



Production and evaluation of biodiesel from rambutan

(*Nephelium lappaceum* L.) seed oil

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ABSTRACT

The study aimed to search for alternative fuels that could potentially solve the gradual depletion of worldwide petroleum reserves and the increasing exhaust emissions leading to environmental hazards. Food wastes can be a potential alternative energy source that can be a substitute for petroleum-based diesel fuels, and the main focus of this study was to use rambutan seeds as the source. Various tests were performed to determine if rambutan seed oil converted into rambutan methyl ester (RME) or B100 and B2 by transesterification is an efficient source of energy, and to identify the percent yield of the extracted oil. The rambutan seed oil was also compared to coco methyl ester (CME) 100 and CME2 to determine if the extracted oil meets the specification set by the Philippine National Standard (PNS) of the Biofuel Act of 2006. The extracted crude seed oil has undergone base-catalyzed transesterification using sodium hydroxide (NaOH) as the catalyst to produce biodiesel using % by weight proportion of NaOH to oil. It was then tested to confirm the product's conformity to the PNS for biodiesel and its blend, B2. The highest oil yield gathered was a result of variation in soaking time. Emission testing for car engines was assessed for its application, with comparable results to the commercially available diesel. The results of the experiments revealed that the soaking extraction method for rambutan seed oil could produce a higher yield at its optimum contact time. Qualitative tests of physicochemical properties showed that rambutan methyl ester is comparable to the commercial coco methyl ester. The five-day contact time and weight % proportion of NaOH to oil for transesterification produced the highest % of methyl ester. The results suggest the potentiality of rambutan seed oils as local feedstock for biodiesel production and as an innovative additive to present diesel available.

Keywords: rambutan seed oil, soaking extraction method, base-catalyzed transesterification, methyl ester, biodiesel

1. Introduction

As a rural country, the Philippines is reliant on non-renewable energy assets such as fossil fuels to meet its energy needs for transporting products and maintaining a comfortable standard of living. However, non-renewable resources have a finite supply and will decline over time, which has fueled enthusiasm for biofuels as an alternative source of energy. The Philippine government has promoted the use of biodiesel, resulting in the enactment of the Philippine Biofuels Act 9637 in 2006. It has been implemented to encourage the use of biofuels requiring the mandatory mixing of a 2% biodiesel blend by volume.

The mono-alkyl esters of long-chain unsaturated fats generated from vegetable oils or animal fats make up biodiesel, a renewable fuel. Because it is non-combustible and non-lethal, biodiesel can be used as a substitute fuel in typical diesel engines while also reducing pollutants at the source [1]. In the Philippines, coconut oil is the most well-known source of biodiesel. However, rambutan seeds have been gaining interest in research as potential sources of biodiesel because of their high lipid content.

Rambutan (*Nephelium lappaceum* L.) is a medium-sized tropical tree that is native to Malay-Indonesian regions and is closely related to other edible tropical fruits such as lychee, longan, and mamoncillo [2].

According to [3] and [4], rambutan seeds produce oil at a rate of between 30% and 43%. Rambutan seeds are frequently discarded as waste, making it worthwhile to investigate their conversion to a commercially available fuel substitute.

The study primarily aimed to produce and evaluate the biodiesel produced from rambutan seed oil. The properties of rambutan seed oil extracted by using the soaking extraction method were determined. The percentage yield from transesterification, the difference in biodiesel properties, and the efficiency of blended biodiesel in engine emission tests were also identified. A comparison of the diesel blended rambutan seed oil and rambutan seed oil biodiesel with the PNS of CME was also made.

2. Materials and methods

2.1. Rambutan seed oil extraction and characterization

Rambutan seeds were obtained from Brgy. Tinga, Batangas City. Washing and air-drying were done outside of the laboratory. It was followed by grinding using mortar and pestle, and oven drying at 70°C for 1 hour to reduce moisture, and then weighed to obtain 30 kg of fine rambutan seeds. Moreover, extraction and transesterification processes were adopted from the studies of [5] and [6], respectively. Oil was

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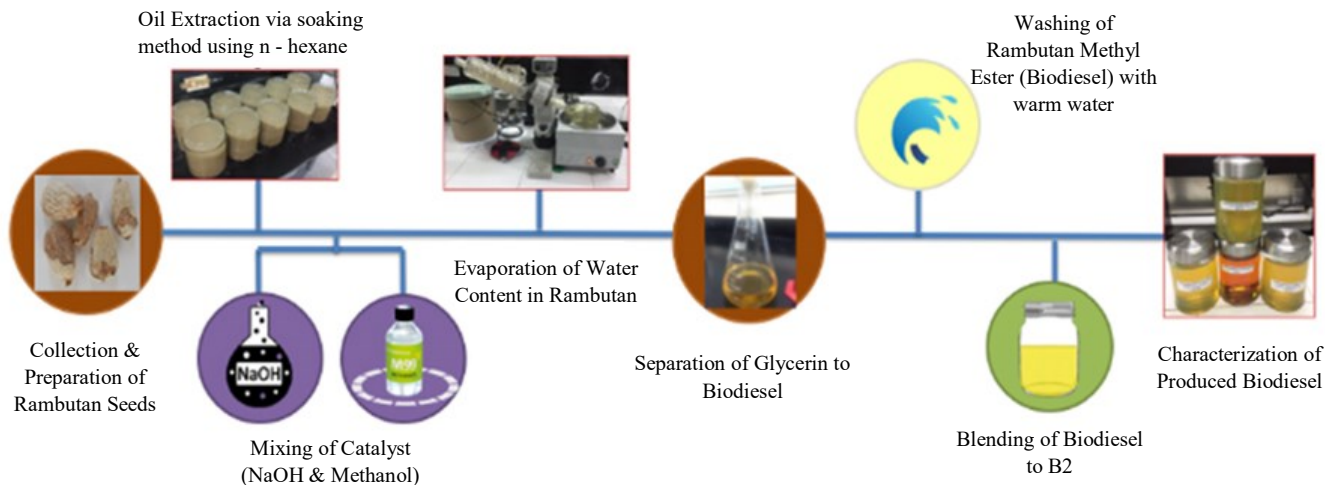


Figure 1. Schematic of the rambutan biodiesel production, characterization, and evaluation.

extracted from seeds using the soaking process at a ratio of 1:2. (400 g seed: 800 mL solvent) at varying soaking time in 4, 5 and 6 d. Following a period of soaking, the grains were filtered and pressed to extract the oil. The extracted liquid was shaken at 70°C in a rotary shaker until no n-hexane was detected (Figure 1).

The percent oil yield was obtained by using the following formula:

$$\% \text{ oil yield} = \frac{\text{weight of oil (grams)}}{\text{weight of seed used (grams)}} \times 100 \quad (1)$$

The rambutan seed oil density was determined by using a pycnometer. The following characteristics were characterized based on the Official Methods of Analysis of the Association of Official Agricultural Chemists [7]: fatty acid content, AOAC No. 940.28; free fatty acid content, AOAC No. 963.22; moisture and volatile matter, AOAC No. 926.12; and saponification value, AOAC No. 950.16.

Following extraction and characterization, base-catalyzed transesterification was used to convert the oil's triglycerides to biodiesel, with an oil: methanol molar ratio of 1:8 and NaOH at a concentration of 2% w/w (oil basis) [8]. The 500 mL rambutan seed oil was heated to 55°C until all of the fats melted. The oil was then treated with a solution of 500 mL methanol and 7g NaOH as a catalyst. After 2 h of agitation, the mixture was transferred to a tightly sealed container for 24 h of settling.

After settling, a biphasic solution was observed. The topmost layer yielded rambutan seed biodiesel, whereas the bottom yielded glycerin. The biodiesel and glycerin were separated by decantation. Washing was performed to remove any remaining methanol and glycerin from the biodiesel.

Warm water was poured into the separatory funnel to separate the methyl esters, and after settling, the water was drained from the bottom. This procedure was continued until the water had a pH of 6-7 and no bubbles were visible. Any turbidity indicates the existence of residual water in the fuel, which could be evaporated by heating the fuel to 100°.

To compute the biodiesel yield percentage, the following equation was used:

$$\% \text{ BY} = \frac{VB \text{ (ml)}}{VO \text{ (ml)}} \times 100 \quad (2)$$

where:

% BY = biodiesel yield

VB = volume of biodiesel produced, mL

VO = volume of oil taken, mL

To generate B2 biodiesel, a 980 mL of neat automotive diesel oil (ADO) and 20 mL of rambutan seed pure biodiesel (B100) was blended. Biodiesel properties were analyzed according to the standard test methods.

2.2. Determination of biodiesel properties

The biodiesel properties were determined based on the standards of the American Society for Testing and Materials International [9].

To determine the density (ASTM D4052-18a) approximately 0.7 mL of the 15°C sample was introduced into an oscillating sample tube to determine the density of the sample [10].

Time was measured for a fixed volume of liquid to flow under gravity through a capillary tube of a calibrated viscometer for the kinematic viscosity determination (ASTM D445-19) following the below equation

$$V = C \times t \quad (3)$$

where: V is the kinematic viscosity (mm²/s); C is the calibration constant of the viscometer (mm²/s); and t is the flow time (s) [11].

Distillation (ASTM D86) was done on a 100 mL specimen using a laboratory batch distillation unit under ambient pressure and temperatures of 227°C and 337°C for RME B100 and RME B2 respectively. It was designed to provide approximately one theoretical plate fractionation [12].

The specimen was tested at a constant slow rate which was heated directly with simultaneous stirring and interruption using a brass test cup with a cover to determine the flash point (ASTM D93) [13]. Preliminary heating was done and it was cooled and examined using PSL pour point

tester at intervals of 3°C. The observed lowest temperature was recorded as the pour point (ASTM D97) [14].

A colorimetric method was used for the determination of the acid number (ASTM D664) and p-naphthol-benzene was added to the solution. After neutralizing the sample with KOH it underwent a color change of orange to blue-green which indicated the end point [15]. The acid number was derived using the equation

$$\frac{V_{KOH} - 5.61}{W} \quad (4)$$

where:

V_{KOH} = Volume of potassium hydroxide (ml)

W = Weight of fatty acid oil being examined (g)

The carbon residue was measured after evaporation of the sample and heating it to 500°C in an inert atmosphere based on ASTM D4530. This method covers the determination of the amount of carbon residue formed after evaporation and pyrolysis of petroleum materials under certain conditions and is intended to provide some indication of the relative coke forming tendency of such materials [16].

Two correlations in SI units have been established between the ASTM cetane number and the density and 10%, 50%, and 90% distillation recovery temperatures of the fuel. This procedure has been developed for diesel fuels meeting the requirements of specification D975 Grades 1-D Low Sulfur, No. 1-D, 2-D, and 4-D. This relationship was given by the below equation

$$CCI = 45.2 + (0.0892)(T_{10N}) + [0.131 + (0.901)(B)][T_{10N}] + [0.0523 - (0.420)(B)][T_{90N}] + [0.00049][T_{10N}^2 - T_{90N}^2] + (107)(B) + (60)(B)^2 \quad (5)$$

where: CCI is the calculated cetane index by four-variable equation; D is the density at 15°C, g/mL (ASTM D1298 or D4042); DN is the $D - 0.85$; B is the $(e^{-3.5 DN}) - 1$; T_{10} is the 10% recovery temperature, °C; T_{10N} is $T_{10} - 215$; T_{50} is the 50% recovery temperature, °C; T_{50N} is $T_{50} - 260$; T_{90} is the 90% recovery temperature, °C; and T_{90N} is $T_{90} - 310$. All temperature determinations were based on ASTM D86, and were corrected to standard barometric temperature [17].

The sulphur content in the biodiesel was determined by using the ultraviolet fluorescence method (ASTM D4294). This standard method provides rapid and precise measurement of total sulfur in petroleum and petroleum products with a minimum of sample preparation. A typical analysis time is 1 min to 5 min per sample [18].

The measurements of glycerol and polyalcohols, which contain three or more adjacent hydroxyl groups, and the total glycerin were based on the standards of the American Oil Chemists Society (AOCS) [19]. Because glycerol reacts with sodium periodate in an acidic solution which in turn forms aldehydes and formic acid. AOCS Ea 6-51[20] determines the glycerol content of industrial glycerin. The glycerol content is expressed as a percentage (m/m). Determination of total glycerol was done after saponification, free glycerol was

taken directly from the sample and the combined glycerol was determined by difference [21].

2.3. Performance, emission, and combustion tests

The biodiesel performance was evaluated based on the engine emission characteristics including hydrocarbon (HC), carbon monoxide (CO), and nitrogen oxides (NO_x). A combustion test was also conducted. The results were compared with those of the commercially available diesel.

3. Results and discussion

The yield percentage of extracted oil soaked in with rambutan seeds from 4 d, 5 d, and 6 d are shown in Table 1. Soaking was done in triplicate per day. The highest yield of oil is 45.97% which was obtained from seeds that were soaked for 5 d. The result was higher than that of [6] which yielded only about 30-40%.

Table 1. Percentage yield of rambutan seed oil.

Day	Amount of Rambutan	Amount of Oil Obtained	Percentage Yield
4	200	63.33	36.40%±0.72
5	200	73.00	45.97%±1.41
6	200	66.67	38.32%±0.88

The properties of the rambutan seed oil that was soaked for 5 d are presented in Table 2.

Table 2. Properties of rambutan seed oil after 5 d of soaking.

Oil Properties	
Density (g/mL)	0.87
Moisture and Volatile Matter, %w/w	2.62
Free Fatty Acid (as Oleic Acid)	74.5
Saponification number	185
Fatty Acid Profile, w/w	
Caproic (C6)	0.215
Undecanoic (C11)	0.066
Palmitic (C16)	4.38
Palmitoleic (C16:1)	0.465
Stearic (C18)	5.42
Oleic (C18:1)	46.3
Linoleic (C18:2)	3.20
Linolenic (C18:3)	24.9
Cis-11-Eicosenoic (C20:1)	8.48
Cis-11,14-Eicosadienoic (C20:2)	0.016
Cis-8,11,14-Eicosatrienoic (C20:3n6)	0.014
Behenic (C22)	4.69
Erucic (C22:1n4)	1.57
Cis-11,14,17-Eicosatrienoic (C20:3n3)	0.027
Cis-5,8,11,14,17-Eicosapentaenoic (C20:5)	0.0203

The results of the measurement of rambutan seed oil moisture and volatile matter, free fatty acid, and saponification number showed less soap formation in the biodiesel production which increased the biodiesel yield.

The rambutan seed oil then underwent base transesterification in triplicates using NaOH as the catalyst to convert the rambutan seed oil to RME as shown in Table 3. The average volume of converted RME was at 484.33 mL and achieved a percent yield of biodiesel of 96.87%.

Table 3. Percentage yield of rambutan methyl ester.

	Amount of Rambutan Seed Oil (mL)	Amount of Converted Rambutan Methyl Ester (mL)	Percentage Yield (%)
Average	500	484.33	96.87±0.81

The produced RME was then washed to eliminate the impurities since the experimental procedures involved the use of different chemical bases. Washing is necessary because the impurities can potentially cause some damage to the engine and the equipment used in testing the RME's physio-chemical properties. The average volume of washing RME recovered was 464 mL and the percentage yield was 92.8±1.59% (Table 4).

Table 4. Percentage yield of converted RME after washing.

	Amount of Rambutan Seed Oil (mL)	Amount of Converted Rambutan Methyl Ester (mL)	Percentage Yield (%)
Average	500	464	92.8±1.59%

In compliance with the PNS based on the Biofuels Act of 2006, the determination of the properties of RME B100 (Table 5) and RME B2 (Table 6) was done.

Table 5. Experimental values of physical, thermal, and chemical properties of RME B100 (analysis in triplicates).

Test Analysis	Results (Average)
Acid Number, mg KOH/g sample	0.32±0.01
Carbon Residue, %mass	0.4±6.80x10 ⁻¹⁷
Cetane Number	45.33±0.58
Density at 15 °C, kg/L	0.8880±0.0002
Distillation AET @ 90% Recovered, C	270.67±1.15
Flash Point (PM), C	30.67±1.15
Pour Point, C	8±0.00
Kinematic Viscosity @ 40 C	2.77±0.06
Free Glycerin, %mass	0.0238±0.0003
Total Glycerin, % mass	0.20±0.01
Sulfur Content, %mass (ppm)	0.0533±0.0001

Table 6. Experimental values of physical, thermal and chemical properties of RME B2 (analysis in triplicates).

Test Analysis	Results
Acid Number, mg KOH/g sample	0.35±6.80x10 ⁻¹⁷
Carbon Residue, % mass	0.4±6.80x10 ⁻¹⁷
Cetane Number	50±0.00
Density at 15°C, kg/L	0.8485±0.00
Distillation AET @ 90% Recovered, C	336.67±0.00
Flash Point (PM), C	81.33±1.15
Pour Point, C	-15±0.00
Kinematic Viscosity @ 40°C	3.25±0.06
Free Glycerin, %mass	0.01±0.00
Total Glycerin, % mass	0.4±3.40x10 ⁻¹⁷
Sulfur Content, % mass (ppm)	0.26±0.006

It is important to take note of the acid number because high fuel acidity is related to potential corrosion and engine deposits [22]. The experimental value of the acid numbers of RME B100 (0.32mg KOH/g) and RME B2 (0.35mg KOH/g) were lower than that of the PNS (0.5mg/KOH).

Conradson Carbon Residue (CCR) is a test used to provide an indication of the coke forming tendencies of oil [23]. This measures the amount of carbonaceous residue remaining after the oil's evaporation and pyrolysis. The result of CCR shown in RME B100 and RME B2 were both higher (0.4 wt%) than the PNS. This implies that rambutan seed oil could potentially yield great amounts of carbon deposits.

Cetane number is the indicator of the combustion speed of diesel fuel and the needed compression for ignition. This is an important factor in determining the quality of diesel fuel [24]. A high value of cetane fuel would mean shorter ignition delay periods than lower cetane fuels. The gathered test results of RME B100 and RME B2 were 45.33 and 50, respectively. The value of RME B100 did not meet the PNS of 50.

Density is an important factor to consider in fuel because densities that are too far apart would lead to the settling of the heavier blend [6]. The experimental values of RME B100 were 0.8880 kg/L at 15°C, which failed to meet the PNS of 0.820-0.860 kg/L at 15°C.

Distillation of the fuel indicates the temperature at which 90% of the fuel is recovered. This property affects the heat requirement of the combustion to atomize the fuel. The gathered result of RME B100 was 270°C and RME B2 was 337°C.

For the flashpoint, it is the temperature at which the fuel will ignite when the combustion conditions are met. A high flash point would mean that it is safer to store the fuel at ambient temperature environments than with fuels that have low flashpoints. The result of the flashpoint test of RME B100 was 30.67°C and RME B2 was 80.67°C. By comparing the flashpoint values of [25] which were 65°C with a PNS of 100 and 55 min for CME B100 and CME B2, respectively.

Thus, RME B100 would have a greater tendency to ignite at ambient temperature.

For the pour point, it is the temperature below where the liquid loses its flow characteristics, and it is also the minimum temperature in which the oil can pour down from a beaker [26]. The test result of the pour point for the RME B100 and RME B2 is 8°C and 15°C, respectively. Thus, below ambient temperature, the RME B100 and B2 respectively can solidify and be less efficient.

For the kinematic viscosity, which is a property of liquid fuels to resist movement due to intermolecular attraction forces. Fuels that have low viscosity would have small spray particles and vice versa for the high viscosity fuels [25]. The RME B100 and RME B2 results for kinematic viscosity were 2.77 and 3.25, respectively. Therefore, the produced methyl ester was proven to be within the PNS.

The glycerol portion of the compounds is referred to as bound glycerol and if this is added to the free glycerol, the sum is known as the total glycerol. The test result of free glycerin RME B100 was 0.0238% mass and RME B2 was 0.01% mass. The results of the total glycerin measurement were the same for RME B100 and RME B2 (0.20%). With the given data, it was concluded that the conversion of seed oil to biodiesel was efficiently done because it is less than the 0.24% standard value.

Lastly, for the sulphur content, this is used to facilitate sulphate particulate emission reductions that are necessary for meeting the 1994 emission standards for heavy-duty highway engines. The result of the test for RME B100 was 0.0553% mass and for RME B2, 0.26% mass. These results show that the RME B100 has a value that exceeds the PNS, however, RME B2 has conformed with the given standard.

Table 7 and 8 shows the comparison of the results of RME to the PNS for biodiesel. Table 7 reveals that the RME B100 properties of carbon residue, flash point, free glycerin and sulphur content have not complied to the PNS B100. Thus, it could not be considered as an alternative fuel. On the other hand, most of the properties' results shown in Table 8 have complied with the PNS except for the carbon residue.

Table 7. Comparison of RME B100 to the PNS B100.

Properties	RME B100	PNS B100	Remarks
Acid Number	0.32	0.5 max	Conform
Carbon Residue	0.4	0.05 max	Not conform
Cetane Number	45.33	42 min	Conform
Density	0.8880	0.87-0.89	Conform
Distillation	270.67	250 min	Conform
Flash Point	30.67	100 min	Not conform
Pour Point	8	9 max	Conform
Kinematic Viscosity	2.7	2.0-4.5	Conform
Free Glycerin	0.0238	0.02 max	Not conform
Total Glycerin	0.20	0.24 max	Conform
Sulfur Content	0.0533	0.05 max	Not conform

Table 8. Comparison of RME B2 to the PNS B2.

Properties	RME B100	PNS B2	Remarks
Acid Number	0.35	0.5 max	Conform
Carbon Residue	0.4	0.30 max	Not conform
Cetane Number	50	50 min	Conform
Density	0.8485	0.82 – 0.86	Conform
Distillation	337	300 min	Conform
Flash Point	80.67	55 min	Conform
Pour Point	-15	5 max	Conform
Kinematic	3.25	2.0 – 4.5	Conform
Free Glycerin	0.01	0.02 max	Conform
Total Glycerin	0.20	0.24 max	Conform
Sulfur Content	0.26	0.30 max	Conform

3.1. Statistical analysis of the laboratory results

There is no significant difference between the properties of biodiesel (B100) and biodiesel blend (B2) to the PNS for CME (Table 9).

Table 9. Difference between the properties of B100 produced from rambutan seed oil with the PNS for CME.

Properties	p-values	Computed t-values	Decision on H ₀	Verbal Interpretation
Density	0.237	-1.391	Accept	No significant difference
Kinematic Viscosity	0.540	0.669	Accept	No significant difference
Distillation	0.00002	-23.434	Reject	Significantly different
Flash Point	0.000	78.617	Reject	Significantly different
Pour Point	0.0002	12.247	Reject	Significantly different
Acid Number	0.00003	22.045	Reject	Significantly different
Carbon Residue	0.000	-60.321	Reject	Significantly different
Cetane Number	0.001	-9.999	Reject	Significantly different
Free Glycerin	0.00004	-19.379	Reject	Significantly different
Total Glycerin	0.0003	11.029	Reject	Significantly different
Sulfur Content	0.000003	-37.796	Reject	Significantly different

The values of acid number, flash point, pour point, and total glycerin, were statistically lower than the PNS for 100% CME. However, the carbon residue, cetane number, distillation, free glycerin, and sulphur content has given a significantly higher value than the given national standard for 100% CME. Non-significant properties such as density and kinematic viscosity are known to be within the range of values (Table 10).

Table 10. Difference between the properties of B2 produced from rambutan seed oil with the PNS for CME.

Properties	p-values	Computed t-values	Decision on H ₀	Verbal Interpretation
Density	0.502	-0.736	Accept	No significant difference
Kinematic Viscosity	0.997	0.005	Accept	No significant difference
Distillation	0.000001	-55.000	Reject	Significantly different
Flash Point	0.000008	-29.103	Reject	Significantly different
Pour Point	0.000	344.691	Reject	Significantly different
Acid Number	0.00001	25.852	Reject	Significantly different
Carbon Residue	0.0003	-12.247	Reject	Significantly different
Cetane Number	1.000	0.000	Accept	No significant difference
Free Glycerin	0.00007	17.235	Reject	Significantly different
Total Glycerin	0.002	6.967	Reject	Significantly different
Sulfur Content	0.000008	-29.515	Reject	Significantly different

According to Table 10, RME B2's acid number, pour point, free glycerin, total glycerin, and sulphur content are significantly lower compared to the PNS for 2% CME and the non-significant properties such as cetane number, density, and kinematic viscosity are in the range of the national standards. However, the carbon residue, distillation, and flash point values are significantly higher than the PNS for 2% CME.

3.2. Performance testing for RME blend

Using the engine test, the application of RME B2 was analyzed, measured and compared to the emission of the commercial diesel (Table 11).

Table 11. Engine emission results for the RME B2.

Properties	Commercial	Rambutan Methyl
Hydrocarbon (HC)	220 ppm	180 ppm
Carbon Monoxide (CO)	0.08%	0.09%
Carbon Dioxide (CO ₂)	4.4%	4.1%

A car engine with a maximum rotation per minute (rpm) of 2000 was used for the engine testing. The gathered data for commercial diesel has garnered higher values compared to RME B2 in terms of hydrocarbons and carbon dioxide. It also implies that as the RME content of the commercial diesel increases the hydrocarbon and carbon dioxide composition decreases which indicates an improvement within its combustion capabilities and is only a good additive. The increase in carbon monoxide production in RME denotes incomplete combustion that has occurred due to a number of

reasons such as poor air ratio because of faulty oxygen sensor or defective throttle position sensor [27]. Ideally, the biodiesel blends are done to reduce the emissions of the petroleum diesel and ultimately decrease the greenhouse gases that cause air pollution.

4. Conclusions

The based catalyzed transesterification is an effective way of converting rambutan seed oil to methyl ester, which resulted in a high percentage yield. There is a significant difference between the properties of B2 produced from rambutan seed oil with the PNS for CME except for cetane number, density, and kinematic viscosity. The majority of the results have met the PNS for 2% CME. Emission test results had shown a low value of carbon dioxide and hydrogen carbon emissions suggesting that the biodiesel produced can only improve the combustion of fuels and serves as an additive to commercially available diesel. The following investigations are warranted, which include determining the effect of increasing the soaking time; apart from transesterification, the use of alternative techniques to convert seed oil to biodiesel; formulation with different ratios for the other blending element of the biodiesel-diesel fuel mixes, and evaluating its other potential influence on engine power, torque, fuel consumption, and exhaust emission testing.

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