



Biodiesel-Alkaline Transesterification Process for Methyl Ester Production

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ABSTRACT

The world needs to increase renewable and alternative fuel sources such as Biomass, Bioethanol, and Biodiesel to compete with fossil fuels. Biodiesel is an important renewable fuel source since it can be used in regular diesel vehicles without requiring engine modifications. Conventional biodiesel production takes around 90 min of reaction time. A longer reaction time is not suitable for commercial production. Furthermore, traditional products such as oil react with biodiesel methoxide to produce a maximum of 90% biodiesel yield. As the catalyst is not involved with the reaction, pure methanol and methoxide (methanol with KOH catalyst) are separately added to the system to enhance the pre-reaction step. By changing the methanol to methoxide ratio, biodiesel is produced, and yield is calculated. The highest yield, which is 95%, is obtained with a 5:15% methanol to methoxide ratio. The total reaction time with the new experimental procedure is only 20 min. That is a significant reduction by saving operating costs such as energy consumption. Produced biodiesel show similar properties to that of standard biodiesel.

INTRODUCTION

Global warming and environmental pollution are alarming today due to fossil fuel combustion in the power generation and the transport sector. The atmospheric carbon dioxide (CO₂) concentration hit 417 ppm (parts per million) in May 2020, which was 414 ppm in May 2019 (Andrew & Chris 2020). CO₂ levels have reached unprecedented heights in human history, as shown in Fig. 1.

The impact of man on the environment through his daily activities is complex and irreversible. Most of the intentional or unintentional causes are contributing to environmental pollution and changes in the ecosystem. There is no single cause that can be considered the main reason for environmental pollution, as most of them are interrelated. However, the following causes can be viewed as the main contributors to ecological damage and environmental pollution (Manisalidis et al. 2020).

- Population growth and deforestation
- Industrialization and urbanization

Vehicle emissions

There is a considerable impact of population growth on the environment. With the population increase, we consume more and more natural resources and deplete the resources faster than before. Simultaneously, more and more waste

is generated and disposed of in the open environment polluting the water, air, and soil. Both ways, the negative impact on the environment is significant (Manisalidis et al. 2020).

The population growth rates are not at an environmentally sustainable level. The increasing demands for the consumables such as food, water, land, power, and energy lead to devastating conditions worldwide. It should be addressed immediately to avoid resource depletion and irreversible impact on the environment. According to Fig. 1, it is clear that around 37 billion tons of CO₂ are emitted into the atmosphere annually (Our World in Data n.d).

CO₂ emissions from vehicles account for a substantial part of CO₂ emissions due to fossil fuel combustion. Even though fossil fuel resources are depleting and the prices are escalating, it does not discourage the usage as they are easily accessible to consumers. The use of fossil fuels such as coal, oil, and gas, over many years, directly causes global warming and climate change (Ogunwole 2012, Swapan & Bablu 2020).

To stabilize the concentration of CO₂ in the atmosphere, the world needs to increase the usage of renewable and alternative fuel sources such as biomass, bioethanol, and biodiesel to compete with fossil fuels. Biodiesel is an important renewable fuel source since it can be used in regular diesel vehicles without requiring engine modifications.

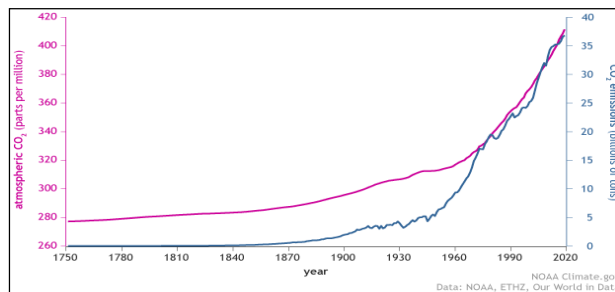


Fig. 1: Atmospheric CO₂ Concentration from the year 1750-2020. (Our World in Data n.d)

Bio-Diesel Production

Several edible and non-edible raw materials are used to produce biodiesel around the world (Onyinyechukwu et al. 2018). Edible oils can be categorized as coconut oil, palm oil, sunflower oil, rapeseed oil, etc., and non-edible oils can be classified as rubber seed oil, algal oil, Jatropha oil, and waste cooking oil.

There are multiple ways of producing biodiesel and alkaline esterification on the top of the list due to flexibility, simplicity, and the production yield with readily available raw materials. Biodiesel's manufacturing cost is the most important factor in its ability to compete with fossil diesel. To minimize the production cost, raw material cost, operating cost, and the energy requirement to produce biodiesel have to be minimized. The cost of the raw material has a direct promotional relationship with the overall biodiesel cost (Braga et al. 2015).

Alkaline Esterification

Alkaline esterification is the process by which the glycerides present in the oil react with alcohol (methanol, ethanol) in the presence of an alkaline catalyst (KOH, NaOH) to form esters and glycerol as a byproduct (Banerjee & Chakraborty 2009, Enweremadu & Mbarawa 2009). To perform the forward reaction, it is essential to perform the reaction with the catalyst present (Fig. 2).

The catalyst is prepared by adding potassium hydroxide (KOH) into alcohol (methanol) and stir it properly until it dissolves completely. The ultimate goal of the catalyst preparation step is to produce methoxide (CH₃O⁻), which may then be processed with triglycerides in the oil sample. The active catalyst for the production of methyl esters is methoxide ion, -OCH₃, which attacks triglyceride molecules and creates methyl esters. It is regenerated at the end of each reaction step when a hydrogen ion is stripped from a nearby methanol molecule.

If ethanol is being used, then the corresponding catalyst is called ethoxide, -OCH₂CH₃. During the catalyst preparation step, OH⁻ ions abstract the hydrogen ion to form water (H₂O). Water will increase the formation of soap when free fatty acids are available, eventually reducing the yield of the biodiesel production process. It is important to process the oils with low free fatty acids (FFA) (<2% of the oil weight) to avoid soap formation by saponification reaction. To maintain the FFA% below 2, oils with higher FFA have to go through an acid esterification reaction before the alkaline esterification (Paraj et al. 2019).

It has been reported that the transesterification process depends on many factors, such as reaction temperature, mixing speed, reaction time, catalyst, alcohol percentage, and FFA in the oil. Therefore, base case experiment is performed

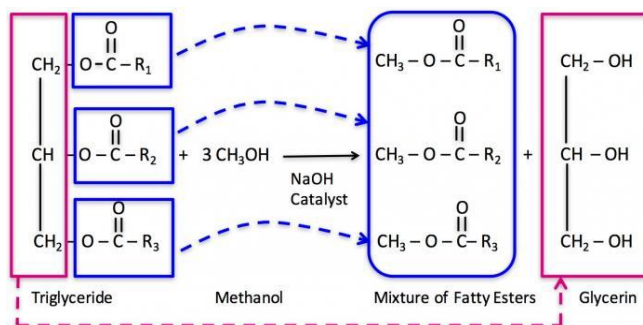


Fig. 2: Graphical representation of the esterification reaction.

with the operating conditions which are given in the literature without any changes such as 60°C operating temperature, 600RPM mixing speed, KOH as a catalyst and 1% of oil weight, <2% FFA content in the oil, methanol as alcohol and 20% of oil volume (Wanodya & Arief 2013, Phan & Phan 2008, Corro et al. 2011, Endang et al. 2018).

MATERIALS AND METHODS

Pure coconut oil is used as the feedstock for this study. The total methanol amount taken is 20 mL since the oil sample is 100 mL. To meet the 1% W/W of the oil weight, 1 g of KOH is used. The operating temperature is 60°C with 600 RPM, and 90 min reaction time is selected for the present experiment. The FFA% of coconut oil is 0.5% with the titration method. Considering previous studies, a homogeneous base catalyst such as KOH is used. At first, the oil sample is preheated to remove any oil moisture to avoid soap formation during the reaction. The exact amount of KOH is added to the methanol and mixed well to prepare the methoxide. The prepared methoxide is then added to the heated oil sample while the stirring process continues.

Table 1: Properties of fresh coconut oil.

Property	Test Method	Unit	Value ^a
Specific gravity at 60°F	Gravity Bottle Method	-	0.9331
Kinematic Viscosity at 40°C	ASTM D 445 - 01 Using Cannon - Fenske Routine Viscometer	cSt	27.71
Flash Point	ASTM D 92 - 05a Using COC	°C	309.0
Water Content	ASTM D 4377 - 00	% by mass	0.12
Total Sulphur Content	ASTM D 129 - 00	% by mass	0.42

a: Tested by the Petroleum and Lubricant Testing Laboratory, Industrial Technology Institute (ITI), Colombo 07, Sri Lanka

Table 2: Fatty acid composition of fresh coconut oil.

Acid type	Formula	Method	Composition % ^a
Caproic acid	C ₆ H ₁₂ O ₂	CML/MM/05/51/003	0.38
Caprylic acid	C ₈ H ₁₆ O ₂		6.81
Capric acid	C ₁₀ H ₂₀ O ₂		5.45
Lauric acid	C ₁₂ H ₂₄ O ₂		48.28
Myristic acid	C ₁₄ H ₂₈ O ₂		19.50
Palmitic acid	C ₁₆ H ₃₂ O ₂		9.12
Stearic acid	C ₁₈ H ₃₆ O ₂		2.70
Oleic acid	C ₁₈ H ₃₄ O ₂		6.18
Linoleic acid	C ₁₈ H ₃₂ O ₂		1.51
Arachidic acid	C ₂₀ H ₄₀ O ₂		0.07

a: Tested by the Chemical and Microbiological Laboratory, Industrial Technology Institute (ITI), Colombo 07, Sri Lanka

RESULTS AND DISCUSSION

The properties of the fresh coconut oil and the oil sample composition are given in Table 1 and Table 2. According to Table 2, it can be clearly seen that coconut oil majorly comprises Lauric acid (C₁₂H₂₄O₂). Coconut oil's physical properties were tested by the Petroleum and Lubricant Testing Laboratory, Industrial Technology Institute (ITI), Colombo 07, Sri Lanka. The fatty acid composition analysis was carried out by the Chemical and Microbiological Laboratory, Industrial Technology Institute (ITI), Colombo 07, Sri Lanka. Using the fatty acid composition, the molecular mass of the fresh coconut oil can then be estimated. In this research, the molecular mass of fresh coconut oil is estimated as 679.3 g.mol⁻¹.

The transesterification reaction was performed in the batch reactor. After 90 min of reaction time, the reacted oil sample was moved to the separation funnel and kept overnight for separation. After 24 hours of separation, the top layer fatty acid methyl ester (FAME) and the bottom layer glycerin (glycerol) were identified (Fig. 3).

The top layer of FAME is separated and washed with distilled water which is 50% of the FAME sample. The

washing process is carried out at 60°C with constant stirring. After completion of washing, the washed sample is added to the separation funnel for further separation. The upper layer is taken and the pH value is analyzed. The lower layer is discarded to remove all the residual by-products like excess alcohol, excess catalyst, glycerine, and soap. If the pH of washed biodiesel is less than 7, we must repeat the washing process until pH reaches 7 (Fig. 4). The washed biodiesel sample was heated to 110°C while being stirred to remove further moisture. Pure biodiesel is used for further testing and composition analyses to verify the quality of the biodiesel.

Parameter Optimization

The parameters' effects on the biodiesel production process are important to optimize the process.

a. Effect of reaction time

Effect of the reaction time for alkaline esterification is vital as it counts the energy consumption and the efficiency of the process. By maintaining 60°C reaction temperature and 600 RPM as the mixing speed, reaction time varied from 5 min to 120 min. During the analysis, oil volume of 100 mL, 20% of methanol by volume of oil, and 1% KOH (Methoxide) by weight of the oil are maintained to evaluate the single parameter effect. The effect of reaction time on the coconut oil conversion efficiency is shown in Fig. 5. The percent yield of biodiesel is calculated on a weight basis concerning the initial oil used for transesterification. With reaction times ranging from 5 to 50 min, the FAME production yield increased rapidly; after that, the conversion efficiency increased slowly and remained stable at around 85% at 60 min. Based on the results, it can be assumed that the reaction rate is steady and in equilibrium after 80 min.

b. Effect of reaction temperature

Methyl esterification of coconut oil was carried out at 30, 40, 50, 55, and 60°C to determine the effect of reaction tem-

perature on the formation of the methyl ester. The methanol and KOH were maintained at a constant value of 20% and 1 wt.% for all these experiments. As can be seen from Fig. 6, for the same final reaction time, the ester formation yield increases with the temperature. Because methanol has a boiling point of 64.7°C, temperatures above 60°C are not used in the study. When the reaction temperature closes to the boiling point of methanol, it will start to vaporize and form a bubble to inhibit the reaction. Higher temperatures can be maintained by changing the operating pressure, which will increase the operating cost. The maximum yield of FAME is achieved at the 60°C reaction temperature and selected as the best operating temperature for further analysis.

c. Effect of reaction stirring speed (RPM)

In alkaline esterification, the mixing speed (RPM) is critical. To investigate the effect, the mixing speed is varied between 0 and 800 RPM while maintaining constant values of 60°C reaction temperature, 80 min working duration, 20% methanol, and 1% KOH. The reaction at the 0 RPM, which is without mixing, is very slow as the reaction occurs in oil and alcohol interfaces such as methanol. The yield of biodiesel at different mixing speeds is recorded and given in Fig. 7. As it can be seen from Fig. 7, the highest yield, which is 86%, is obtained with 600 RPM.

Process Optimization

a. Methanol to oil ratio

The most effective variable for the biodiesel production yield is identified as methanol to oil molar ratio for transesterification reaction. As transesterification is an equilibrium

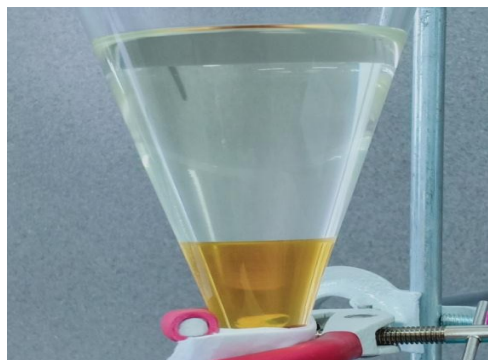


Fig. 3: Separation funnel - FAME at the upper layer and glycerine at the bottom layer.



Fig. 4: Two layers after washing the FAME with distilled water 50% by FAME volume, the top layer is washed biodiesel.

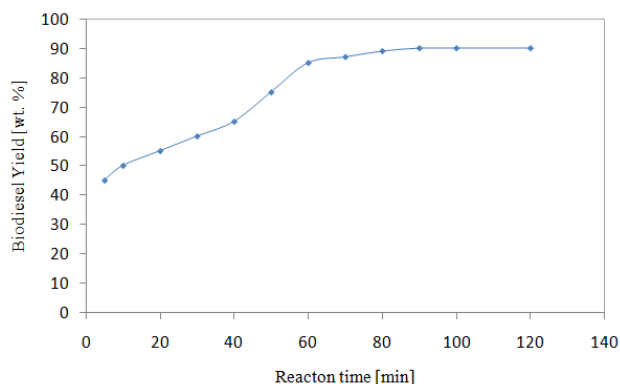


Fig. 5: Effect of reaction time on oil conversion efficiency, under 20 vol% methanol, 1 wt.% KOH at 60°C and 600 RPM.

reaction, an excess amount of alcohol is required to move the reaction forward (Bournay et al. 2005).

The biodiesel production is analyzed by changing the methanol volume with the fixed amount of 100 mL oil sample and 1% KOH by weight. The methanol amount varies from 10% to 30% by oil volume (2.3: 1 to 7:1 molar ratio of methanol to oil). It has been recorded that with the increase of methanol, the yield of the FAME also increased, and the result is given in Fig. 8. However, because there is insufficient Methanol for the reaction, the methanol to oil ratio of 2.3 results in the formation of a unique and opaque layer due to the presence of unreacted triglycerides. However, 20% methanol amount is selected by considering the yield and the cost of methanol, which will directly affect the production cost.

b. Effect of catalyst concentration

The most commonly and widely used catalyst is KOH, which is used for the reaction as they react with the triglycerides to break them apart and form the methyl ester. A wide concentration range of KOH from 0.5% to 2.5% (wt/wt of oil) has been tested as a catalyst that was premixed with methanol to

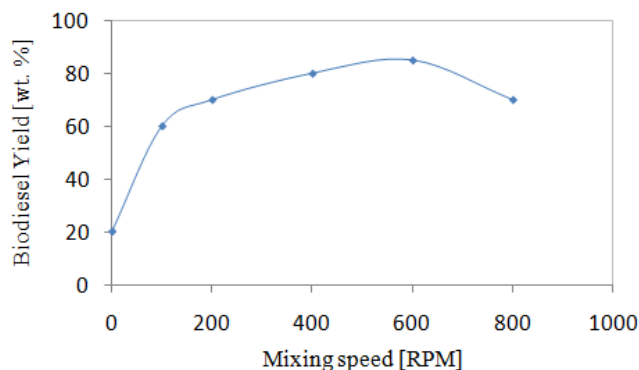


Fig. 7: Effect of stirring speed (RPM) on oil conversion efficiency, under 20 vol% methanol, 1 wt.% KOH, at 80 min reaction time and 60°C temperature.

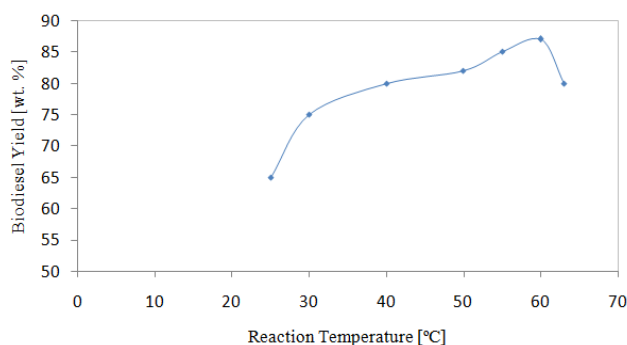


Fig. 6: Effect of reaction temperature on oil conversion efficiency, under 20 vol% methanol, 1 wt.% KOH at 80min reaction time and 600 RPM.

form sodium methoxide (CH_3ONa). The behavior of NaOH concentration regarding the biodiesel production yield is shown in Fig. 9.

The highest biodiesel yields (86%) were obtained with a 1% catalyst concentration. However, as the concentration of KOH increased over 1%, biodiesel production decreased due to the formation of an emulsion, which increased the viscosity of the product.

c. Effect of methanol to methoxide ratio

KOH is the catalyst of the alkaline esterification reaction to produce biodiesel. It has been followed all the time to used Methoxide to the oil to produce the biodiesel. However, changing the pure Methanol and Methoxide separate samples added to the oil for biodiesel production seems promising. The methanol 5% and Methoxide 15%, 10% of Methanol with 10% of Methoxide and 15% of Methanol and 5% of Methoxide were added separately into oil samples to check the reaction performance of the biodiesel yield. Based on the observations, biodiesel was formed with each case, and yield was analyzed for further processing (Fig. 10).

The maximum yield was achieved at the 5% Methanol and 15% Methoxide sample, around 95%. Other operating

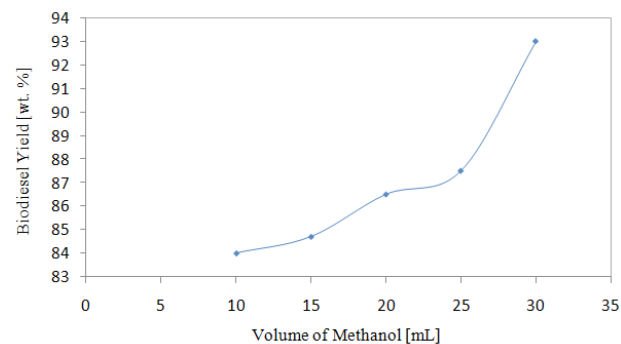


Fig. 8: Effect of Methanol to oil ratio on oil conversion efficiency, under 1 wt.% KOH, at 80min reaction time and 60°C temperature.

parameters were maintained at the fixed condition during the process, such as reaction time, temperature, KOH concentration, and the RPM of the mixer as base case values. The yield obtained with the new raw material setup was much higher than the conventional methoxide reaction with the oil sample.

d. Effect of the time interval between methanol and methoxide

The KOH is not the reactant of the transesterification process; it is only the catalyst. As a result, it is critical to examine catalyst activity to comprehend the catalyst's true significance. It was done by maintaining the time interval between the addition of pure methanol to the oil sample and the addition of methoxide to the reactor constant. The biodiesel process was performed and the yield of the process was examined by varying the time interval. The 'x' axis of Fig. 11 indicates the time between methanol to methoxide feeding. However, the total reaction time was maintained as 90 min for the production process. Fig. 11 shows no significant variation of the yield with the different time intervals of methanol and methoxide. For the reaction to begin, methanol and methoxide must be added to the oil sample at the same time. The methanol sample was introduced to the oil sample at a 70-minute interval for methoxide addition, with a maximum yield of 95%. Following that, the biodiesel yield decreased. Therefore, 20 min of reaction time (90min-70min) is sufficient for biodiesel production with a 95% yield.

Therefore, the experiment is repeated to identify the optimum reaction time with the selected 5% methanol + 15% methoxide mixer as the biodiesel process catalyst. The experimental setup we used to develop Fig. 5 is repeated to identify the optimum reaction temperature. Fig. 12 represents the maximum yield that is obtained at 20 min reaction time.

Biodiesel Quality comparison

The optimum final product of methyl ester developed by optimized parameters was tested for its composition, as shown in Table 3. The methyl ester's primary fatty acid composition

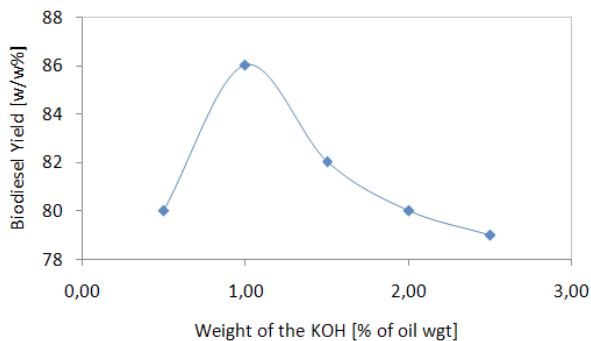


Fig. 9: Effect of catalyst concentration on oil conversion efficiency, under 20 vol% methanol, at 80min reaction time and 60°C temperature.

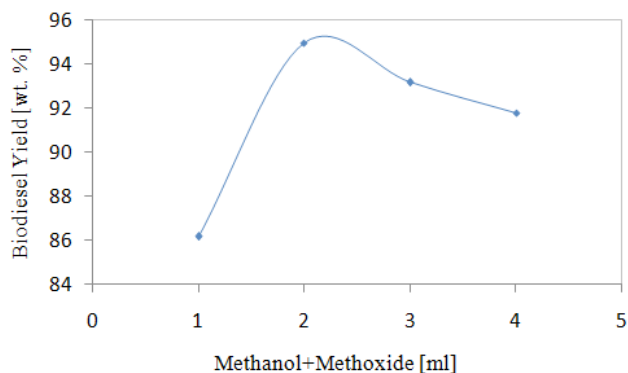


Fig. 10: Effect of methanol to methoxide ratio on oil conversion efficiency, under 20 vol% total methanol, 1 wt.% KOH, at 80min reaction time and 60°C temperature.

1: 0%Methanol+20% methoxide, 2: 5% methanol+15% methoxide, 3: 10% methanol+ 10% methoxide, 4: 15% methanol+5% methoxide

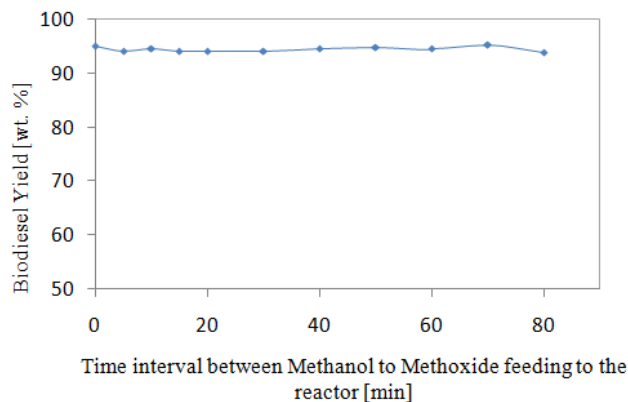


Fig. 11: Effect of the time interval between methanol to methoxide feeding on Oil conversion efficiency, under 20 vol% total methanol, 1 wt.% KOH, at 90min total reaction time and 60°C temperature.

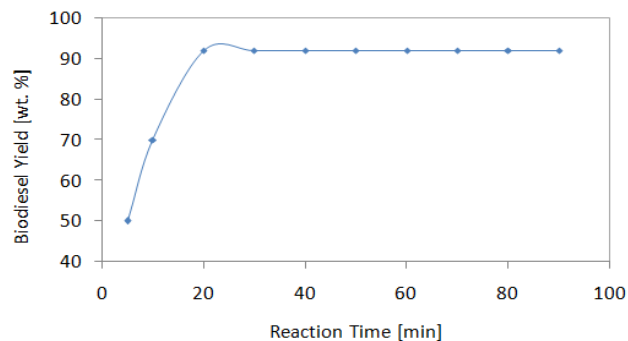


Fig. 12: Effect of reaction time on Oil conversion efficiency, under 5 vol% Methanol + 15%Methoxide, 1 wt.% KOH, at 60 °C and 600 RPM.

Table 3: Fatty acid composition of methyl ester (biodiesel).

Acid type	Formula	Method	Composition % ^a
Caproic acid	C ₆ H ₁₂ O ₂	CML/MM/05/51/003	0.34
Caprylic acid	C ₈ H ₁₆ O ₂		6.94
Capric acid	C ₁₀ H ₂₀ O ₂		5.68
Lauric acid	C ₁₂ H ₂₄ O ₂		46.72
Myristic acid	C ₁₄ H ₂₈ O ₂		19.45
Palmitic acid	C ₁₆ H ₃₂ O ₂		9.31
Stearic acid	C ₁₈ H ₃₆ O ₂		2.98
Oleic acid	C ₁₈ H ₃₄ O ₂		6.78
Linoleic acid	C ₁₈ H ₃₂ O ₂		1.65
Arachidic acid	C ₂₀ H ₄₀ O ₂		0.09
Linolenic acid	C ₁₈ H ₃₀ O ₂		0.04
Behenic acid	C ₂₂ H ₄₄ O ₂		0.02

a: Tested by the Chemical and Microbiological Laboratory, Industrial Technology Institute (ITI), Colombo 07, Sri Lanka

Table 4: Analysis of physical and chemical properties of biodiesel and petroleum diesel.

Parameter	Test Method	Values achieved in this study ^a	Petroleum diesel ^b	Standard Biodiesel - EN14214 ^b
Density [15°C, kg.m ⁻³]	ASTM D 1298-99	884.3	NA	860-900
Flash Point [°C]	ASTM D 3278-96	109	>65	>101
Cloud Point [°C]	ASTM D 2500 - 99	8.0	NA	NA
Kinematic Viscosity at [40°C, cSt]	ASTM D 445 - 01	3.842	3.0 - 8.0	3.5 - 5.0
Total sulfur content [wt. %]	ASTM D 129-00	0.003	<0.05	<0.01
Moisture content [wt. %]	ASTM D 4377 - 00	0.20	NA	<0.05
Gross calorific value moisture free basis [MJ.kg ⁻¹]	ASTM D 240 - 06	39.3	41.8	NA
Micro carbon residue [m/m%]	ASTM D 4530-06	0.06	0.3	0.3
Total acid number [mg KOH.g ⁻¹]	ASTM D 664/18	0.20	<0.1	<0.5
Cetane index	ASTM D - 91	39.5	>49	>51

a: Tested by the Petroleum and Lubricant Testing Laboratory, Industrial Technology Institute (ITI), Colombo 07, Sri Lanka

^b: Meng et. al, 2008.

is methyl laurate, 46.72%, methyl myristate 19.45%, and methyl palmitate, 9.31%. The fatty acid type and composition analysis influence the cetane number, affecting ignition quality in the engine.

The physical and chemical properties play a vital role in the qualifying process of diesel engines' operation. Biodiesel derived from coconut oil was analyzed by Petroleum and Lubricant Testing Laboratory, Industrial Technology Institute, Sri Lanka, and the results are shown in Table 4.

Biodiesel, as an alternative fuel, should have some physical and chemical properties that make it suitable for use in diesel engines. These properties play a vital role in quality control in the petroleum industry. Biodiesel derived

from fresh coconut oil was analyzed at the Petroleum and Lubricant Testing Laboratory, Industrial Technology Institute (ITI), Colombo 07, Sri Lanka, and the results are shown in Table 4. As is indicated, the biodiesel sample meets EN14214 standards for density, kinematic viscosity, carbon residue, and acid value. The increased flashpoint of the biodiesel sample produced is advantageous for safe operation. The decreased sulfur concentration is beneficial since it lowers SO₂ emissions during the combustion process.

CONCLUSION

The following values were obtained for the maximum yield; Reaction time: 20 min, Reaction temperature: 60°C,

RPM: 600, Methanol: 20 vol%, KOH: 1 wt.%, Methanol + Methoxide: 5%+15%. A maximum biodiesel yield of 95% is achieved with the optimized operating conditions. It has been shown that adding fresh methanol and methoxide into the coconut oil sample produces a higher yield of biodiesel with minimum reaction time. Rather than the conventional biodiesel production method, this process of biodiesel production has less energy consumption as well. The yielded methyl ester was tested for its fuel properties and was in good agreement with the standard biodiesel. However, the use of edible oil for biodiesel production is not encouraging due to the crisis of the food industry. As a result, the results of the experiments are used to improve the production process and operating parameters. Other sources of biodiesel production such as waste cooking oil, algal oil, non-edible oil should be implemented with the optimized operating conditions.

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