

RESEARCH ARTICLE



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Influence of Various Operating Characteristics on the Biodiesel Preparation from Raw *Mesua ferrea* oil

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Abstract

Objectives: The aim of this study is to produce biodiesel from crude *Mesua ferrea* oil and to determine the influence of different operating characteristics, especially catalyst content, methanol to oil molar ratio and reaction temperature on the evaluation of biodiesel yield. Likewise, to assess the physicochemical properties of *Mesua ferrea* oil biodiesel for diesel engine applications. **Methods:** Prior to biodiesel production, the crude oil was degummed, bleached and deodorized to remove free fatty acids. Subsequently, the *Mesua ferrea* oil was converted into biodiesel using the basic transesterification process. The catalyst concentration was varied as 1%, 1.5% and 1.75%, respectively, while the molar ratios were 6:1, 8:1 and 10:1, and the reaction temperatures of 50°C, 60°C, and 70°C were considered. Finally, the physicochemical properties were estimated with the aid of ASTM standards. **Findings:** The individual conversion efficiencies of MOBD (*Mesua ferrea* oil biodiesel) were 98.8%, 98.6%, and 98.1%, respectively. In addition, KOCH₃ catalyst has shown better yield of biodiesel than other catalysts. The better yield of biodiesel is found at 8:1, 1.5%, and 60°C and the individual yields are 98.8%, 98.6% and 98.1%, respectively. **Novelty:** The influence of different operating parameters on the evaluation of optimal biodiesel yield when using hydroxide and methoxide-based catalysts is a new approach. The biodiesel yield of the methoxide-based (NaOCH₃ and KOCH₃) catalyst was higher than that of the hydroxide (NaOH and KOH) catalyst. Similarly, the cetane number of the biodiesel is increased, which is a primary fuel property during combustion in diesel engines.

Keywords: *Mesua ferrea*; Biodiesel; Conversion Efficiency; Catalyst; Temperature; Molar

1 Introduction

Massive production of petrodiesel and other petroleum-based commodities has led to serious depletion of petroleum resources. Industrialization, rapid population growth, transportation, urbanization all contribute to this extreme energy demand. As a result, non-renewable fossil resources are being depleted, and at the same time an energy

crisis is threatening to hit modern society. Due to their environmental friendliness and ability to be produced in a sustainable manner, biofuels such as biodiesel have attracted a great deal of interest from the scientific community as potential alternative fuels^(1,2). Diesel engines are more likely to use biodiesel. Lower sulfur and aromatics content, as well as higher flash point, good oxygen presence, higher cetane number, and better lubricity are some of the advantages that vegetable oils offer when used as diesel fuel. Their disadvantages include very high viscosity, higher pour point, lower heating value and lower volatility^(3,4). Transesterification of vegetable oils with chemical catalysts is the standard method for producing biodiesel. A catalyst is used to speed up the process by increasing the solubility of the alcohol. Homogeneous alkali catalysts can be used to convert triglycerides into methyl esters with high yields, little time, and low cost⁽⁵⁾. Although the properties of biodiesel fuel make it a viable alternative to diesel fuel, the production of biofuel is not economically viable due to the high cost of raw materials and processing. Therefore, increasing biodiesel yield is one of the critical parameters and choosing the best catalyst among different catalysts^(6,7). Most research on the conversion of vegetable oil to methyl esters has focused on homogeneous base catalysts because they are cheap and widely available. Potassium hydroxide (KOH) and sodium hydroxide (NaOH) are two catalysts that have been shown to be effective in the production of biodiesel. These processes took place under conditions similar to those found in nature, such as fast reaction times and high biodiesel production. The byproduct is water, which reduces the total amount of biodiesel that can be produced by this method. The production of biodiesel with sodium methoxide or potassium methoxide is more efficient than with sodium hydroxide or potassium hydroxide, since these processes do not produce water^(8,9). The conflict between food and fuel would be solved if oil sources were not edible. To alleviate the lack of energy sources, biodiesel made from non-edible oils is a good alternative, and *Mesua ferrea*, along with *Jatropha*, *Pongamia*, etc., has great potential as a biodiesel resource^(10,11).

After summarizing the research conducted so far, the influence of base catalysts in biodiesel production was investigated. However, the use of methoxide catalysts in biodiesel production is limited. Moreover, biodiesel was obtained from *Mesua ferrea* seed oil with few acidic and basic catalysts. However, the use of methoxide catalysts in the production of biodiesel and the study of its yield is a completely new investigation. With this background, the purpose of this research is to study the production of biodiesel from *Mesua ferrea* oil, a potential source of non-edible materials, under different operating conditions to evaluate the yield (or) methyl ester conversion using methoxide catalysts and the physicochemical properties of the product.

2 Methodology

2.1 Collection of materials, oil extraction, and chemicals

The tree *Mesua ferrea*, often called Ceylon ironwood, belongs to the family Caryophyllaceae and is known by its common name. After drying in an oven at a temperature of 60°C, the seeds were then crushed with a seed mill and the oils extracted. It is estimated that the oil yield is in the range of 60-63% by weight and has a type that can be described as reddish-brown. The oil is contained in the seed kernel and each seed can produce one to four kernels. The purity level of the substances used in this study was 99.99%. The chemicals used for the study were bleaching powder, 0.1N NaOH solution, methanol, isopropyl alcohol, NaOH, KOH, NaOCH₃, KOCH₃, phosphoric acid, phenolphthalein indicator, and so on.

2.2 Free fatty acid (FFA) content of *Mesua ferrea* oil

The free fatty acid content (FFA) in vegetable oils has a significant effect on the process of methyl ester conversion, and oils with a high FFA content are difficult to convert to biodiesel. However, attempts to convert the oil to soaps result in a reduction in biodiesel conversion efficiency.

The FFA content of the oil was determined by the titration method. A 10 grams of raw *Mesua ferrea* oil with 50 ml of isopropyl alcohol was taken in a container. Subsequently, 3-5 drops of phenolphthalein indicator was added to the oil and alcohol mixture. This solution was subjected to titration with solutions of 0.1 N NaOH and the results were recorded in the burette. The FFA of the oil was determined as follows after these data were collected and analyzed.

$$FFA = \frac{282 \times NaOH \text{ Normality} \times \text{Sample rundown in burette}}{10 \times \text{Weight of Mesua ferrea oil sample}}$$

According to the results, the FFA concentration in the oil is 6%; Therefore, the oil has to go through the degumming process to get rid of the FFA.

There is a high probability that the FFA content could remain after the degumming process. However, all the gums in the oil is removed. Initially, the raw *Mesua Ferrea* oil was diluted with a mixture of 0.01% H₂O and 0.1% H₃PO₄. Thereafter, it was heated in an electric heater with stirring for about 40 minutes while keeping the temperature constant at 60°C. After heating, the mixture was allowed to cool for 24 hours, and then the gums that had sunk to the bottom were removed. The degummed oil was

then bleached to get rid of all other impurities. Bleaching powder was added during the heating process, making up between 1-3% of the weight of the oil. The reaction was also sustained for 35 minutes as the temperature was increased to 125°C. The oil was then allowed to cool before being filtered with filter paper to remove all impurities. Subsequent to the bleaching process, the oil was allowed for deodorization process. Deodorization is a technique that requires both high vacuum pressure and high temperature. Using nitrogen as the carrier gas, this process was maintained at temperatures between 250°C and 270°C and a vacuum pressure of 760 mm of Hg. This could remove the fatty acid content from the oil. The final oil FFA was lowered to 2% approximately.

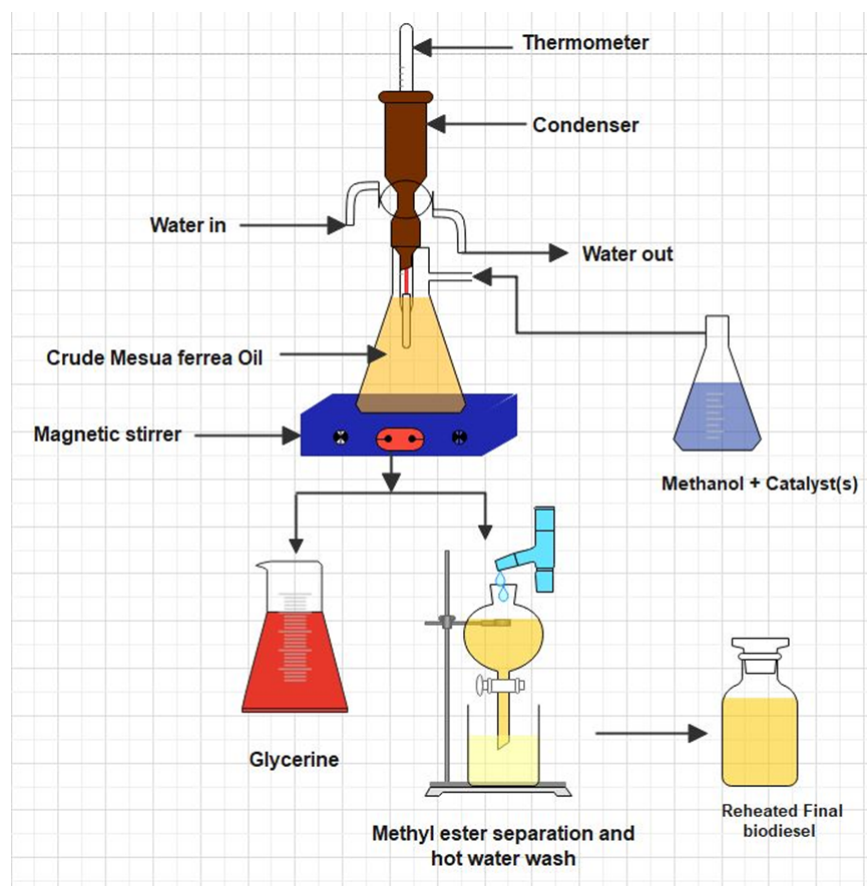


Fig 1. Transesterification process

2.3 Transesterification process

Then, a mixture of methanol (alcohol) and catalyst (NaOH) was added to the heated oil in varying proportions and the condenser was positioned to be cooled by running water. The magnetic pellet was used to stir this solution and the rotation speed was kept at 900 rpm during the mixing process. Different catalysts were used in the analysis based on the different solubility, methanol/oil molar ratio (from 4:1 to 10:1), catalyst amount (from 1% to 2%) and temperatures ranging from 60°C to 70°C for 60 minutes. After the reaction was complete, the procedure was stopped and the product left in the separating funnel for a period of 10 hours. This resulted in a two-layer separation of the glycerol and the methyl ester, with the methyl ester occupying the top layer and the glycerol the bottom. The oil was then cleaned again in 60°C water to remove any remaining cleaning agents (soaps). Eventually, the methyl ester was heated (at 90°C) to drive off the moisture. The properties of biodiesel were estimated in accordance with ASTM standards ASTM D1298, ASTM D4809, ASTM D976, ASTM D445, ASTM D130, ASTM D 97, and ASTM D92 were used to investigate the characteristics of specific gravity, heating value, cetane number, viscosity, copper corrosion, cloud point, and flashpoint, respectively. The properties are listed in Table 1 and all properties comply with ASTM standards.

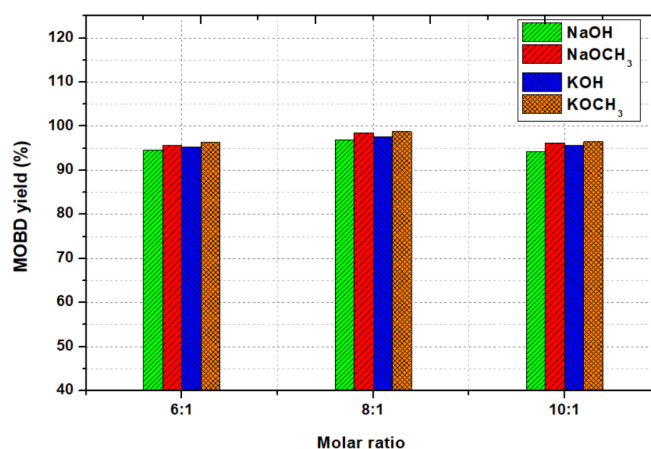
Table 1. Properties of *Mesua ferrea* oil biodiesel

Fuel property (units)	Testing method	Range as per ASTM standards	<i>Mesua ferrea</i> biodiesel (B100)			
			NaOH	NaOCH ₃	KOH	KOCH ₃
Specific gravity 15°C	ASTM D1298	860-900	0.888	0.886	0.884	0.882
Heting value (kJ/kg)	ASTM D4809	42	39223	39317	39338	39364
Cetane Number	ASTM D976	47 (min.)	55	55	55	55
Viscosity at 40°C (Cts)	ASTM D445	2.5-6	4.34	4.26	4.18	4.16
Copper corrosion	ASTM D130	1 (max)	1a	1a	1a	1a
Cloud point (°C)	ASTM D97	6 (max.)	-3	-3	-4	-4
Flashpoint (°C)	ASTM D92	130 (min.)	152	150	148	147

3 Results and Discussion

3.1 Methanol to oil molar ratios

Figure 2 shows the variation of MOBD yield as a function of methanol to oil molar ratio. The methanol to oil ratio was increased from 6:1, resulting in an upward trend in the amount of biodiesel produced. In addition, the biodiesel yield was optimized at 8:1, and increasing the molar ratio beyond this point resulted in a decrease in the amount of product produced. At a molar ratio of 8:1, the maximum yield was achieved with the entire catalyst. When the molar ratio was increased to 10:1, the amount of biodiesel produced decreased, mainly due to the constant presence of alcohol in large amounts and the decrease in the chemical activity of the catalyst. The increase in insolubility affects the insulating ability of glycerol. In addition, the methoxide-based catalyst has shown higher MOBD yield than the hydroxide catalysts. At a molar ratio of 8:1, the yields of MOBD for NaOH, NaOCH₃, KOH and KOCH₃ were 96.8%, 98.5%, 97.6% and 98.8%, respectively. Among all the catalysts used, KOCH₃ was found to have a better yield of 98.8% compared to the other catalysts.

**Fig 2.** Variation of yield with respect to molar ratio

3.2 Influence of catalyst proportion

The effect of catalyst concentration on MOBD yield is shown in Figure 3. The range of the catalyst was changed from 1% to 1.75%, and when the catalyst concentration reached 1.5%, the yield of MOBD started to decrease. This was due to lower glycerol deposition, which was affected by the emulsification of methyl ester.

According to the results of the current study, a higher catalyst concentration resulted in a lower MOBD conversion rate compared to the value determined as optimal. Moreover, if the concentrations are not high enough, it is not possible to convert the biodiesel through the process of transesterification. Therefore, the optimum value of catalyst concentration must be determined. The yields of MOBD for NaOH, NaOCH₃, KOH, and KOCH₃ were 95.7%, 97.9%, 97.1%, and 98.6%, respectively, when the catalyst concentration was 1.5%. Compared to the other catalysts used, the yield with the KOCH₃ catalyst was 98.6%

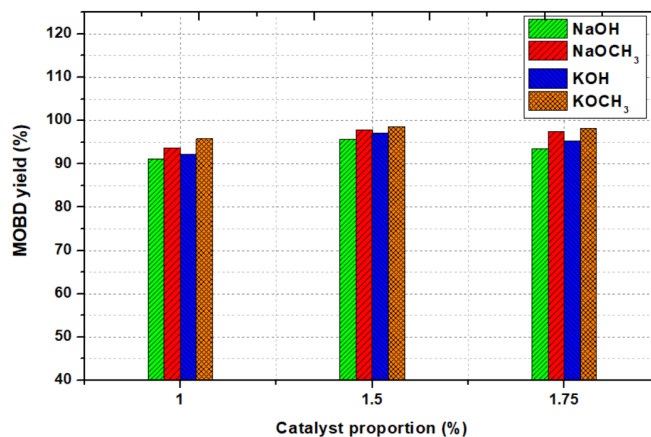


Fig 3. Variation of yield with respect to catalyst

higher than the others due to absence of water presence during the biodiesel preparation.

3.3 Reaction temperature

The change in MOBD yield shown in Figure 4 is the result of varying the reaction temperature. It is clear that increasing the reaction temperature from 50°C to 70°C resulted in an increase in the amount of MOBD produced. The reaction rate improves to some extent with an increase in temperature, but further increase in temperature may decrease the yield, so it was limited to 70°C. This is due to emulsification of the biodiesel and excessive loss of methanol, which contributes to an increase in yield at higher temperatures. Methanol loss at high temperatures is also a contributing factor. For this reason, the amount of glycerol extracted during separation is much lower, which in turn leads to a lower yield of MOBD. At a reaction temperature of 70°C, the yields of MOBD for NaOH, NaOCH₃, KOH, and KOCH₃ were 94.6%, 97.1%, 96.1%, and 98.1% respectively; however, the maximum yield for KOCH₃ was 98.1% compared to other catalysts.

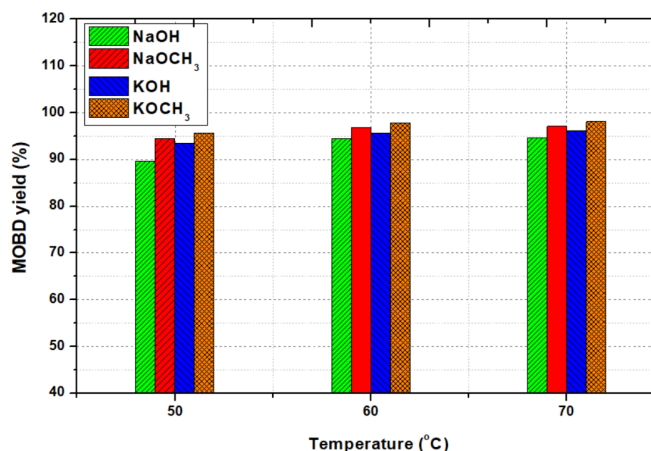


Fig 4. Variation of yield with respect to reaction temperature

4 Conclusion

The present work was carried out with non-edible *Mesua ferrea* oil to produce the biodiesel, evaluate the yield and assess the physicochemical properties with different hydroxide and methoxide-based catalysts. In addition, the FFA content of the oil was reduced by the degumming method and the biodiesel was produced. After analyzing the effects of different catalysts

used in the preparation of *Mesua ferrea* biodiesel (MOBD), it was found that potassium methoxide (KOCH_3) followed by sodium methoxide (NaOCH_3) gave the best results in terms of yield and physicochemical properties. The optimal conditions for biodiesel conversion (98.8%) are 8:1, 1.5%, and 60°C . In addition, the KOCH_3 catalyst has been shown to provide a higher yield of biodiesel compared to other catalysts. The biodiesel produced from *Mesua ferrea* oil met all acceptable parameters specified by ASTM standards in terms of its properties. There was an increase in the cetane number, which should lead to an improvement in diesel engine combustion.

The conclusions show that the biodiesel yield of *Mesua ferrea* can be increased using methoxide-based catalysts compared to hydroxide catalysts. Also, the physicochemical properties of *Mesua ferrea* biodiesel correlate well with the ASTM range, so *Mesua ferrea* biodiesel blends can be used in diesel engines to evaluate performance and emissions. Future studies may use metal oxide catalysts and nanocatalysts to increase yield of *Mesua ferrea* biodiesel.

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