# Sustainable Biodiesel Synthesis from Waste Cooking Oil Using Eggshell and Coffee Husk Catalyst

Vebria Ardina[1], Vany Mei Lia[2], Rahmi[3], Lenny Marlinda[4]

{vebriaardina@unja.ac.id[1], vanymeilia2@gmail.com[2], rahmi.chem@unja.ac.id[3], marlindalenny@unja.ac.id[4]}

Department of Industrial Chemistry, University of Jambi, Jambi, Indonesia[1,4], Department of Chemical Engineering, University of Jambi, Jambi, Indonesia[2], Department of Chemistry, University of Jambi, Jambi, Indonesia[3]

Corresponding author: marlindalenny@unja.ac.id

**Abstract.** The production of biodiesel from renewable waste materials offers a sustainable alternative to fossil fuels. This study utilized waste cooking oil (WCO) as a feedstock and developed a heterogeneous CaO–$K_2O$ composite catalyst derived from eggshells and coffee husks. The catalyst was synthesized through calcination at 900 °C for 3 hours and characterized using X-ray Diffraction (XRD). Transesterification was conducted at 65 °C with a methanol-to-oil molar ratio of 9:1, a 60 minute reaction time and catalyst loadings ranging from 0.5–3 wt%. XRD analysis confirmed the successful formation of active CaO phases and the incorporation of potassium species. The highest FAME yield of 71.7% was obtained at 0.5% catalyst loading. The produced biodiesel exhibited a density of 0.91-0.92 g/mL and a heating value of 5.016 kcal/kg. These results indicate that eggshell and coffee husk composites are potential low-cost catalysts for biodiesel production.

**Keywords:** Biodiesel, waste cooking oil, CaO–$K_2O$ catalyst, eggshell, coffee husk.

## 1 Introduction

The global energy crisis and the depletion of fossil fuel reserves have encouraged the exploration of renewable energy alternatives such as biodiesel. In Indonesia, the dependency on petroleum-based fuels has become a critical issue, prompting the government to promote biofuel utilization through Presidential Instruction No. 1 of 2006. Among various renewable resources, waste cooking oil (WCO) has emerged as a promising and low-cost feedstock for biodiesel production due to its abundance and environmental benefits[1][2]. Indonesia's palm oil cooking oil consumption reaches approximately 16.2 million kiloliters per year, with an estimated 3 million kiloliters of recoverable waste cooking oil that can be used as biodiesel feedstock; this volume is projected to supply around 32% of national biodiesel demand, provided quality standards and economic feasibility are met. Converting this waste into biodiesel not only mitigates environmental pollution but also provides a sustainable energy solution with economic value.

SICBAS 2025, November 06, Palembang, Indonesia

DOI 10.4108/eai.6-11-2025.2364458

Waste cooking oil (WCO) typically contains high levels of free fatty acids (FFA) ranging between 3% and 40%, which strongly influence the reaction pathway and biodiesel yield [3]. When the FFA content exceeds 2%, an acid-catalyzed esterification step is generally required to prevent soap formation during transesterification [4]. Furthermore, WCO tends to have higher viscosity and oxidation levels compared to fresh oil, factors that can limit the conversion efficiency. Thus, optimizing the catalytic system is crucial for effective FFA reduction and efficient biodiesel synthesis[5].

Heterogeneous catalysts have gained increasing attention as environmentally friendly alternatives to homogeneous catalysts due to their reusability, thermal stability, and ease of separation from the product[6]. Calcium oxide (CaO), derived from natural sources such as eggshells and mollusk shells, is one of the most studied heterogeneous catalysts because of its strong basicity, low toxicity, and low cost [7]. The eggshell which contains approximately 94% calcium carbonate ($CaCO_3$), making it an excellent precursor for CaO-based catalysts[8]. Similarly, coffee husk waste, which represents nearly 35% of total coffee production residues, contains potassium (K) compounds that can be transformed into potassium oxide ($K_2O$) another active basic oxide for transesterification[9]. Several recent studies have shown that such composite catalysts significantly increase biodiesel yield while maintaining high thermal stability and reusability. However, the performance of $CaO–K_2O$ composites derived specifically from eggshells and coffee husks remains underexplored, particularly regarding the relationship between catalyst loading and biodiesel yield in transesterification reactions involving WCO.

Therefore, this study aims to synthesize a sustainable $CaO–K_2O$ composite catalyst derived from eggshell and coffee husk waste for biodiesel production from waste cooking oil. The catalyst was characterized using XRD analysis to determine its crystalline structure. The resulting biodiesel was further analyzed in terms of calorific value, density, and yield. By concentrating on these key parameters, this work provides a focused insight into the feasibility of waste-based heterogeneous catalysts, supporting circular economy principles in the development of renewable fuels.

## 2 Material and methods

### 2.1 Materials

The synthesis of biodiesel in this work utilized cooking oil (WCO) as the primary feedstock, sourced from a local fried chicken vendor. To ensure the quality of the feedstock, the oil underwent a simple pre-treatment involving filtration and polishing with activated carbon to eliminate food residues and impurities. For the catalyst components, two types of biomass waste were repurposed: chicken eggshells and coffee husks, both collected from local suppliers and processing facilities. These materials were selected for their high content of calcium carbonate ($CaCO_3$) and potassium oxide ($K_2O$), respectively these components function as the primary catalytic centers to facilitate the transesterification process. Chemical reagents, including methanol ($CH_3OH$) for the transesterification process and distilled water for cleaning stages, were used without further purification. By utilizing these waste-derived resources, the study emphasizes a cost-effective and sustainable approach to renewable energy production.

### 2.2 Catalyst preparation and characterization

Before catalyst synthesis, the eggshells and coffee husks were pretreated to remove impurities and excess moisture. The eggshells were carefully washed with tap and distilled water to eliminate residual organic matter, then sun-dried for 24 hours and oven-dried at 105 °C for 2 hours. After drying, they were finely ground to increase surface area and ensure uniformity. The coffee husks were washed, dried, and ground into smaller particles to promote even heat transfer during calcination.

The precursors underwent thermal treatment in a muffle furnace, maintained at 900 °C for 3 hours. The calcination process decomposed calcium carbonate ($CaCO_3$) in the eggshells into calcium oxide (CaO) and converted potassium compounds in the coffee husks into potassium oxide ($K_2O$). Upon reaching ambient temperature, the calcined powders were combined using a predetermined ratio to produce a CaO–$K_2O$ composite catalyst. The composite was kept in a hermetically sealed vessel to prevent moisture absorption and re-carbonation with atmospheric $CO_2$.

The structural properties and crystalline phases of the synthesized catalyst were then verified through X-ray diffraction (XRD) analysis. This characterization ensured the successful formation of the active CaO and $K_2O$ phases required for the subsequent transesterification process.

### 2.3 Transesterification process

The experimental setup for the reaction consisted of a 500 mL three-neck flask equipped with a reflux condenser to prevent solvent loss, alongside a magnetic stirrer and thermometer for precise parameter control. The reactor vessel was charged with the waste cooking oil, methanol, and the synthesized CaO–$K_2O$ composite. The process was allowed to proceed at a controlled temperature of 65 °C, utilizing a 9:1 methanol-to-oil molar ratio for a duration of 60 minutes. To evaluate the impact of catalyst loading on biodiesel yield, the catalyst was varied at 0.5%, 1%, 1.5%, 2%, 2.5%, and 3% (w/w) relative to the oil mass.

Throughout the reaction, continuous stirring ensured homogeneous mixing and maximized the exposure of the feedstock mixture to the heterogeneous catalyst. Post reaction, the resulting solution was left undisturbed for approximately 8 hours to achieve natural phase separation, forming a lighter biodiesel layer above a denser glycerol layer. The upper biodiesel phase was then carefully separated and rinsed using heated distilled water to effectively eliminate residual catalyst, methanol, and glycerol residues. The refined biodiesel was subsequently heated at 105 °C to evaporate any remaining water content before further characterization and analysis.

## 3 Results and discussion

### 3.1 Characterization of waste cooking oil (WCO)

Following the Indonesian National Standard (SNI) 7709:2019, the collected WCO samples were assessed to verify their suitability as biodiesel feedstock. The evaluation primarily focused on the free fatty acid (FFA) perccentage, a decisive factor in determining whether samples can be directly transesterified under basic conditions or requires prior esterification. The presence of high FFA in waste cooking oil has a significant influence on both the transesterification reaction

and the quality of the biodiesel produced. Elevated FFA levels tend to lower the conversion rate and trigger saponification during washing, while the acid and saponification values show a direct and inverse relationship with the FFA percentage, respectively [10]. The results of the free fatty acid (FFA) analysis before and after pretreatment are shown in Table 1.

**Table 1.** Free Fatty Acid (FFA) content analysis results.

| No | Name | FFA Content % | SNI 7709:2019 |
|---|---|---|---|
| 1 | Before pretreatment | 1.29 | <2% |
| 2 | After pretreatment | 0.51 | |

The experimental results revealed that the free fatty acid (FFA) content decreased from 1.29% to 0.51% after purification with activated carbon, indicating a 0.78% reduction. This result complies with the SNI 7709:2019 standard, which specifies that FFA should be below 2%, confirming the samples is viable for direct base catalyzed transesterification without the need for acid esterification. Maintaining FFA concentrations under this threshold is crucial to prevent saponification, a side reaction in which FFAs interact with alkaline agents to yield fatty acid salts. The fatty acid salts consumes the catalyst, reduces its availability for transesterification, and interferes with the distinct stratification of the methyl ester phase and the glycerol byproduct, ultimately lowering conversion efficiency and product purity [11]. Achieving an FFA concentration of 0.51% is paramount for maximizing methyl ester yield, as it minimizes the neutralization reactions between FFA and the catalyst's basic active sites, thereby preserving catalytic activity for triglyceride alcoholysis. This is particularly important under the specified operating parameters of 65 °C, molar ratio of methanol : oil is 9:1 for 60 minute reaction time[12]. The presence of low FFA content ensures that the transesterification process proceeds efficiently, leading to high conversion rates of oil into biodiesel. This finding aligns with previous reports emphasizing that controlling FFA levels below 2% is essential for achieving stable transesterification and high-purity biodiesel[13]. This optimal FFA level is supported by various studies that highlight the importance of controlling FFA content to enhance biodiesel yield and quality. Studies have shown that feedstocks with FFA content below 3% achieve high methyl ester conversion rates exceeding 90% [10].

The reactivity of waste cooking oil (WCO) in fatty acid methyl ester (FAME) synthesis is significantly influenced by its physicochemical properties, including viscosity and moisture content. High moisture content and viscosity can impede the mass transfer between immiscible reactants, leading to increased generation of fatty acid salts, which adversely affects the final fuel conversion rate and its physicochemical properties. Understanding these properties and their effects is imperative for enhancing the efficiency of the FAME synthesis workflow [14]. The activated carbon pretreatment employed in this study effectively reduced these impurities through selective adsorption, improving oil quality and ensuring compatibility with the heterogeneous CaO–$K_2O$ catalyst used in the subsequent process[5][15]. Thus, the pretreated WCO not only met the SNI 7709:2019 requirement but also provided a stable and clean feedstock for efficient biodiesel synthesis in the next stage of this work.

### 3.2 XRD characterization of CaO–$K_2O$ catalyst

X-ray diffractometry (XRD) was employed to investigate the crystallinity of the synthesized CaO–$K_2O$ composite to confirm the phase transformations occurring during the calcination process.

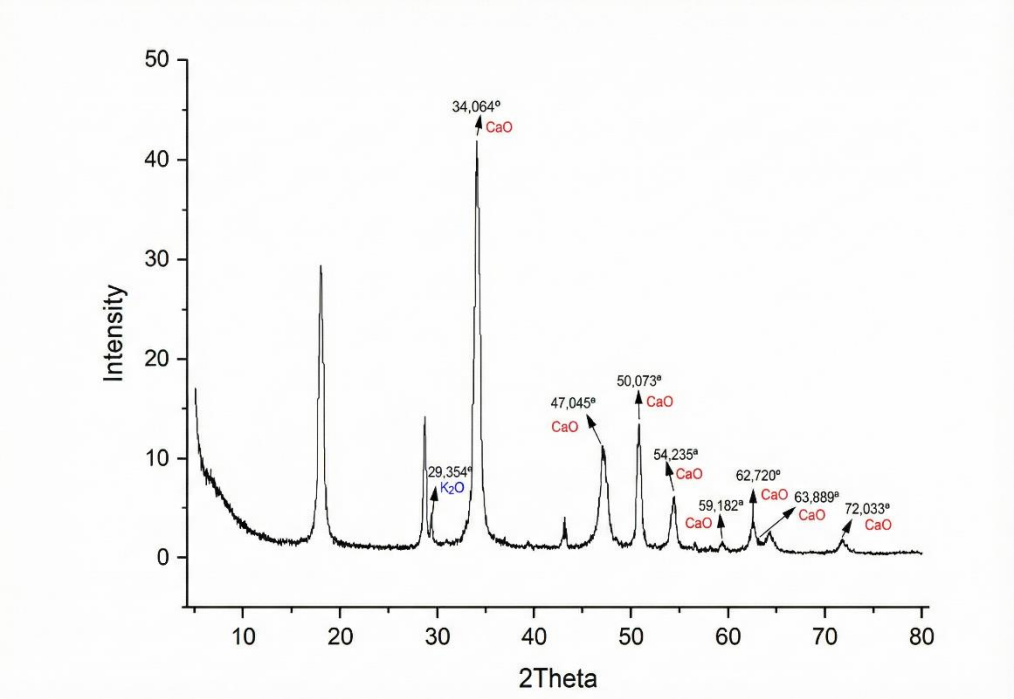


**Fig. 1.** Graph of XRD (X-ray diffraction) analysis results of CaO-K2O composite catalyst.

As ilustrated in **Figure 1** the diffraction pattern displayed prominent peaks at 2θ values of approximately 34°, 47°, 50°, 54°, 59°, 62°, and 72°. These peaks correspond to the characteristic planes of calcium oxide (CaO), indicating that the calcium carbonate ($CaCO_3$) from eggshells was thoroughly decomposed after calcination at 900 °C for 3 hours. Furthermore, minor peaks observed near 29° were attributed to potassium oxide ($K_2O$) derived from the coffee husk, which confirms the successful incorporation of alkali metal oxides into the CaO matrix. Traces of silica ($SiO_2$) and alumina ($Al_2O_3$) were also detected, most likely derived from the biomass ash, which may help improve structural rigidity and reduce sintering during catalyst reuse.

The crystalline framework observed in the XRD analysis is consistent with the primary chemical components of the catalyst, where CaO serves as the dominant phase followed by $K_2O$. This stable crystalline structure verifies that the calcination and compositing stages effectively produced a CaO-$K_2O$ heterogeneous catalyst. Similar results have been reported in previous studies, where eggshell-derived CaO catalysts calcined at 900 °C showed strong CaO diffraction peaks with high crystallinity and enhanced catalytic activity[16][17]. Furthermore, the presence of $K_2O$ has been shown to increase the number of basic sites and promote electron donation, thereby enhancing the transesterification reaction rate[18].

The coexistence of CaO and $K_2O$ crystalline phases provides synergistic catalytic effect. The CaO component contributes robust Brønsted basicity essential for methoxide ion generation, while $K_2O$ introduces additional Lewis basic sites that facilitate triglyceride activation[19]. This dual functionality supports efficient transesterification under the conditions applied in this study.

Overall, the XRD results confirm that the CaO–$K_2O$ composite catalyst possesses a well-defined crystalline structure with high potential for biodiesel synthesis. The integration of CaO and $K_2O$ not only enhances catalytic basicity and thermal stability but also demonstrates the valorization potential of eggshell and coffee husk waste as sustainable catalyst precursors.

### 3.3 FAME composition and biodiesel yield

The catalytic capability of the CaO-$K_2O$ composite was assessed by the biodiesel yield and its core physical characteristics. As illustrated in **Figure 2**, the FAME conversion derived from used frying oil varied considerably with catalyst loading. The highest yield of approximately 71.7% was achieved at the lowest catalyst mass of 0.5%. However, further increases to 1–1.5% resulted in a marked reduction in the conversion rate, reaching approximately 60% at 2% loading. This reduction is likely attributed to catalyst particle aggregation and the subsequent loss of accessible active sites. Interestingly, a modest rebound in yield occurred at 2.5–3%, though the values did not exceed the peak seen at 0.5%.

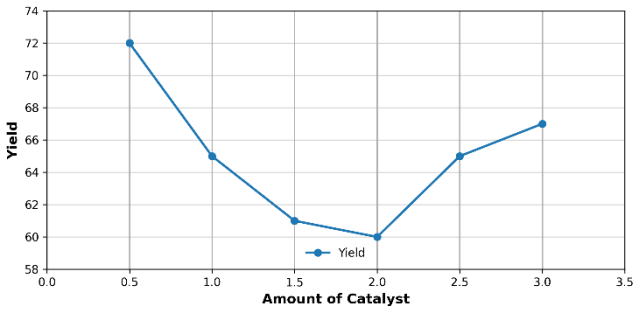


**Fig. 2.** The influence of catalyst loading on the FAME conversion rate derived from used cooking oil.

These current observations results are consistent as reported by Saleem *et al.*, who observed that heterogeneous catalysts derived from CaO exhibit maximum catalytic efficiency at lower loadings. Increasing the catalyst concentration beyond this point tends to cause surface saturation and diffusion constraints, ultimately leading to reduced product yield [20]. A comprehensive review by Yaghi (2025) further emphasises that catalyst loading must balance sufficient active site availability with minimal diffusion hindrance to maximise yield [21].

The fuel quality was further assessed through heating value and density analyses. The heating value of FAME derived from waste cooking oil (WCO) was evaluated under six catalyst loadings (0.5 %, 1 %, 1.5 %, 2 %, 2.5 %, and 3 %) and the results, shown in **Figure 3**, revealed fluctuations in energy output.

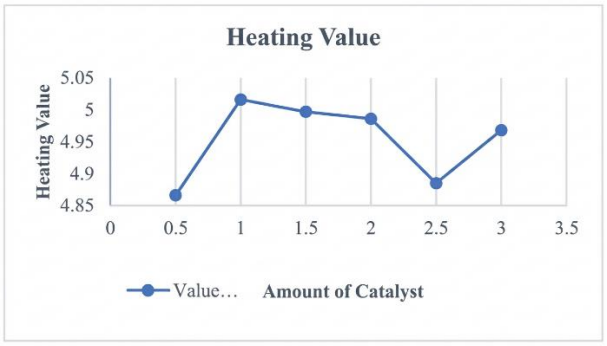


**Fig. 3.** Graph of calorific value of FAME from used cooking oil.

The highest heating value of 5.016 kcal/kg (≈ 21.0 MJ/kg) recorded at 1 % loading. This finding underscores the importance of optimizing catalyst mass to maximize energy content. Biodiesel produced from WCO achieved a heating value of 41.35 MJ/kg (~9,859 kcal/kg) under optimal conditions, reinforcing that higher energy density translates to lower fuel consumption per unit mass[22]. Furthermore, another research demonstrates that inadequate purification and the presence of residual contaminants such as moisture or unpurged glycerol substantially diminish calorific value by engendering energy losses during the combustion process[23].

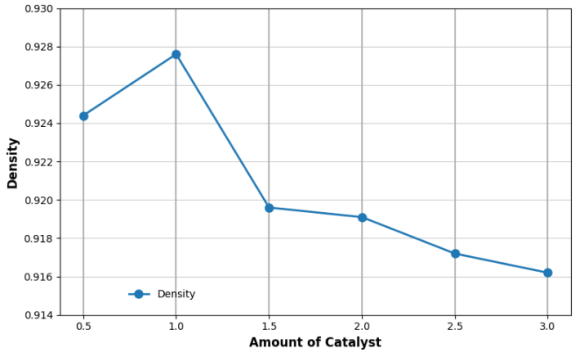


**Fig. 4.** Graph of the effect of catalyst mass on the density of FAME from waste cooking oil.

Regarding physical properties, the density of the biodiesel ranged between 0.91 and 0.92 g/mL, as shown in **Figure 3**. Higher density readings at lower catalyst loadings (0.5%–1%) may stem from insufficient transesterification or trapped glycerol, factors known to impede atomization efficiency. This observation is supported by Wahyudi et al., who identified a direct correlation between higher densities in WCO-biodiesel blends and increased fuel consumption[24]. Furthermore, Yadav et al. compiled a large dataset (n = 2.389) for WCO-derived biodiesel and emphasised that density exceeding ~0.88 g/mL strongly correlates with poorer spray characteristics and higher emissions [25]. Given that all samples in this work exceed typical biodiesel density standards (~0.88 g/mL), their application in conventional diesel engines may be limited

without further purification or blending optimisation to improve fuel injection, atomisation and engine durability.

## 4 Conclusion

This work successfully utilized a composite $CaO–K_2O$ catalyst synthesized from eggshells and coffee husks to drive the transesterification of waste cooking oil. Based on XRD analysis, the material developed the active oxide phases necessary for the reaction. Experimental results identified 0.5 wt% as the most effective catalyst loading, yielding a peak FAME conversion of 71.7%, whereas higher dosages caused a drop in efficiency. Although the fuel retained a heating value of 5,016 kcal/kg, the recorded density (0.91–0.92 g/mL) suggests that post-reaction purification is required to meet commercial standards. Ultimately, this study offers a viable strategy for valorizing residue materials into renewable energy.

**Acknowledgment.** The authors would like to thank the Faculty of Science and Technology, Universitas Jambi for the facilities and support provided to conduct this research.

## References

[1] Yunitasari, B., Susila, I.W., Arsana, I.M., Drastiawati, N.S.: The Production of Waste Cooking Oil Biodiesel by Non-Catalytic Methods. IOP Conference Series: Earth and Environmental Science. Vol. 1359, p. 012036 (2024)
[2] Pilicita, J., Domínguez, J.A., Torresano, C., Salazar, B.: Analysis of the use of waste cooking oil as an alternative fuel. Multidisciplinar. Vol. 3, p. 204 (2025)
[3] Marchetti, J.M., Miguel, V.U., Errazu, A.F.: Heterogeneous esterification of oil with high amount of free fatty acids. Fuel. Vol. 86, pp. 906–910 (2007)
[4] Kusumaningtyas, R.D., et al.: Optimisation of Free Fatty Acid Removal in Nyamplung Seed Oil (Callophyllum inophylum L.) using Response Surface Methodology Analysis. Pertanika J. Sci. Technol. Vol. 29 (2021)
[5] Tiong, A.N.: Pre-treatment of waste cooking oil by combined activated carbon adsorption and acid esterification for biodiesel synthesis via two-stage transesterification. Biofuels. pp. 1–11 (2023)
[6] Heravi, M.M., Bamoharram, F.F.: Applications of heteropoly acids as heterogeneous catalysts. In: Heterogeneous Catalysis in Sustainable Synthesis. pp. 141–203. Elsevier (2022)
[7] Basumatary, S.F., et al.: Advances in CaO-Based Catalysts for Sustainable Biodiesel Synthesis. Green Energy Resour. (2023)
[8] Flores, J., et al.: Novel synthesis and characterization of bio-based hydroxyapatite obtained from a residue of eggshells. J. Appl. Res. Technol. Vol. 22, pp. 518–526 (2024)
[9] Hoseini, M., Cocco, S., Casucci, C., Cardelli, V., Corti, G.: Coffee by-products derived resources. A review. Biomass Bioenergy. Vol. 148, p. 106009 (2021)
[10] Maniam, G.P., Hussin, N.M.: Effect of Free Fatty Acid on Transesterification of Waste Cooking Oil. Chemical Science and Technology. Vol. 3 (2023)
[11] Chanakaewsomboon, I., Phoungthong, K., Palamanit, A., Seechamnanturakit, V., Cheng, C.K.: Biodiesel produced using potassium methoxide homogeneous alkaline catalyst: effects of various factors on soap formation. Biomass Convers. Biorefinery. pp. 1–11 (2021)

[12] Encinar, J.M., González, J.F.G., Martínez, G., Nogales-Delgado, S.: Use of NaNO3/SiAl as Heterogeneous Catalyst for Fatty Acid Methyl Ester Production from Rapeseed Oil. Catalysts. Vol. 11, p. 1405 (2021)
[13] Maheshwari, P., et al.: A review on latest trends in cleaner biodiesel production: Role of feedstock, production methods, and catalysts. J. Clean. Prod. Vol. 355, p. 131588 (2022)
[14] Chanakaewsomboon, I., Moollakorn, A.: Soap Formation in Biodiesel Production: Effect of Water Content on Saponification Reaction. International Journal of Civil, Architectural and Environmental Sciences. (2016)
[15] Selvi, S.T., Krishna, N., Jeevanantham, G., Abhishek, R.: Adsorptive Removal of Impurities from Transformer oil using Activated carbon. In: 2024 International Conference on Advances in Electrical, Computing, Communication and Sustainable Technologies (ICAECT). pp. 1–8. IEEE (2024)
[16] Linggawati, A.: Preparation and Characterization of Calcium Oxide Heterogeneous Catalyst Derived from Anadara Granosa Shell for Biodiesel Synthesis. KnE Engineering. Vol. 1 (2016)
[17] A. Buasri, J. Kamsuwan, J. Dokput, P. Buakaeo, P. Horthong, and V. Loryuenyong, "Green synthesis of metal oxides (CaO-K2O) catalyst using golden apple snail shell and cultivated banana peel for production of biofuel from non-edible Jatropha Curcas oil (JCO) via a central composite design (CCD)," J. Saudi Chem. Soc., 2024, doi: 10.1016/j.jscs.2024.101836.
[18] Istadi, I., Prasetyo, S.A., Nugroho, T.S.: Characterization of K2O/CaO-ZnO Catalyst for Transesterification of Soybean Oil to Biodiesel. Procedia Environ. Sci. Vol. 23, pp. 394–399 (2015)
[19] Ortega, M.B., Hurtado, A.C., Duarte, S.A.G.: Triglyceride transesterification in heterogeneous reaction system with calcium oxide as catalyst. Rev. Fac. Ing. Antioquia. No. 57, pp. 7–13 (2011)
[20] Saleem, M., et al.: Enhancing the Catalytic Activity of Eggshell-Derived CaO Catalyst and Its Application in Biodiesel Production from Waste Chicken Fat. Catalysts. Vol. 12, p. 1627 (2022)
[21] Mazaheri, H., et al.: An Overview of Biodiesel Production via Calcium Oxide Based Catalysts: Current State and Perspective. Energies. Vol. 14, p. 3950 (2021)
[22] Mohan, B., Yang, W., Tay, K.L., Yu, W.: Experimental study of spray characteristics of biodiesel derived from waste cooking oil. Energy Convers. Manag. Vol. 88, pp. 622–632 (2014)
[23] Adhikesavan, C., Ganesh, D., Augustin, V.C.: Effect of quality of waste cooking oil on the properties of biodiesel, engine performance and emissions. Clean. Chem. Eng. Vol. 4, p. 100070 (2022)
[24] Wahyudi, W., Nadjib, M., Apriyanto, A.: Correlation between Physical Properties and Specific Fuel Consumption in Jatropha -Used Cooking Oil Biodiesel Mixtures. Semesta Tek. Vol. 26 (2023)
[25] Pratas, M.J., Freitas, S.V.D., Oliveira, M.B., Monteiro, S., Lima, Á.S., Coutinho, J.A.P.: Biodiesel Density: Experimental Measurements and Prediction Models. Energy & Fuels. Vol. 25, pp. 2333–2340 (2011)