Chapter 30

Transesterification Optimization of Vhembe Macadamia Nuts Oil into Biodiesel Using Inorganic KOH Catalyst

Mamookho Elizabeth Makhatha[1*], Malekgatle Linah Mojapelo[1], Patrick Ehi Imoisili[2], Tien-Chien Jen[2], and Boikanyo Diseko[3]

[1]Department of Metallurgical Engineering, University of Johannesburg, Doornfontein Campus, Johannesburg, South Africa
[2]Department of Mechanical Engineering Science, University of Johannesburg, Johannesburg, South Africa
[3]Department of Chemical Science, University of Johannesburg, Doornfontein Campus, Johannesburg, South Africa

Abstract

This study investigates the optimization of biodiesel production from macadamia nut oil (MNO) using potassium hydroxide (KOH) catalyst and methanol via transesterification. Response Surface Methodology (RSM) with a Box–Behnken design (BBD) was applied to optimize the influence of reaction temperature, alcohol-to-oil molar ratio, and catalyst weight on biodiesel yield. Experimental results showed that optimal conditions (alcohol-to-oil ratio of 6:1, temperature of 60 °C, and 1 wt.% catalyst) produced a maximum yield of 91%. Physicochemical analysis revealed that the properties of the biodiesel, including viscosity, flash point, and cetane number, met ASTM D6751 specifications, confirming successful transesterification. Gas chromatography–mass spectrometry (GC-MS) and Fourier transform infrared spectroscopy (FTIR) further validated the conversion of macadamia nut oil to biodiesel. The findings highlight the potential of macadamia nut oil as a viable feedstock for sustainable biodiesel production.

Keywords: *Macadamia nut oil, KOH catalyst, Methanol, Biodiesel, Physicochemical properties, Reaction parameters*

Introduction

The global transition towards sustainable energy is driven by increasing energy demand, climate change concerns, and the depletion of fossil fuel reserves. Fossil-based diesel remains the dominant transportation fuel worldwide, yet its contribution to greenhouse gas emissions and dependency on finite resources highlights the urgent need for renewable alternatives. Biodiesel has therefore emerged as a promising renewable substitute due to its biodegradability, non-toxic nature, reduced exhaust emissions, and compatibility with conventional diesel engines[1]. Over the past two decades, research has

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[1] L. C. Meher, et al., "Technical Aspects of Biodiesel Production by Transesterification - A Review," *Renewable and Sustainable Energy Reviews*, 10, no. 3 (2006): 248–268, https://doi.org/10.1016/j.rser.2004.09.002.

DOI: 10.18502/rq457c78_30

focused on exploring diverse feedstocks such as vegetable oils, animal fats, and waste cooking oils for biodiesel production. However, issues such as feedstock cost, competition with food sources, and variable physicochemical properties remain challenges in scaling up biodiesel.

South Africa presents unique opportunities for biodiesel development due to its rich agricultural base, large rural economy, and government policies that encourage renewable energy policies. One underutilized yet abundant agricultural product is macadamia nut oil (MNO), particularly in Limpopo Province where macadamia farming is well established. Unlike staple oilseeds, macadamia is not a major dietary component, and non-premium nuts and processing by-products can be utilized, minimizing competition with food resources. MNO's high oleic acid content makes it chemically stable and suitable for transesterification[2]. Furthermore, the potential for scaling production through continuous reactor systems and modular plant designs supports high-throughput processing and decentralized energy generation. This study investigates the optimization of biodiesel production from MNO using potassium hydroxide (KOH) catalyst and evaluates the fuel properties of the resulting biodiesel against international standards.

Materials and Methods

Macadamia nuts were sourced from Tshakhuma market in Limpopo Province. The oil was extracted using Soxhlet extraction with n-hexane as solvent. In practice, the Soxhlet method provides high extraction efficiency since it continuously washes the biomass with hot solvent, ensuring near-complete recovery of oil. The extraction process typically lasted six hours with a solvent-to-nut ratio of 10:1, producing a golden-yellow oil that was subsequently dried to remove residual solvent.

The transesterification reactions were carried out with methanol and inorganic KOH catalyst under varying alcohol-to-oil molar ratios (2:1–12:1), temperatures (50–70°C), and catalyst loads (1–5 wt.%). The significance of alcohol-to-oil molar ratios in driving transesterification forward has been emphasized in prior studies, since excess alcohol improves reaction completion but may complicate separation[3]. To optimise the combined influence of these factors, Response Surface Methodology (RSM) was applied using a Box–Behnken Design (BBD). This statistical design is widely employed because it requires fewer experimental runs compared to central composite designs while still allowing robust modeling of quadratic interactions[4].

Characterization of MNO and biodiesel was conducted following ASTM D6751 standards, including density, viscosity, flash point, and cetane number. The chemical conversion was confirmed using gas

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[2] Sandra L.B. Navarro & Christianne E.C. Rodrigues, "Macadamia oil extraction methods and uses for the defatted meal byproduct", *Trends in Food Science & Technology*, 54, (2016): 148-154, https://doi.org/10.1016/j.tifs.2016.04.001.

[3] Musa, Atadashi, "The effects of alcohol to oil molar ratios and the type of alcohol on biodiesel production using transesterification process," *Egyptian Journal of Petroleum* 25 (2016): 21–31, https://doi10.1016/j.ejpe.2015.06.007.

[4] Aworanti et al., "Statistical Optimization of Process Variables for Biodiesel Production from Waste Cooking Oil Using Heterogeneous Base Catalyst", *British Biotechnology Journal*, 3, no. 2, (2013): 116-132, http://doi:10.9734/bbj/2013/1381.

chromatography–mass spectrometry (GC-MS) to identify fatty acid methyl esters, and Fourier transform infrared spectroscopy (FTIR) to detect ester functional groups. These techniques provide complementary evidence, with GC-MS offering detailed compositional data and FTIR rapidly confirming chemical bond transformations[5].

The design parameters used are summarized in Table **1**.

Table 1: The experimental design parameters.

Factor	**Unit**	**Level**	
		Low	**High**
Alcohol: Oil ratio	Mol	2:1	12:1
Temperature	°C	50	70
Catalyst Amount	wt.%	1	5

Results and Discussion

Optimal biodiesel yield of 91% was obtained at 6:1 alcohol-to-oil molar ratio, 60 °C, and 1 wt.% KOH catalyst. This compares favourably with studies on other feedstocks, such as parsley seed oil[6] and flaxseed oil[7] which reported yields in the range of 85–90% under optimized conditions. The high yield from macadamia nut oil underscores its potential as a robust feedstock. Yields decreased at higher alcohol ratios (e.g., 12:1) and higher catalyst loads, likely due to soap formation and reverse reactions, consistent with findings reported by Musa Atadash[3], shows the detailed biodiesel yields under different conditions are presented in Table **2**, while the experimental setup and model validation are shown in Figure **1** and Figure **2**[8].

Table 2: Biodiesel yield under different conditions.

Runs #	***Factor A*** **Oil: Alcohol Molar Ratio (Mol)**	***Factor B*** **Temperature (°C)**	***Factor C*** **Catalyst Weight (wt.%)**	***Response*** **Biodiesel Yield (%)**
1	12	60	1	78
2	6	70	3	69
3	6	50	3	70
4	6	60	5	76
5	9	60	3	76
6	9	60	3	78

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[5] M. A. Dubé, et al., "A Comparison of Attenuated Total Reflectance-FTIR Spectroscopy and GPC for Monitoring Biodiesel Production," *Journal of the American Oil Chemists' Society,* 81, no. 6 (2004): 599–603, https://doi.org/10.1007/s11746-006-0948-x.

[6] S. O. Bitire, et al., "Production and optimization of biodiesel from parsley seed oil using KOH as catalyst for automobiles technology," *International Journal of Advanced Manufacturing Technology*, 116 (2021): 315–329, https://doi.org/10.1007/s00170-021-07415-6.

[7] M. Danish, et al., "Optimization of process variables for biodiesel production by transesterification of flaxseed oil and produced biodiesel characterizations," *Renewable Energy,* 139 (2019): 1272–1280, https://DOI:10.1016/j.renene.2019.03.036.

[8] Bitire, et al., "Production and Optimization," 320, https://doi:10.1080/14786451.2021.1890737.

Table 2: Continued.

Runs #	*Factor A*	*Factor B*	*Factor C*	*Response*
	Oil: Alcohol Molar Ratio (Mol)	**Temperature (°C)**	**Catalyst Weight (wt.%)**	**Biodiesel Yield (%)**
7	12	70	3	75
8	9	60	1	86
9	12	60	3	82
10	9	70	5	66
11	6	60	1	91
12	9	60	3	89
13	6	50	5	74
14	9	70	1	87
15	12	60	3	76
16	9	50	1	82
17	12	60	5	61

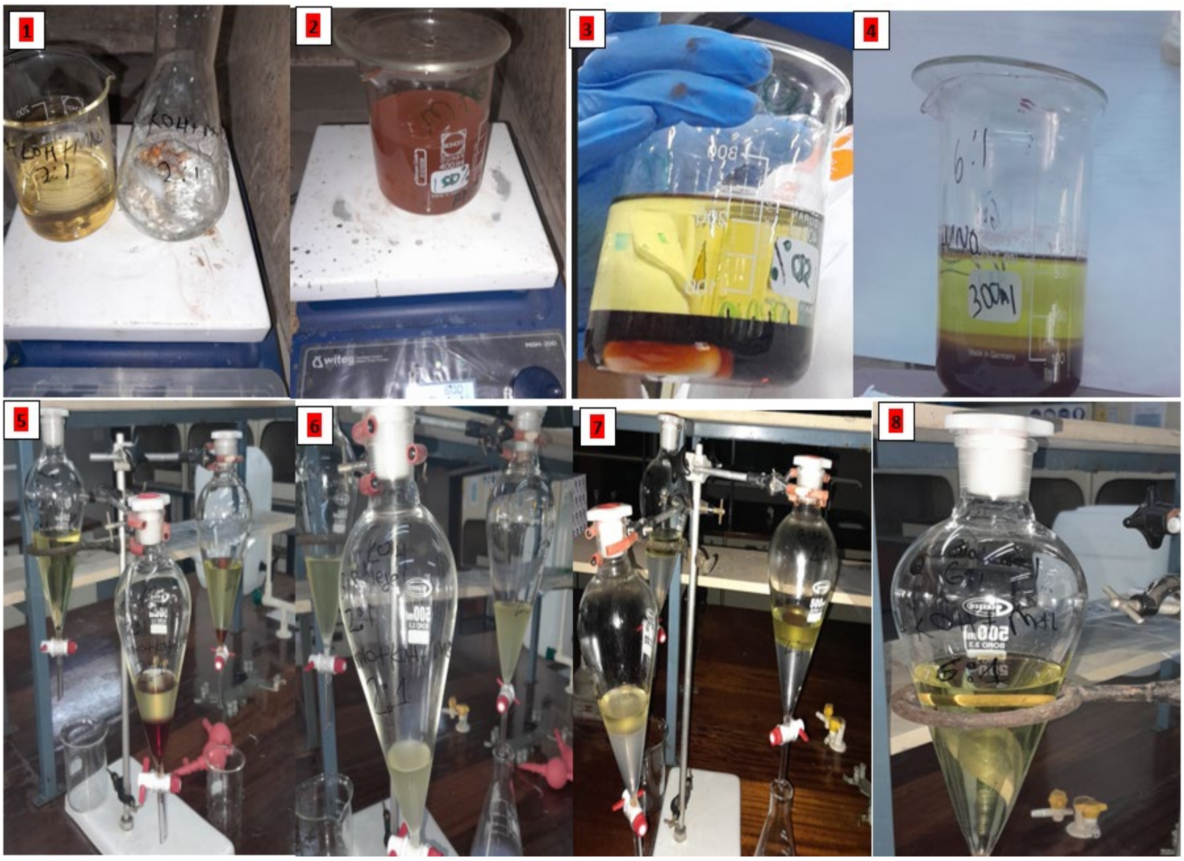

Figure 1: Synthesis and phase separation of macadamia nut biodiesel with reagent preparation and mixing (1–4), followed by phase separation of the reaction mixture (5–8).

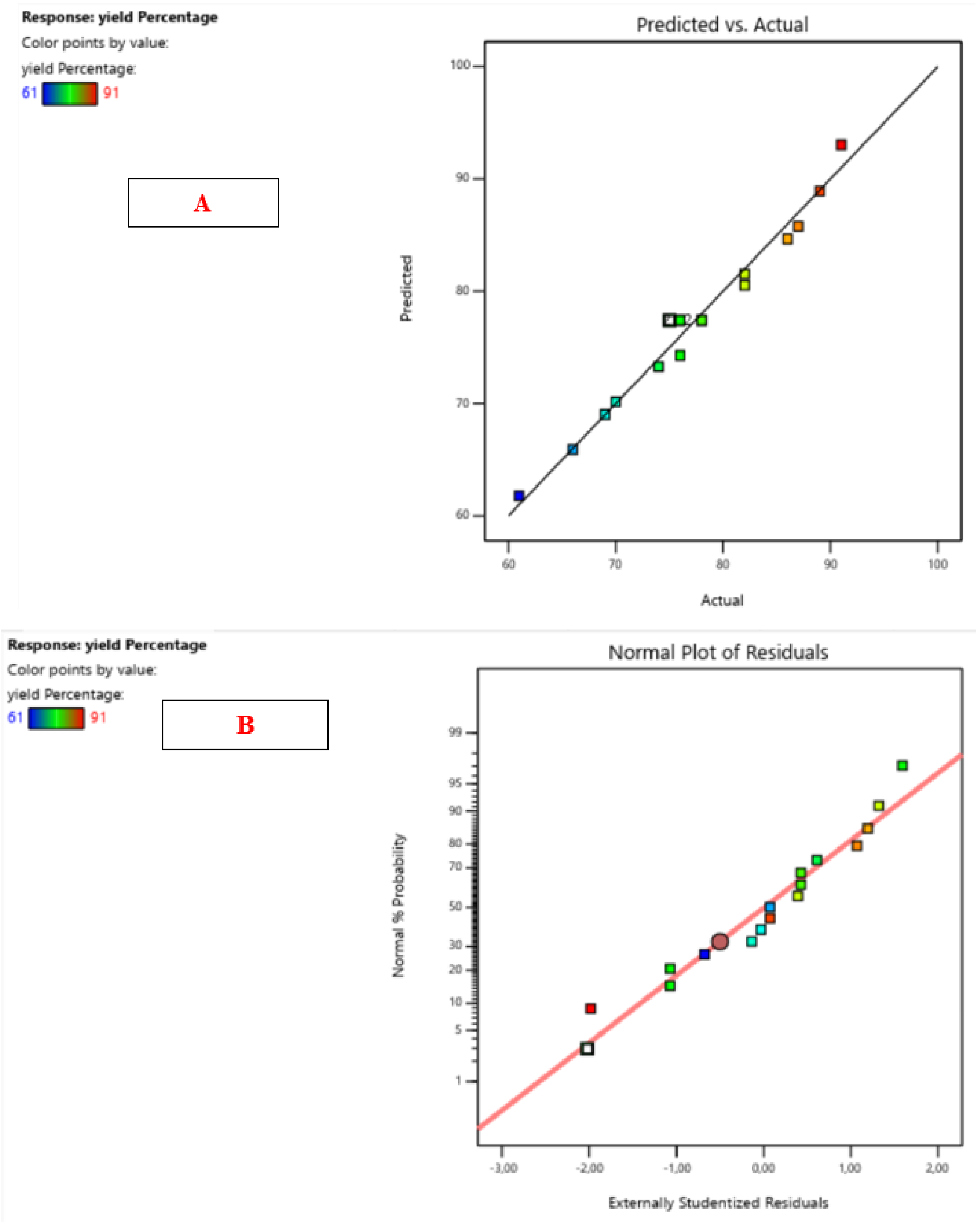

Figure 2: (A) The Predicted and Actual True Values (B) Normal Plot of Residuals (KOH).

Physicochemical analysis revealed that the biodiesel produced from MNO had properties comparable to both petroleum diesel and ASTM D6751 specifications. The viscosity of 4.07 mm^2/s falls comfortably within the ASTM range of 1.9–6.0 mm^2/s, ensuring proper fuel atomization in diesel injectors. The flash point of 143.7 °C is higher than that of conventional diesel, making the biodiesel safer to handle and transport. The cetane number of 52 is slightly above the ASTM minimum of 45, suggesting excellent ignition quality and smoother engine operation[9]. The detailed values are listed in Table **3** and Table **4**, which also highlight that the density, cloud point, and pour point of macadamia biodiesel align with acceptable ranges. These findings position macadamia biodiesel as a high-quality renewable fuel.

Table 3: Physicochemical properties.

Parameters	Values
Density at 15°C kg/m^3	869.2
Pour point (°C)	0
Acid value (mg KOH/g)	0.49
Cloud point (°C)	8
Viscosity (mm^2/sec) @ 40°C	4.07
High heating value (KJ/Kg)	40.24
pH	7,2
Specific gravity	0.878
Fire point (°C)	120.30
Flashpoint (°C)	143.70
Cetane number	52

Table 4: Physicochemical properties vs ASTM standards continued.

Parameters	Produced biodiesel	ASTM D6751	Diesel (ASTM D975)
Density	869.2	-	832
Acid value (mg KOH/g)	0.49	0.80 (max)	-
Flash point (°C)	143.70	100-170	60 to 80
Viscosity (mm^2/sec) @ 40°C	4.07	1.90 to 6.0	1.3-4.1
High heating value (KJ/Kg)	40.24	-	42
Cloud point	8	-3 to 12	-15 to 5
pH	8.7	7 to 9	-
Specific gravity	0.878	0.87 to 0.98	0.85
Fire point (°C)	120.30	-	-
Cetane number	49.97	> 45	40 – 50
Pour point (°C)	0	-15 to 13	(-35) -15

Spectroscopic analysis provided further confirmation of biodiesel formation. The GC-MS chromatogram (Figure **3**) identified key fatty acid methyl esters (FAMEs), notably oleic acid methyl ester as the dominant component, followed by palmitic and lauric acid methyl esters. Oleic acid contributes to fuel stability and higher cetane number, while palmitic acid enhances oxidative stability. The FTIR spectrum (Figure **4**) displayed characteristic ester absorption bands, particularly strong C=O stretching around 1740 cm^{-1} and C–O stretching near 1170 cm^{-1}, which are clear indicators of

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[9] G. Knothe, “Designer biodiesel: optimizing fatty ester composition to improve fuel properties,” *Energy Fuels*, 22 (2008): 1358–1364, https://doi.org/10.1021/ef700639e.

transesterification[10]. Together, these results confirm successful conversion of macadamia oil into biodiesel.

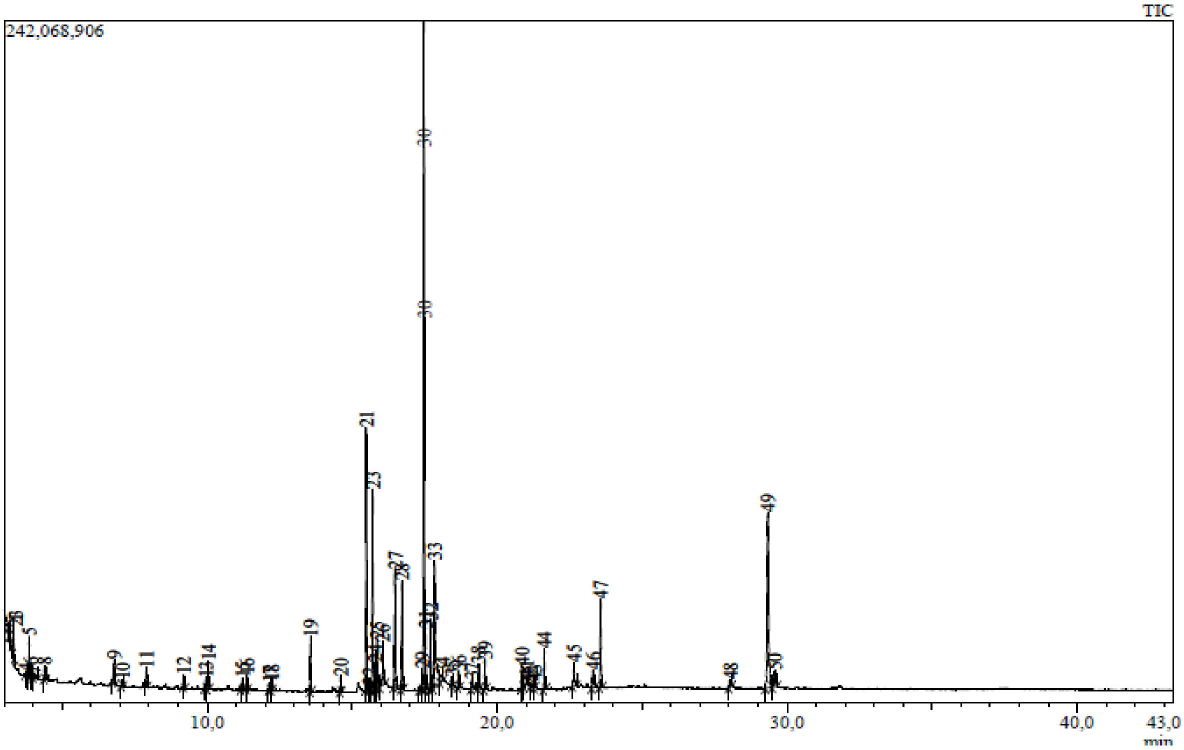

Figure 3: The GC-MS Chromatogram of KOH Biodiesel.

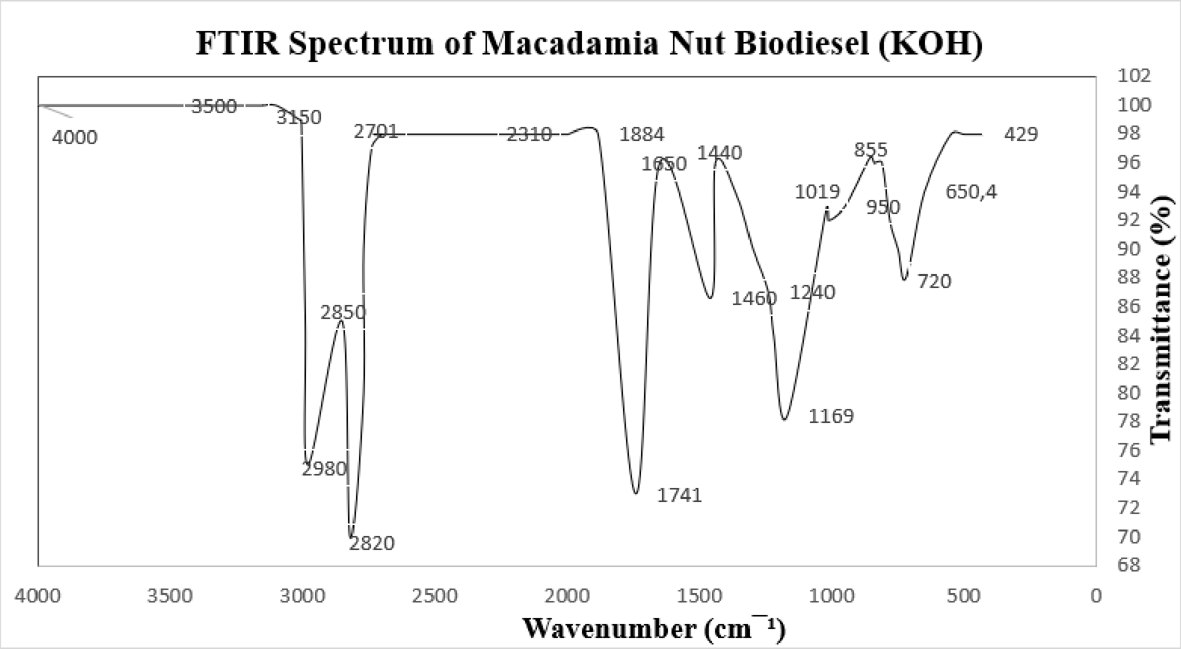

Figure 4: FTIR spectrum.

Beyond laboratory validation, these results carry broader implications. The favourable properties of macadamia biodiesel suggest potential for blending with petroleum diesel at ratios of 10–20% (B10–B20), commonly used in commercial biodiesel markets. Furthermore, South Africa's significant

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[10] Dubé et al., "A Comparison of Attenuated Total Reflectance-FTIR Spectroscopy," 600, https://doi.org/10.1007/s11746-006-0948-x.

macadamia production provides a sustainable, locally available feedstock that could support rural economic development while reducing dependence on imported fossil fuels[11].

Conclusion

This study established macadamia nut oil as a viable and high-performing feedstock for biodiesel production. Using KOH catalyst, optimal transesterification conditions yielded up to 91% biodiesel. The produced biodiesel met ASTM D6751 specifications and exhibited fuel properties comparable, and in some cases superior, to conventional diesel. These findings support the potential of macadamia biodiesel as a sustainable alternative fuel, particularly in regions where macadamia cultivation is abundant.

In addition to confirming its technical viability, the study highlights the strategic role macadamia biodiesel could play in South Africa's renewable energy mix. With increasing global emphasis on decarbonization, scaling up biodiesel production from non-food feedstocks like macadamia oil could diversify the energy portfolio while generating socio-economic benefits for rural communities. Future work should explore heterogeneous catalysts for improved recyclability, continuous transesterification processes for industrial application, and performance testing of macadamia biodiesel in diesel engines under real operating conditions.

Acknowledgements

The authors would like to thank the African Rainbow Minerals for the full study and experimental sponsorship and support. Gratitude is extended to University of Johannesburg, Department of Chemistry and Chemical Engineering for facilities.

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