RESEARCH PROGRESS IN MECHANICAL AND MANUFACTURING ENGINEERING
e-ISSN: 2773-4765

RPMME

Vol. 6 No. 1 (2025) 387-392
https://publisher.uthm.edu.my/periodicals/index.php/rpmme

# Study of Cao-$TiO_2$ Catalyst Calcination Ratios for the Synthesis of Biodiesel

**Muhammad Naqib Zainul Abidin[1], Nurul Fitriah Nasir[1]***

[1] *Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, Parit Raja, 86400, MALAYSIA*

*Corresponding Author: fitriah@uthm.edu.my
DOI: https://doi.org/10.30880/rpmme.2025.06.01.042

**Article Info**

Received: 2 February 2025
Accepted: 24 April 2025
Available online: 31 July 2025

**Keywords**

Biodiesel, catalyst, ratio, palm oil, transesterification

**Abstract**

This study explores how varying CaO catalyst ratios affect the production of biodiesel and its physicochemical qualities. The study employs X-ray diffraction (XRD), scanning electron microscopy (SEM), and Fourier transform infrared spectroscopy (FTIR) to characterize the catalysts, and evaluates the density, kinematic viscosity, and yield of the resulting biodiesel. The findings indicate that the biodiesel's density (0.89 g/cm$^3$ at 15°C and 0.90 g/cm$^3$ at 40°C) and kinematic viscosity (3.7 mm$^2$/s at 40°C) meet ASTM D6751 standards, but the yield is relatively low at 26%. The FTIR analysis shows significant interactions between CaO and $TiO_2$, particularly at the 1:0.25 ratio, which shows a strong carbonate peak. SEM analysis reveals various degrees of particle agglomeration and porosity, which influences catalyst effectiveness. The study shows that optimising reaction conditions and improving catalyst formulations are essential for enhancing biodiesel yield and efficiency.

## 1. Introduction

The exploitation of the world's petroleum reserves and the environmental depletion due to the unsustainable use of fossil fuels have led to the search for alternative and eco-friendly energy resources [1]. The continuing increase of crude oil prices has a direct effect on many business sectors, especially transportation [2]. Fossil fuels, including petroleum, coal, and natural gas, are the primary energy sources. Fossil fuels provide 80% of the world's energy needs. Most industries rely on diesel-powered machinery for manufacturing. By 2030, the dependence on renewable energy is expected to increase from 24 to 77 %, where the transportation sector will face the highest demand, mainly for diesel [3]. This condition makes daily living highly dependent on fossil fuels. Fossil oils are fuels derived from ancient animals and microorganisms. Fossil fuel creation takes millions of years. Thus, fossil oils belong to non-renewable energy sources [4]. In this respect, biodiesel is an emerging alternative to diesel fuel derived from renewable and locally available resources which is biodegradable, nontoxic and environmentally friendly [5].

Biodiesel is a promising substitute for fossil diesel fuel because its properties are quite similar to diesel [2], where it derived from bio-oils obtained mainly from plants and their derivatives, is a renewable and perishable resource that offers several advantages over fossil fuels [6]. Unlike fossil fuels, which release harmful greenhouse gases when burned, biodiesel is a clean-burning fuel that significantly reduces carbon dioxide, particulate matter, and sulphur emissions [7]. Biodiesel mainly consists of fatty acid methyl esters (FAME) formed during the transesterification reaction in the presence of an alkali catalyst such as NaOH or KOH, mild conditions such as 60 °C, and atmospheric pressure [8]. Hence, catalyst used plays a crucial role in the reaction's efficiency and sustainability.

Catalysts can be divided into two main types, homogeneous [9], and heterogeneous [10]. A significant disadvantage of homogeneous basic catalysts is their low reusability. Furthermore, these catalysts are difficult to recover and reuse, resulting in higher catalyst consumption and waste production [11]. On the other hand, heterogeneous catalysts provide a variety of advantages over their homogeneous counterparts. They are easily extracted from the reaction mixture using basic separation techniques such as filtering or centrifugation, avoiding the need for further purification processes. In this research, heterogeneous catalyst method will be used because it may be recycled and used several times, resulting in reduced catalyst consumption and waste generation [12].

## 2. Methodology

### 2.1 Materials

Vegetable palm oil (Avena) was purchased from local market in Parit Raja, Batu Pahat, Johor. Calcium oxide (CaO) with a purity of 96%, titanium dioxide ($TiO_2$) with a purity of 99% and methanol of analytical reagent (AR) grade were acquired from fuel analysis laboratory UTHM, Malaysia. Distilled water was used throughout the experiments.

### 2.2 Catalyst Preparation

The CaO and $TiO_2$ were dry mixed at mixing ratios of 2:1, 3:1, 4:1, and 5:1 to form CaO-$TiO_2$ catalysts where CaO are 112g, 168g, 224g and 280g, and $TiO_2$ ratio is constant (80g). Since the materials for the catalyst are limited, the ratios scales are reduced to 1:0.5, 1:0.3333, 1:0.25, and 1:0.2, where $TiO_2$ are 40g, 27g, 20g and 16g, and CaO is constant (56g). All catalyst ratios went to calcination process in a furnace at temperature of 600℃ for 5 hours. After calcination, the catalysts were ground to a fine powder using planetary ball mill machine. The duration of milling and speed rotation are set to 15 minutes and 100 rpm respectively. Then, the ground catalysts are sieved (63 μm) and stored in PP container with lid.

### 2.3 Transesterification Process

Transesterification process is used to produce biodiesel. This process was carried out by mixing 10 wt% 1:0.25 CaO-$TiO_2$ for 1 hour while stirring at ±300 rpm. The mixed catalyst-methanol to palm oil molar ratio of 9:1 were tested in 3-neck glass flux with a reaction time of 120 minutes at constant temperature, 65℃ and stirring speed, ±500rpm on a hot plate. The separation process began once the transesterification reaction had finished and was left overnight. Filtration and washing were then required to separate the biodiesel and glycerin.

## 3. Result and Discussion

### 3.1 X-ray Diffraction (XRD) Analysis

XRD analysis is to study the catalyst crystal structure. XRD patterns of all catalyst ratios were recorded and analyzed. From Fig. 1, it can be observed that $TiO_2$ is the major component of the CaO-$TiO_2$. Catalyst ratios of 1:0.2 and 1:0.3333 shows significant changes in phase composition. In the 1:0.2 ratio, there is a significant presence of Portlandite ($Ca(OH)_2$) with an intensity (Y value) of 116.52%, showing significant CaO hydration, and a high intensity of anatase TiO2 (Y: 183.24%), indicating successful integration. In comparison, the 1:0.3333 ratio shows higher unreacted CaO (Y: 35.93%) and lower Portlandite (Y: 70.29%), indicating less hydration and more active CaO available for catalytic processes, with anatase TiO2 also well-distributed (Y: 139.21%). These results indicate that the 1:0.3333 ratio, with its higher active CaO and significant anatase $TiO_2$ presence, might offer better catalytic performance and stability in biodiesel production compared to the 1:0.2 ratio, where excessive hydration may reduce efficiency.

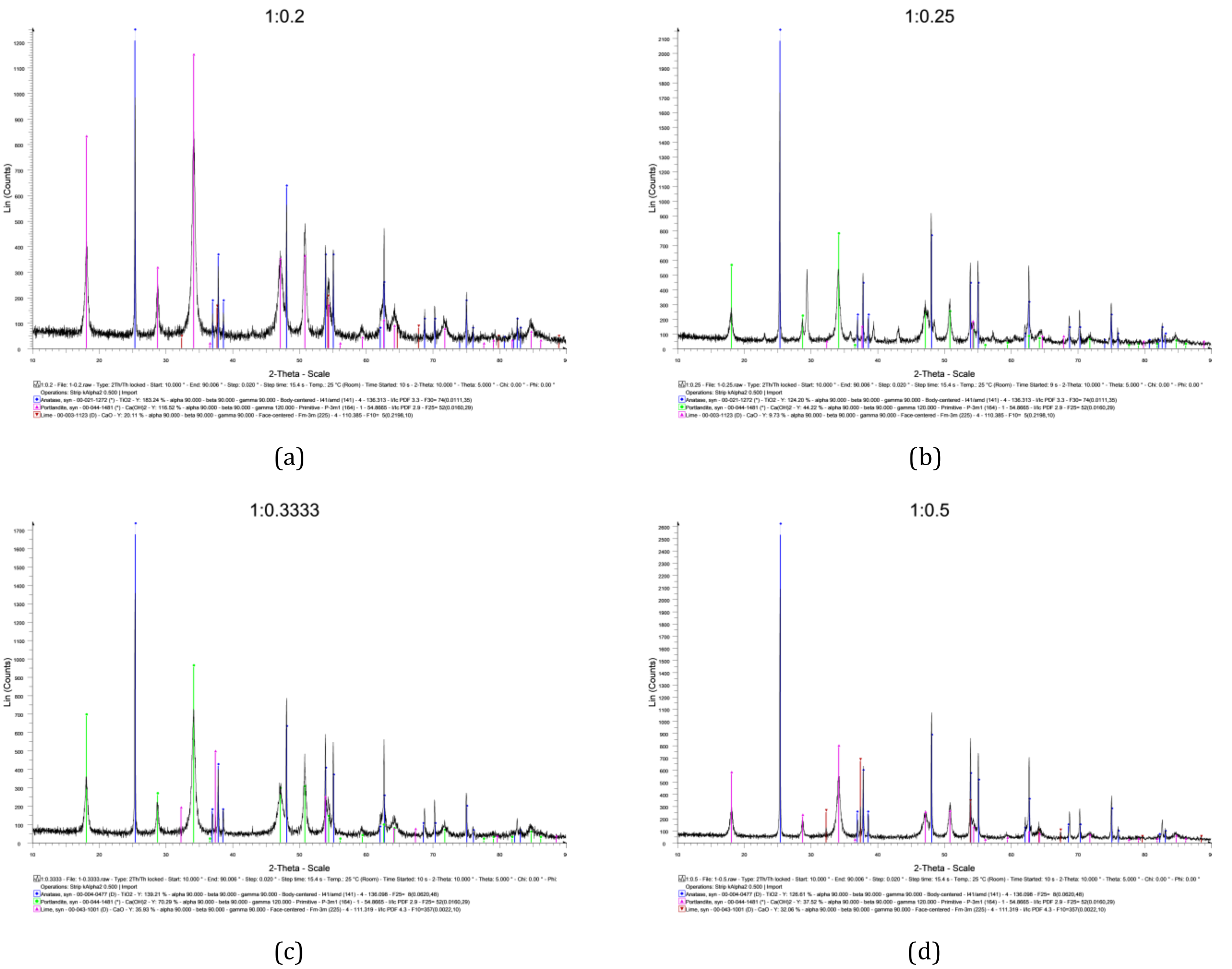


**Fig. 1** *XRD analysis for CaO-$TiO_2$ catalysts: (a) 1:02, (b) 1:0.25, (c) 1:0.3333, (d) 1:0.5*

## 3.2 Scanning Electron Microscopy (SEM) Analysis

SEM analysis of the CaO catalyst ratios shows significant differences in particle distribution, agglomeration, and porosity. For the 1:0.2 ratio, the SEM in Fig. 2 shows a relatively uniform distribution of particles with moderate porosity, indicating a favorable morphology for catalytic activity. As the $TiO_2$ concentration increases in the 1:0.25 ratio, particle agglomeration increases while porosity decreases slightly. This pattern continues with the 1:0.3333 ratio, where significant agglomerates and decreased porosity are observed, potentially impacting the catalyst's effectiveness. The 1:0.5 ratio shows major agglomeration and the least porosity, which could substantially reduce the active surface area available for catalysis. Overall, while increasing $TiO_2$ concentration improves mechanical stability, it also causes increased particle clustering and decreased porosity, which reduces catalyst efficiency. Thus, the 1:0.2 ratio appears to offer the best balance of uniform distribution and porosity for effective catalytic performance in biodiesel production.

## 3.3 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

This analysis shows a clear pattern of increasing $TiO_2$ presence and decreasing hydration as the $TiO_2$ content rises. For the 1:0.2 ratio, the spectrum displays balanced characteristics with notable O-H and Ti-O-Ti vibrations, indicating a mix of CaO and $TiO_2$ with some hydration. As the ratio increases to 1:0.25 and 1:0.3333, the intensity of Ti-O-Ti peaks grows, indicating increased $TiO_2$ concentration and interaction with CaO, whereas O-H peaks from $Ca(OH)_2$ remain present but less intense. The 1:0.5 ratio contains the greatest $TiO_2$ concentration, with significant Ti-O-Ti peaks and minimal hydration, indicating $TiO_2$ as the main phase. This development highlights the increased $TiO_2$ presence and decreased $Ca(OH)_2$, highlighting the chemical interactions and structural evolution critical for optimizing catalyst performance in biodiesel production.

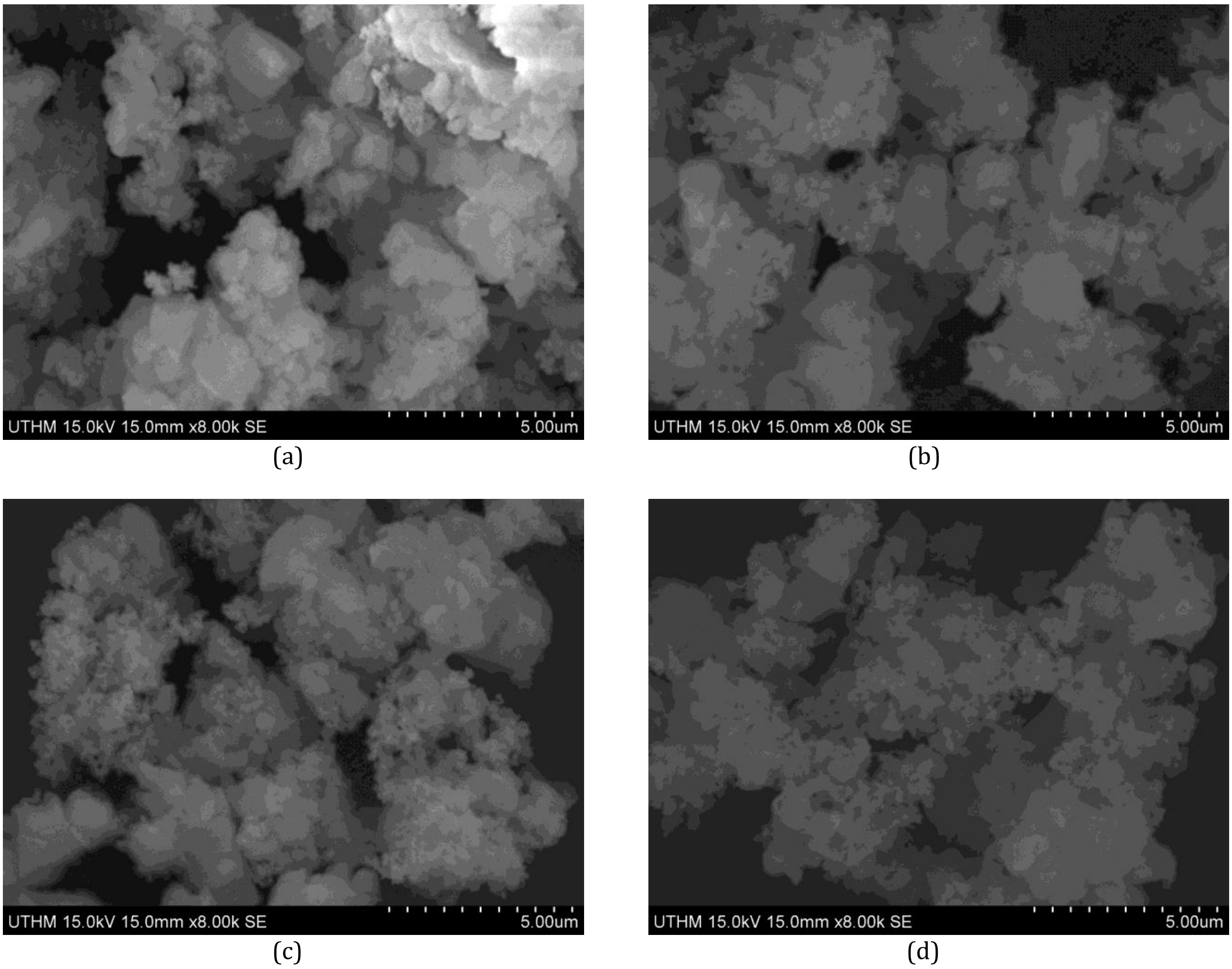


**Fig. 2** *SEM analysis for CaO-TiO₂ catalysts: (a) 1:02, (b) 1:0.25, (c) 1:0.3333, (d) 1:0.5*

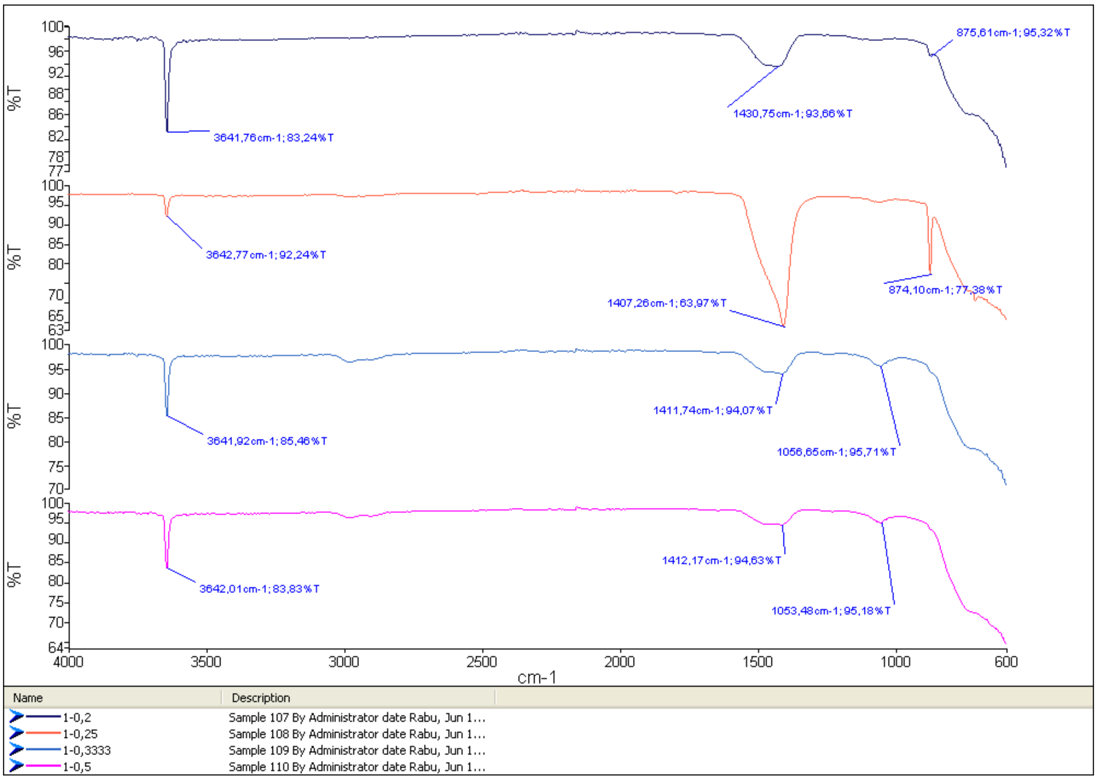


Fig. 3 FTIR analysis for *CaO-TiO₂ catalysts*

### 3.4 Biodiesel Properties

**Table 1** *Compassion between ASTM D6751 and 1:0.25 CaO-TiO₂ catalyst*

| Standard/Sample | 1:0.25 CaO-$TiO_2$ | ASTM D6751 |
|---|---|---|
| Density (15°C, g/cm³) | 0.89 | 0.86 – 0.90 |
| Density (40°C, g/cm³) | 0.90 | 0.86 – 0.90 |
| Kinematic Viscosity (40°C, mm²/s) | 3.7 | 1.9 – 6.0 |
| Yield (%) | 26 | 95 – 100 |

The biodiesel produced has a density of 0.89 g/cm³ at 15°C and 0.90 g/cm³ at 40°C. This fits within the normal range of 0.86 to 0.90 g/cm³, which indicates acceptable quality. The kinematic viscosity at 40°C is 3.7 mm²/s, which is within the ASTM D6751 range of 1.9 to 6.0 mm²/s. This ensures suitable flow characteristics for engine performance. The absence of viscosity measurement at 15°C is consistent with standard practices, as ASTM D6751 focuses on 40°C measurements, and lower temperature viscosities are less critical for operational performance. However, the biodiesel yield is relatively low at 26%, suggesting a need for optimization in reaction conditions, such as mixing, reaction time, catalyst concentration, or feedstock purity, to enhance production efficiency and economic viability.

## 4. Conclusion

This research explored the production of biodiesel using varying CaO catalyst ratios, focusing on the influence of these ratios on the physicochemical properties of the resulting biodiesel. The study included a comprehensive analysis of the catalyst characteristics through XRD, SEM, and FTIR techniques, and evaluated the biodiesel's density, kinematic viscosity, and yield. The FTIR and XRD tests showed considerable changes in the catalysts' chemical interactions and phase compositions, while SEM study revealed varied degrees of particle agglomeration and porosity at different ratios. The results indicated that while the density and kinematic viscosity of the biodiesel met the ASTM D6751 standards, the yield was relatively low at 26%, pointing to potential inefficiencies in the production process. Nevertheless, there are recommendations to be made to improve the overall of this study. The recommendations for future study are:

- Optimizing reaction parameters such as catalyst concentration, reaction duration, and temperature to increase biodiesel yield. Fine-tuning these factors may result in more efficient transesterification and better conversion rates.

- Using different catalyst compositions or adding more co-catalysts may enhance catalytic activity and stability, potentially leading to larger yields and improved biodiesel quality.

- Employing more advanced characterization techniques, such as TEM (Transmission Electron Microscopy) and BET (Brunauer-Emmett-Teller) surface area analysis, can provide deeper insights into the catalyst's structural and surface properties.

## Acknowledgement

The authors would like to express their sincere gratitude to the Faculty of Mechanical and Manufacturing Engineering, Universiti Tun Hussein Onn Malaysia, for providing invaluable technical support and access to essential facilities that made this research possible.

## References

[1] A. S. M. I. S. H. &. M. M. R. Anr, "Biodiesel production from Crude Jatropha oil using a highly active heterogeneous nanocatalyst by optimizing transesterification reaction parameters," *Energy Fuel*, pp. 334-343, 2016.

[2] S. I. A. L. &. S. J.-I. P. Intarapong, "Biodiesel production from palm oil using potassium

hydroxide loaded on ZrO2 catalyst in a batch reactor," *Chiang Mai J. Sci,* pp. 128-137, 2014.

[3] J. M. S.N. Gebremariam, "Biodiesel production technologies: Review," *Aims Energy,* pp. 425-457, 2017.

[4] H. Z. &. L. L. D. Huang, "Biodiesel: an Alternative to Conventional Fuel," *Energy Procedia 16,* pp. 1874-1885, 2012.

[5] L. K. &. D. D. J. Boro, "Transesterification of non edible feedstock with lithium incorporated egg shell derived CaO for biodiesel production," *Fuel Processing Technology,* pp. 72-78, 2014.

[6] S. P. R. H. B. T. A. S. R. Manurung, "Synthesis of nano-CaO catalyst with SiO2 matrix based on palm shell ash as catalyst support for one cycle developed in the palm biodiesel process," *Case Stud. Chem. Environ. Eng.,* p. 100345, 2023.

[7] A. H. K. Masera, "Advancement of biodiesel fuel quality and NOx emission control techniques," *Renew. Sustain. Energy Rev. 178,* p. 113235, 2023.

[8] S. S. M. S. F. D. I. F. H. O. M. C. &. T. M. M. Mofijur, " Effect of nanocatalysts on the transesterification reaction of first, second and third generation biodiesel sources- A mini-review," *Chemosphere 270,* p. 128642, 2021.

[9] T. E. V. Aslan, "Polynomial regression method for optimization of biodiesel production from black mustard (Brassica nigra L.) seed oil using methanol, ethanol, NaOH, and KOH," *Energy, 209,* p. 118386, 2020.

[10] E. A.-S. D. B. E. R. M. G. F. A. M.M. Naeem, "Single-stage waste oil conversion into biodiesel via sonication over bio-based bifunctional catalyst: optimization, preliminary techno-economic and environmental analysis," *Fuel, 341,* p. 127587, 2023.

[11] D. R. A. J. S. M. T. K. S.S. Karkal, "Production optimization, scale-up, and characterization of biodiesel from marine fishmeal plant oil using Portunus sanguinolentus crab shell derived heterogeneous catalyst," *Biocatal. Agric. Biotechnol., 47,* p. 102571, 2023.

[12] B. S. H. E. Y. V. S. T. S. S. B. Maleki, "Transesterification of waste cooking oil to biodiesel by walnut shell/sawdust as a novel, low-cost and green heterogeneous catalyst: optimization via RSM and ANN," *Ind. Crops Prod., 193,* p. 116261, 2023.