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Biodiesel Production from Crude Palm Oil Using Eggshell-Derived Calcium Oxide Catalyst

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ABSTRACT: Biodiesel production from crude palm oil (CPO) presents a sustainable alternative to fossil fuels, addressing energy security and environmental concerns. This study investigates the synthesis of biodiesel using a two-step transesterification process, incorporating an eggshell-derived calcium oxide (CaO) catalyst. The methodology includes purification of CPO, followed by esterification with sulfuric acid and transesterification with the CaO catalyst. Optimized reaction conditions yielded a biodiesel production rate of 78.6%, with the final product meeting established quality standards for density, viscosity, flash point, and cetane number. The environmental advantages of CPO biodiesel are highlighted, including reduced carbon emissions and the promotion of sustainable agricultural practices. Additionally, the research underscores the economic potential of biodiesel in enhancing energy independence and supporting rural development in palm oil-producing regions. The findings advocate for further research and investment to optimize biodiesel production processes and fully harness the benefits of this renewable energy source.

Keywords: Energy Security, Environmental Sustainability, Biodiesel, Crude Palm Oil, Emissions, Catalyst.



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INTRODUCTION

The quest for sustainable energy solutions has become increasingly critical in the face of escalating global energy demands and the pressing need to mitigate climate change. Despite their economic advantages, traditional fossil fuels have been identified as major contributors to greenhouse gas (GHG) emissions, air pollution, and environmental degradation. Consequently, developing and deploying renewable energy sources have emerged as essential strategies for achieving environmental sustainability and energy security. Among these renewable sources, biodiesel has garnered significant attention due to its potential to serve as a cleaner alternative to conventional diesel.

Biodiesel, derived from biological sources such as vegetable oils and animal fats, offers several environmental benefits, including reduced GHG emissions and decreased reliance on finite fossil

fuel reserves (A.Z et al., 2024). One of the most promising feedstocks for biodiesel production is crude palm oil (CPO), primarily due to its high oil yield per hectare and the extensive cultivation of oil palm in tropical regions. Palm oil, a versatile and widely used agricultural product, has the potential to play a pivotal role in the transition to sustainable energy systems (Souza et al., 2010).

Biodiesel emerges as a critical alternative to traditional petroleum-based fuels, particularly as the world faces challenges such as climate change and the depletion of fossil fuel reserves. The economic benefits of CPO-based biodiesel production are substantial, especially in palm oil-producing countries like Indonesia and Malaysia. The establishment of biodiesel plants in these regions stimulates economic growth, creates direct employment opportunities, and fosters related industries, including agriculture, transport, and manufacturing (Fakai, 2023) (Harsono et al., 2012). Additionally, biodiesel production enhances national energy security by reducing dependency on imported fossil fuels, retaining foreign exchange, and insulating economies from global oil price volatility ("Sustainability of Palm Biodiesel in Transportation: A Review on Biofuel Standard, Policy and International Collaboration Between Malaysia and Colombia," n.d.).

Biodiesel production also plays a significant role in rural development and poverty alleviation, particularly benefiting smallholder farmers involved in palm oil cultivation. The integration of these farmers into the biodiesel supply chain ensures a broader distribution of economic benefits, contributing to improved livelihoods and economic resilience in rural communities. On the geopolitical front, biodiesel production from CPO can alter global energy politics by reducing reliance on oil imports and fostering international cooperation in sustainable energy initiatives. This shift can also support global climate change mitigation efforts, aligning countries with international sustainability goals by reducing greenhouse gas emissions (Akhtar, 2023).

The diversification of energy sources through biodiesel production promotes energy resilience and stability. Biodiesel offers a renewable and sustainable energy alternative that can be integrated into existing infrastructures, unlike finite fossil fuels (Tan et al., 2009). This diversification is crucial for withstanding supply chain disruptions caused by natural disasters, geopolitical conflicts, or other unforeseen events. By producing biodiesel domestically, countries can reduce their exposure to global oil price fluctuations, achieving more excellent economic stability (E.E. et al., 2013; Fakai, 2023; Maltitz & Stafford, 2011; Photaworn et al., 2017). The agricultural sector also benefits from this stability, as palm oil producers gain a more predictable market for their products, encouraging further investment and enhancing the resilience of the agricultural economy (Kumar, 2007).

Despite its economic and geopolitical advantages, biodiesel production from CPO raises environmental and sustainability concerns. While biodiesel is considered to have a lower environmental impact than fossil fuels, particularly in reducing emissions of pollutants like sulfur oxides and particulates, the sustainability of palm oil cultivation is critical. Issues such as deforestation and land use changes pose significant challenges, potentially offsetting the environmental benefits of biodiesel (Von Maltitz, 2012). Sustainable cultivation practices, certifications, and standards, such as those from the Roundtable on Sustainable Palm Oil (RSPO), are essential to mitigate these negative impacts and ensure the credibility of biodiesel as a sustainable energy source (R.S.P.O., 2018).

Waste utilization strategies are vital to enhancing the environmental sustainability of biodiesel production. The palm oil industry generates various types of waste, including empty fruit bunches (EFB), palm kernel shells (PKS), and palm oil mill effluent (POME). Proper management and utilization of these waste products can mitigate environmental pollution and contribute to a circular economy. For example, EFB can be used as biomass fuel, PKS can be activated carbon or fuel, and POME can be converted into biogas for electricity generation, reducing reliance on fossil fuels and lowering greenhouse gas emissions (Maltitz and Stafford 2011).

Biodiesel production from CPO presents a promising pathway toward environmental sustainability by reducing carbon emissions, promoting sustainable cultivation practices, and effectively utilizing waste. However, realizing these benefits requires strict adherence to certification standards and continuous efforts to address associated challenges (Lim & Teong, 2010). By fostering sustainable palm oil production, the industry can contribute to global environmental conservation and sustainable development goals, ultimately playing a pivotal role in the transition to a more sustainable and resilient energy future (Lam & Lee, 2012; Yacob et al., n.d.).

Biodiesel has been utilized either as a standalone fuel or in combination with petroleum diesel at various ratios in many countries (Zhang et al., n.d.). This biofuel is produced from a renewable, domestically available resource, thereby reducing reliance on imported petroleum(Zeng et al., 2009). Compared to petroleum-based fuels, biodiesel exhibits a more favorable emissions profile, with lower levels of sulfur dioxide, carbon monoxide, unburned hydrocarbons, and particulate matter. Additionally, biodiesel possesses favorable characteristics such as appropriate viscosity, a higher flash point, and a higher cetane number, and it does not require significant engine modifications (Yang et al., 2014). The cost of raw materials is a considerable factor in biodiesel production. Utilizing low-cost feedstocks is considered an optimal alternative for biodiesel production (Sharma & Singh, 2010).

Transesterification is a reaction that transforms a carboxylic acid ester into another carboxylic acid ester by swapping the organic alkyl groups of non-edible and edible oils with methanol. When alcohol interacts with free fatty acids, they combine to form alkyl esters, which are biodiesel. Several technologies, including a one-step reaction process and a two-step reaction process, have been developed to reduce the cost of biodiesel production from various sources (Dubey et al., 2015; Loong & Idris, 2016).

The one-step reaction is a transesterification process suitable for feedstock oils with low free fatty acid (FFA) content, though it is challenging if the FFA content is high. A significant disadvantage of this method is the higher consumption of catalyst and alcohol compared to the two-step transesterification process (Pisarello & Querini, 2013). The two-step reaction process, on the other hand, involves an initial esterification step followed by transesterification (Cai et al., 2015). Some researchers have also employed an additional two-step esterification process.

This two-step process is applicable to any feedstock oil, particularly those with high FFA content. When the FFA content exceeds 1 wt%, the first esterification step, followed by transesterification, is highly effective. The purpose of the esterification step is to reduce the FFA content as much as

possible to make the feedstock suitable for transesterification. This two-step reaction process has proven to be one of the most efficient methods for utilizing various feedstock oils in biodiesel production (Demirbas, 2011).

This paper aims to explore the synthesis of biodiesel from CPO using a two-step transesterification reaction. It will also explore the environmental benefits of biodiesel, such as reduced carbon emissions and pollution and examine sustainable cultivation practices that can mitigate the ecological impacts of palm oil production.

METHOD

The materials used in this research include crude palm oil (CPO) sourced from Enugu Market, Enugu State, eggshells, isopropyl alcohol (IPA), phenolphthalein indicator, potassium hydroxide (KOH) 0.1 N, sulfuric acid (H2SO4) of analytical grade, potassium hydrogen phthalate, methanol of analytical grade, Whatman 42 filter paper, distilled water, acetone, hydrochloric acid (HCl) 0.5 N, carbon tetrachloride (CCl4), Wijs reagent, potassium iodide (KI), sodium thiosulfate (Na₂S₂O₃), and acetic acid (CH₃COOH).



Plate 1: Crude Palm Oil

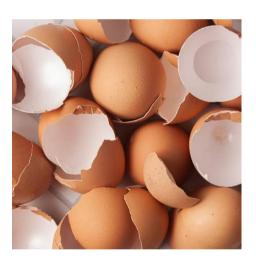


Plate 2: Eggshells

Catalyst Preparation

The CaO heterogeneous catalyst was synthesized using eggshells as the precursor. Initially, the eggshells underwent a cleaning process with water to eliminate dirt and sand, followed by rinsing with distilled water to eliminate any remaining impurities. Subsequently, the cleaned shells were coarsely ground using a mortar and pestle and then subjected to calcination at temperatures of 800 °C and 900 °C for varying durations of 5, 10, and 20 hours. After calcination, the shells were crushed and sieved to obtain particles with a size of 200 mesh. The resulting material was stored in a desiccator for further use (Fakai, 2023).

CPO Purification Process for Biodiesel Synthesis

Before biodiesel synthesis, crude palm oil (CPO) underwent purification through filtration to remove minute particles. Subsequently, it was washed with warm distilled water (50 °C) using a separator funnel and homogenized. Samples were allowed to settle for approximately a day, resulting in the formation of distinct layers, with the lower layer comprising water and the upper layer consisting of washed palm oil. The purified CPO, weighing 100 g, was then heated at 105 °C for approximately 1 hour, rendering it ready for utilization in biodiesel synthesis (Alkabbashi et al., 2009).

Biodiesel Synthesis Procedure

The biodiesel synthesis process comprised a two-stage reaction sequence involving esterification using a sulfuric acid catalyst (H2SO4) and transesterification utilizing a CaO catalyst derived from eggshells. Both reactions were conducted within a 500 mL three-necked batch reactor equipped with a thermometer, a magnetic stirrer, and a reflux condenser. Esterification of Crude Palm Oil (CPO) Using H2SO4 Catalyst Esterification reactions were executed with varied reaction durations (1, 2, 3, and 4 hours) and reaction temperatures (60, 65, 70, and 75 °C). In a three-neck flask containing 100 g of CPO, a mixture of 2 g of concentrated H2SO4 catalyst and methanol (oil to methanol ratio of 1:24) was introduced. The mixture underwent reflux under magnetic stirring for 3 hours at a reaction temperature of 70 ± 2 °C. The resultant mix comprised two layers: the upper layer containing methanol and sulfuric acid and the lower layer consisting of esterified oil. The esterified oil was separated from the methanol and other impurities using a separating funnel (Maulidiyah et al., 2022).

Transesterification of Crude Palm Oil (CPO)

The initiation of the transesterification reaction involved the mixing of 4 g of CaO catalyst, derived from eggshells, and methanol in a three-neck flask, with a molar ratio of oil to methanol set at 1:6. This mixture underwent magnetic stirring for 30 minutes. Subsequently, the oil obtained from esterification was heated to a temperature of 105 °C for approximately 1 hour, followed by a further period at a lower temperature of 50 °C. The oil was then combined with the catalyst and methanol mixture and stirred for 3 hours at a reaction temperature of 60 ± 2 °C. Upon completion of the reaction, the flask was cooled by immersion in cold water, and the resulting mixture was transferred to a separator funnel, left at room temperature overnight, resulting in the formation of two layers: the lower layer consisting of glycerol and the upper layer comprising crude biodiesel. The initially formed biodiesel was subsequently transferred to a separating funnel and subjected to washing with warm distilled water (50-60 °C) at a weight ratio of biodiesel to distilled water. This process was repeated with variations in catalyst weight, reaction time, temperature, oil-to-methanol molar ratio, and the duration of catalyst calcination (Vandkata et al., 2012).

The biodiesel yield was determined using the equation:

% yield =
$$\frac{weight \ of \ biodissel}{weight \ of \ CPO \ used} x \ 100 \dots "Eq. (1)"$$

Characterization of Biodiesel

The characteristics of biodiesel were assessed by analyzing its water content (ASTM D-2709), density (ASTM D-1298), viscosity (ASTM D-445), flash point (ASTM D-93), acid number (ASTM D-664), iodine number (AOCS Cd 1-25), and cetane number (ASTM D-613).

RESULT AND DISCUSSION

Free Fatty Acid Content and Saponification Value

The crude palm oil (CPO) evaluated in this study exhibited a free fatty acid (FFA) content of 5.0%, which is notably higher than the 0.778% reported for Lagenaria siceraria and lower than the 13% reported for neem seed oil. The saponification value of 220 mg KOH/g falls within the range of previously reported values, being lower than the 244.2 mg KOH/g for tobacco boxes (Essien et al., 2013) but higher than the 197.75 mg KOH/g for long-handle dippers (M.K.A et al., 2012). These values are critical in determining the suitability of CPO for biodiesel production, as high FFA levels necessitate pre-treatment steps to prevent soap formation during transesterification (Atabani et al., 2012).

Table 1. Free fatty acid content and saponification value of CPO

Free Fatty Acid	5.0 ± 0.02
Saponification Value	220 ± 0.03

Results are mean, standard deviation of triplicate determination

The FFA content in the provided research data ($5.0 \pm 0.02\%$) aligns with typical values seen in crude palm oil. It is slightly higher than the optimized FFA levels discussed by (T. J. et al., 2023), who reduced the FFA content to below 1% to avoid soap formation during transesterification. Punvichai et al. and Hayyan et al (2011) (Hayyan, 2011). Dealt with CPO varieties or derivatives with varying FFA content but generally advocated for the need to reduce FFA levels for more efficient biodiesel production. The saponification value in the provided research (220 \pm 0.03 mg KOH/g) is relatively high, indicating a significant amount of soap-forming components, which can interfere with biodiesel production. This aligns with the general findings in the scholarly articles where higher saponification values necessitate careful process management to avoid soap formation and ensure high biodiesel yield.

Biodiesel Yield and Properties

Table 2. Results of three-run average data for three selected batches of transesterification.

Experimental Conditions	TRIAL 1	TRIAL 2	TRIAL 3
Cao quantity (g)	1.5	3	4.5
Reaction temperature (°C)	60	60	60
Reaction time (minutes)	90	90	90
CPO to methanol ratio	6:1	6:1	6:1

Biodiesel obtained (cm ³)	110	220	330
By-product obtained (cm ³)	29.5	60	90
Biodiesel yield (%)	78.6	78.6	78.6

The biodiesel yield from CPO in this study averaged 78.6%, which is consistent with other studies utilizing heterogeneous catalysts. For instance, studies utilizing CaO derived from eggshells have reported comparable yields, highlighting the efficiency of this catalyst in biodiesel synthesis (P.L. et al., n.d.). The value is lower when compared to 89.7 (Crude palm oil methyl ester) reported by Naserelldeen et al. (2009), (and 78.6%) reported for Lagenaria Siceraria methyl ester by Fakai (2003) but remarkably higher than (49.8%) for coconut oil methyl ester by musa et al. (2016) (A.K et al., 2009). A similar value was reported for avocado seed methyl ester (78%), as reported by Dagded (2019). Ayodeji et al. (2018) reported a higher value of (97.1) for soybean oil methyl ester. These differences could be attributed to fatty acid composition, free fatty acid contents, catalyst type and concentration, reaction conditions, presence of impurities, processing techniques, and feedstock variability.

Table 3. Properties of Biodiesel Produced from CPO in comparison with related works.

Parameters	% Yield	Density	K. Viscosity	Flash Point	C. Number
ASTM Standard		D-1298	D445	D93	D613
Present Study	78.6	0.88	4.0	268	47
Bottle guard oil (Fakai, 2023)	88.4	N/A	3.94	130	47.7
Coconut oil (Musa et al., 2016)	49.8	0	4.7	100	N/A
Avocado seed oil (Dagde, 2019)	78	0.86	3	162	N/A
Soybean Oil (Ayodeji et al., 2018)	97.1	0.87	2.7	142	51
Acid Value	0.8	N/A	0.5	0.18	0.89

The properties of the produced biodiesel, including its density, kinematic viscosity, flash point, and cetane number, align with ASTM standards, confirming its suitability as an alternative fuel. The yield of 78.6% in the provided research is comparable to yields reported in scholarly articles, such as Hayyan et al. (2011), who achieved yields of 80-90%. Jansri and Prateepchaikul's study, which used a two-stage process to mitigate the effects of high FFA content, achieved slightly higher yields at 86.6% (J. J. & G, 2011). The provided research yield is typical for biodiesel produced from CPO with similar FFA content, indicating that the process conditions were reasonably practical. However, there is room for optimization to reach the higher yields noted in other studies.

Fatty Acid Composition

Table 4. Shows the fatty acid Composition of Crude palm oil.

Fatty Acid	Molecular Weight	Percentage
Saturated Fatty Acid		•
Lauric (c12:0)	200	0.33
Myristic(c14:0)	282	0.99
Palmitic (c16:0)	256	41.49
Stearic (c18:0)	284	4.49
Arachidic (c20:0)	312	0.40
Behenic (c22:0)	340	0.09
Other	327	0.19
Monounsaturated Fatty Acid		
Palmitoleic (c16:1 n-7c)	254	0.17
Elaidic (c18:1 n-9t)	282	0.11
Oleic (c18:1 n-9c)	282	40.18
Eicosenoic (c20:1 n-9c)	310	0.14
Other	366	
Polyunsaturated Fatty Acid		
Linoleic (c18:2 n-6cc)	280	9.95
Linolenic (c18:3 n-3ccc)	278	0.28
Other	271	012

Average Mw of fatty acid 271.5

The fatty acid composition analysis of CPO revealed a predominance of palmitic (16:0) and oleic (18:1) acids, accounting for 41.49% and 40.18%, respectively. This aligns with existing literature on CPO's fatty acid profile, which typically exhibits high levels of saturated and monounsaturated fatty acids (Knothe, 2010). The balanced composition of saturated and unsaturated fatty acids contributes to the desirable properties of biodiesel, such as oxidative stability and cold flow performance (Moser, 2009).

Sustainable Energy Development

The transition to sustainable energy sources is imperative in mitigating climate change and reducing dependence on finite fossil fuels. Biodiesel, mainly derived from crude palm oil (CPO), emerges as a viable candidate for this transition, as supported by the findings of this study. The results are discussed in the context of various parameters that underscore the feasibility and environmental benefits of CPO-derived biodiesel, aligning with the broader goals of sustainable energy development.

Energy Security and Environmental Sustainability

Biodiesel production from CPO contributes significantly to energy security by providing a renewable and locally sourced energy alternative. This reduces dependency on imported fossil fuels and enhances energy independence (Ogunlowo et al., 2020). Additionally, the environmental benefits are substantial, as biodiesel combustion results in lower greenhouse gas emissions compared to conventional diesel. Lifecycle analyses have demonstrated that biodiesel from CPO can reduce GHG emissions by up to 85%, highlighting its potential in mitigating climate change (Lam et al., 2009).

The utilization of waste products, such as glycerol, further enhances the sustainability of biodiesel production. Glycerol, a byproduct of the transesterification process, can be repurposed for various industrial applications, promoting a circular economy and reducing environmental burden (Choo et al., 2002). The adherence to certification standards, such as those established by the Roundtable on Sustainable Palm Oil (RSPO), ensures that CPO production meets stringent environmental, social, and economic criteria, thus promoting sustainability within the industry (RSPO, 2020).

CONCLUSION

The synthesis of biodiesel from crude palm oil (CPO) holds significant promise as a sustainable energy solution, as evidenced by the findings presented in this paper.

Firstly, the utilization of heterogeneous catalysts derived from eggshells for biodiesel synthesis demonstrates the conversion of waste to catalyst. The synthesis process involved cleaning, calcination, and sieving of eggshells to obtain calcium oxide (CaO) catalyst particles [. This method not only utilizes a readily available and inexpensive precursor but also contributes to waste valorization by repurposing eggshell waste(Kumar Tiwari et al., n.d.). The use of CaO catalysts in transesterification reactions offers advantages over traditional homogeneous catalysts, including reduced soap formation and ease of separation, enhancing the efficiency and scalability of biodiesel production processes.

Furthermore, the optimization of reaction parameters, such as catalyst quantity, reaction temperature, and duration, plays a crucial role in maximizing biodiesel yield. The results of transesterification trials indicate a consistent biodiesel yield of 78.6%, highlighting the robustness of the synthesis process. By systematically varying reaction conditions and analyzing their impact on biodiesel production, researchers can identify optimal operating conditions to improve process efficiency and yield while minimizing resource consumption and waste generation(Punvichai Biodiesel Production from Jatropha Oil (Jatropha Curcas) with High Free Fatty Acids: An Optimized Process, n.d.).

Environmental sustainability is a key driver behind the shift towards biodiesel production from CPO. The paper underscores the importance of sustainable cultivation practices and waste utilization strategies in mitigating the environmental impacts associated with palm oil production. By adhering to certification standards and implementing best practices such as agroforestry, palm oil producers can minimize deforestation, preserve biodiversity, and reduce carbon emissions, thereby enhancing the sustainability of biodiesel production processes.

Moreover, biodiesel derived from CPO offers tangible benefits in terms of energy security and diversification. By reducing dependency on finite fossil fuel reserves and mitigating geopolitical risks associated with fossil fuel imports, biodiesel production from CPO contributes to national energy independence and resilience (Oliveira et al., 2018). The localization of biodiesel production also stimulates economic growth, creates employment opportunities, and fosters innovation in the

renewable energy sector, thereby enhancing overall energy security and socio-economic development.

The synthesis of biodiesel from crude palm oil represents a solution to the complex challenges of climate change, energy security, and environmental sustainability. By leveraging innovative catalyst preparation methods, optimizing reaction parameters, and embracing sustainable practices, biodiesel production from CPO offers a viable pathway toward a cleaner, more secure, and sustainable energy future. Continued research and development efforts in this field are essential to improve process efficiency further, reduce environmental impacts, and realize the full potential of biodiesel as a renewable energy source.

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