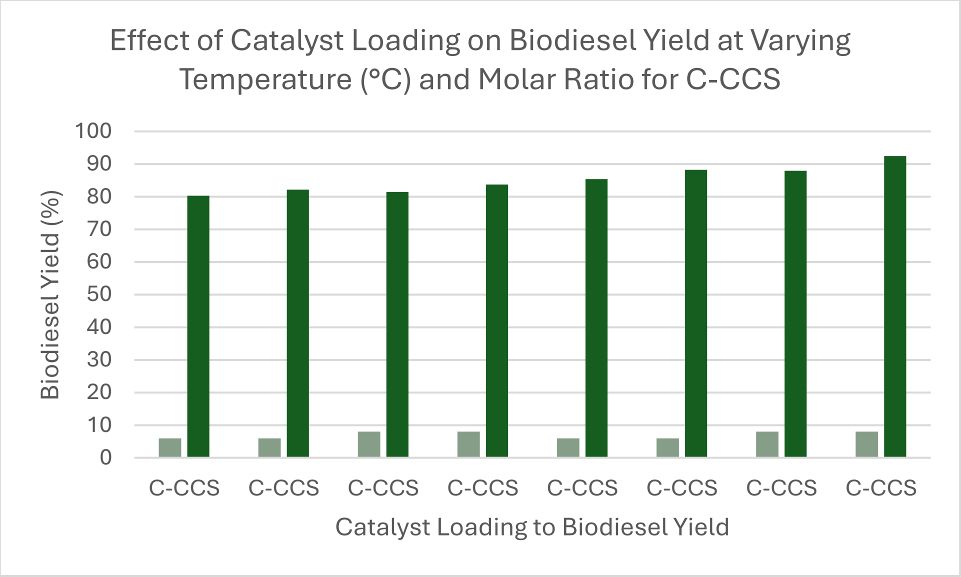

Figure 4.6 Effect of Catalyst Loading on Biodiesel Yield

In these experiments, we used five grams of filtered and dried waste cooking oil, which was added to a three-necked flask. Methanol was added in a consistent ratio of 10:1 in all ways; all the trials showed a similar degree of unsatisfactory performance. The catalyst was added in certain loadings of either 6 wt% or eight wt%. The reaction mixture was then heated to the desired temperature, and it was stirred constantly, as it was important to keep the reaction homogeneous. Following this, the solid products were allowed to cool, and the liquid layers were possibly separated using a centrifuge. The biodiesel yield found in the top layer was then quantified in the analysis of the recovery process.

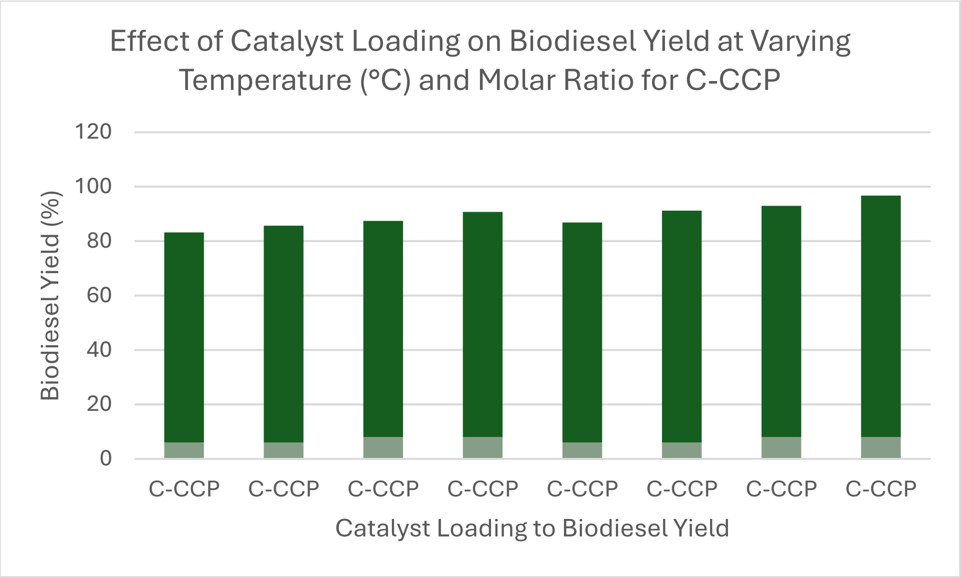

Figure 4.7 Effect of Catalyst Loading on Biodiesel Yield

When considering the extent of the incorporation of the catalyst, our studies showed a significant influence on biodiesel synthesis. In general, increasing the amount of catalyst content also enhanced biodiesel production, meaning that a higher amount of catalyst would be effective in converting more triglycerides to biodiesel. In the case of the YP-6 catalyst, the biodiesel production yield was relatively lower at lower catalyst loading conditions 6 wt % instead of 8 wt %. For instance, results showed, as presented in Figure 4.7, that with the C-CCS catalyst, at a temperature of sixty degrees Celsius, the yield has boosted from 85. 36% to 87. The catalytic activity of 89% was found with 6 wt% of the catalyst load, which was enhanced to as high as 8 wt% for the reaction. Likewise, at 65 °C, improvement was again noticed in the yield, which was recorded as 88. 19% to 92. 48%.

This implies that if a sufficient amount of a catalyst is not used in the transesterification process, then many of the active sites available for the reaction may not be occupied by the triglyceride molecules, thus making the conversion rate low. On the other hand, productivity increases when more catalysts are used in the biodiesel manufacturing process, showing that process efficiency is enhanced with higher catalyst loadings. A higher concentration of catalyst increases the availability of active sites in a favored direction, which carries a close relation with reaction kinetics, which almost increases the conversion rate of triglycerides to biodiesel. However, there are concerns such as the price of the catalysts and the availability of the catalysts in the market, which play a significant role in deciding the amount of catalyst that should be used. It is important to note that vital loadings can improve biodiesel yields simultaneously, escalating production costs and a higher value of loadings does not necessarily imply higher or better levels of process efficiency.

4.2.4 Effect of Reaction Temperature

Transesterification reactions depend on the temperature of the reaction medium, which influences the rate and the conversion of the biodiesel process. Temperature affects reaction rates, solubility of the substances participating in the reaction, and convective mass transfer, all of which dictate the effectiveness of transesterification in producing biodiesel. In our experimental setup, we investigated the effects of two different reaction temperatures: activation temperature at 60 °C and 65 °C, employing both C-CCS and C-CCP catalysts, with fixed methanol to oil ratio and amount of catalyst used.

For these experiments, 5 grams of the filtered and dried WCO were introduced into a three-necked flask. Methanol was added in a 10: The reactant to reward ratio was fixed at 1, while the amount of the catalyst was fixed at 6 wt. % or 8 wt. %. The reaction mixture was then heated to temperatures above either 60 °C or 65 °C with a thermostat and constant stirring of the reaction mixture to have a homogenized reaction mixture during the whole reaction. Finally, after the reaction in the flask, the flask was left to cool down, and then the layers were separated using a centrifuge. The biodiesel product in the top layer was quantitatively assessed to evaluate the biodiesel yield at the end of the experiment. The data is presented in Figure 4.8.

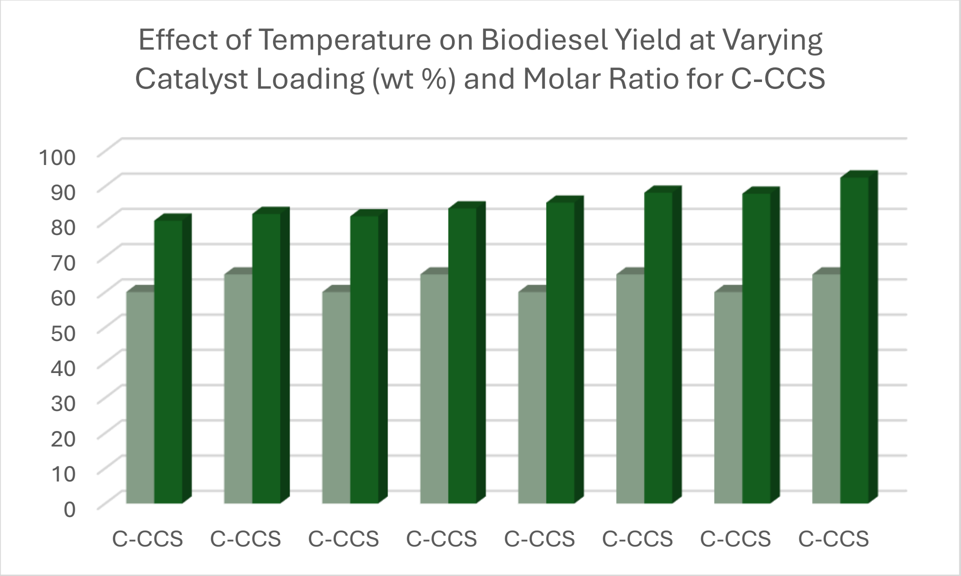

Figure 4.8 Effect of Temperature on Biodiesel Yield

Higher reaction temperature means more conversion of triglycerides to biodiesel and vice versa. As expected, when the reaction temperature was set to 60 °C as opposed to 65 °C, we obtained lower biodiesel yields. This result indicates that there could be an inhibitory effect at low temperatures, thereby making the conversion of the triglycerides partial due to slow reaction rates. For example, the yield was enhanced from 85 percent to more than 90 percent when using a C-CCS catalyst. For resin content, the values obtained ranged from as low as 36% at 60 °C to as high as 88. The maximum yield obtained was 19% yield by reaction at 65 °C and 6 wt% of catalyst loading. A yield of 87 was obtained using a catalyst loading of 8 wt % higher than that of the first cycle, as seen, and the result shows an increase in the yield. This may be due to its lower recovery when maintained at 88% at 60 °C to 92%. 48% at 65 °C. Comparable patterns were seen with the C-CCP catalyst, with yields rising from 80% at 100 ºC to 94% at 120 ºC. 91% to 85. 19% and from 84. 92% to 88. It is, however, important to note that the percent increase in tensile strength is differing and ranges from 78% for 6 wt% and 8 wt% loadings. The data is presented in Figure 4.9.

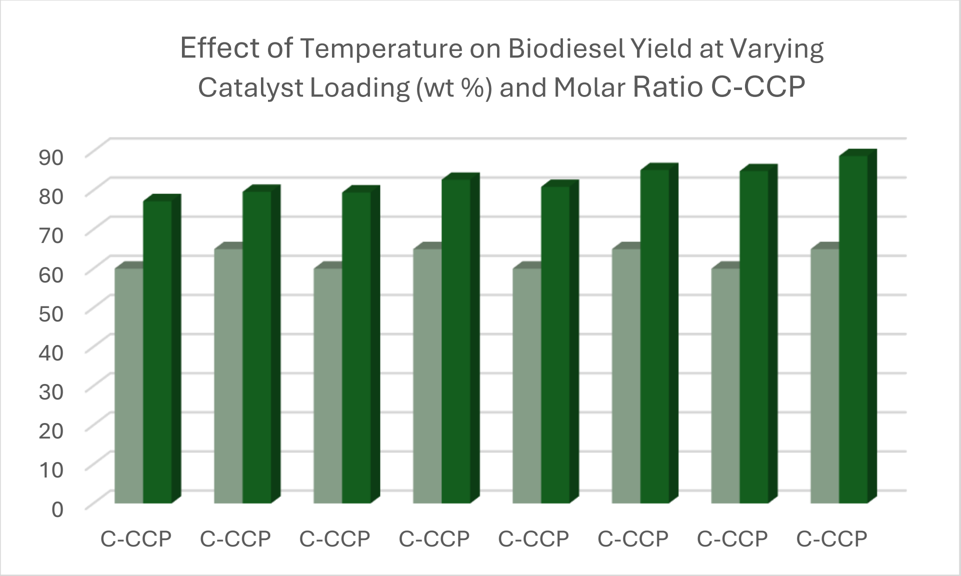

Figure 4.9 Effect of Temperature on Biodiesel Yield at Varying Catalyst

High temperatures decompose reacting materials more rapidly, dissolve the reacting components more effectively, and aid in difficult mixing, favoring a near stoichiometric conversion of the triglycerides to biodiesel. However, since certain factors, like energy consumption for the reaction and the capacity of the equipment used, affect the temperature, certain limitations are looked into when choosing the best temperature for a reaction to occur. Another advantage of higher temperatures is the increased production of biodiesel. Still, on the other note, it leads to high energy costs, and in some cases, one might have to invest in additional equipment to regulate temperatures. The research evidence indicated that a reaction temperature of 65 °C produces higher biodiesel yields without compromising on the practical implications of further increased reaction temperatures.

4.3 Optimized Biodiesel Physiochemical Characteristics

The optimized biodiesel yield derived from the ECC-catalyzed transesterification process exhibits favorable physiochemical characteristics aligned with the stringent requirements outlined by ASTM D6751 standards as presented in Table 4.2. With a flash point measured at 154 °C, significantly surpassing the minimum threshold of ≥120 °C, the biodiesel demonstrates enhanced safety during storage and handling, crucial for commercial viability. Moreover, its density, recorded at 0.88 g/cc at 25 °C, falls comfortably within the acceptable range of 0.82–0.90 g/cc, ensuring optimal fuel density for efficient combustion and engine performance. Furthermore, the viscosity of the optimized biodiesel, measured at 4.47 cSt at 40 °C, falls within the recommended range of 1.9–6.0 cSt, indicating favorable flow characteristics essential for engine operation and longevity. Additionally, the biodiesel exhibits a low acid value of 0.49 mg NaOH/g oil, well below the maximum limit of ≤0.8 mg NaOH/g oil, indicating minimal free fatty acid content and enhanced stability, thereby reducing the risk of engine corrosion. With a moisture content of only 0.01% (w/w oil), significantly below the maximum limit of <0.03%, the optimized biodiesel ensures prolonged shelf life by mitigating the risk of microbial contamination, further underscoring its suitability as a sustainable and environmentally friendly transportation fuel.

Table 4.2 Physiochemical Characteristics of Optimized Biodiesel

Property	**Test Methods**	**ASTM D6751 Range**	**Biodiesel**	**Conventional Diesel**	**ASTM D975 Range**
Flash Point (°C)	ASTM D93	≥120	**153**	60-80	≥52
Density @ 25°C (g/cc)	ASTM D445	0.82–0.90	**0.88**	0.81-0.87	0.82–0.88
Viscosity @ 40°C (cSt)	ASTM D445	1.9–6.0	**4.47**	1.9-4.1	1.9–4.1
Acid Value (mg NaOH/g oil)	ASTM D664	≤0.8	**0.49**	≤0.02	≤0.5
Moisture Content (% w/w oil)	–	<0.03	**0.01**	≤0.05	≤0.05
Cloud Point (°C)	ASTM D2500	Varies by region	**6**	-15 to 5	Varies by region

Chapter 5

Conclusion and Future Work Recommendations

The experimental outcome highlighted to some extent the prospect of using calcium oxide (CaO) supported on coconut shell-derived activated carbon (C-CCS) and pith-derived activated carbon (C-CCP) as biodiesel synthesis catalysts. Various reaction parameters like methanol to oil molar ratio, catalyst concentration, and reaction temperature were duly investigated to establish conditions that could yield maximum biodiesel. It is also worth noticing that the material named C-CCS demonstrated better performance in comparison with C-CCP; it can be concluded that the catalyst selection and the synthesis technique play a crucial role in determining the efficiency of a material. Additionally, the characterization of the catalysts through XRD, FTIR, and SEM offered rich information about the structural/morphology aspects of the catalysts, enabling a description of the process that controls the catalytic behaviour of the materials.

Besides the characterization of the catalyst, the physiochemical characterization of the biodiesel blend with the optimum yield per the stipulated Meta standards revealed its feasibility for use. The achievement of the optimized flash point, density, viscosity, acid value, and moisture content to some optimum values, which are on par with the existing fuels, also supports the idea of the proposed methodology and can be inferred that the coconut-derived catalysts can occupy a wider market in biodiesel production. Because a large part of the world’s tropical countries generates significant amounts of coconut waste, there is a unique chance for a two-fold treasure using coconut resources for catalyst synthesis – on the one hand, this would help solve the problem of proper waste disposal; on the other hand, it would contribute to the creation of renewable energy sources. Therefore, for large-scale practical applications using coconut-derived catalysts, it is suggested that a pilot scale trial and techno-economic analysis be done to get a clear evaluation of the effectiveness of the catalysts in biodiesel production plants. Such studies would be of tremendous value in establishing the commercial viability of applying the coconut waste-derived catalyst for large-scale biogas productions since they reveal valuable information on factors such as scalability, process economics, and the overall environmental impact of the catalyst.

The effective characterization of catalysts can cause individual characteristics and improvement processes to be unveiled and help researchers better understand the formulation of superior catalysts. However, alongside the efforts toward synthesizing new and improved catalysts, the investigation of process improvements towards higher biodiesel yield, product purity, and process sustainability should not be ignored either. These are looking for new reaction media, exploring other reaction parameters, kinetic modelling of the conversion reactions, and considerations of process integration features to enhance biodiesel yield while reducing energy and material utilization. Also, it is necessary to examine the possibilities of incorporating renewable energy sources like sunlight or energy enacted from biomass into biodiesel manufacturing processes to decrease greenhouse gas emissions and improve the general comprehensibility of increased sustainability.

Even though using coconut-based catalysts is viable, it is conceivable to focus on other biomasses, which include algae, jatropha, waste cooking oil, and microalgae, as potential sources of renewable lipids. Through feedstock diversification, more options are opened to researchers with regards to fuel feedstocks; it makes biodiesel production systems more robust and sustainable; and overall, the feedstock competition with food crops will be minimized together with any probable land use and environmental impacts that may stem from feedstock production. Future research must continue to investigate new sources of feedstocks and focus on new materials and methods of catalysis to enhance biodiesel production yield, quality, and effectiveness of the conversion process. This is also about new approaches to catalyst synthesis, including the discovery of new forms of catalysts like nanostructured catalysts, supported metal nanoparticles, and solid acid catalysts, which can improve the activity and selectivity of the catalyst and its stability.

An exciting area that requires further exploration for future work is the utilization of complex real-time process monitoring and control techniques that could be employed to enhance biodiesel production processes. In addition, there is an increasing approachability of the overall life-cycle implications of biodiesel production that must be examined in terms of feedstock farming and processing as well as distribution and utilization. Further studies need to be devoted to the number and optimization of LCA to evaluate the environmental impacts of biodiesel production systems and determine the possible changes in the efficiency of resource utilization, the level of GHGE, and other characteristics that can increase the overall sustainability of the systems. Also, there is an increased concern in measuring the impact that biodiesel production creates, especially in job creation opportunities, rural economic development, and improved energy security for users.

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A. Ahsan, F. Jamil, M. A. Rashad, M. Hussain, M.H. Hamayun, A Comparative Review on Electrocatalytic Reduction of Nitrogen and Nitrate to Ammonia.