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The effect of ultrasound duty cycle in biodiesel production from Ceiba pentandra

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Abstract. This study investigated the effect of the duty cycle for the transesterification of Ceiba pentandra oil. The important parameters of a duty cycle (pulse-mode operation) and energy usage in the transesterification reaction process were presented and showed that a maximum biodiesel yield of 99.24 % was achieved for 75% duty cycle with a pulse combination of 6 sec ON and 2 sec OFF. That was under the optimum conditions of 60% methanol to oil ratio, 1.00 wt% of KOH, reacted for 50 minutes. The obtained biodiesel was then analyzed using FTIR spectroscopy, and physiochemical properties of the Ceiba pentandra biodiesel then determined. It found that all the properties comply with the fuel specifications set by both ASTM D6751 and EN 14214 standards.

1. Introduction

The world is experiencing significant growth since the end of World War II in 1945. Commonly referred to as the Great Acceleration, there is a tremendous surge in the usage of natural resources. It is used mainly to power the economy and population growth. From 1995 to 2015, the global energy usage has seen an exponential rise from 8,588.9 Mtoe to 13,147.3 Mtoe, an increment of more than 53% [1]. More alarmingly, the forecast showed the global energy demand for natural gas would increase by a whopping 50%, while coal and petroleum will increase by 15% by 2040 [2]. This high consumption has led to a prediction that the world will run out of fossil fuels, especially petroleum. With the current extraction rate of 94,718 thousand of barrels per day, the global petroleum reserve an expected to be deplete in merely 53 years [3]. This prediction triggers an alarm, especially in the transportation sector, since it relies heavily on various types of fuels originating from petroleum.

Nevertheless, there is another equally or a more severe issue due to the increase of global fossil fuel consumption, which is the global warming phenomenon. Due to the unprecedented fossil fuel usage, this, in turn, increased CO and CO₂ emissions, which consequently alter the equilibrium of the ecosystem disastrously, leading to global warming [4]. Even though the impact of global warming would differ from one place to another, the effectshowed in various ways, such as the rise in sea level, extreme change in weather which lead to draughts or unusual floods, and the increasing appearances of hurricanes [5].

Biofuel is considered a holistic approach that has a tremendous potential to solve both global warming and the depletion of fossil fuels simultaneously. It can see as an alternative source of energy

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that is considered sustainable and less harmful to the environment, compared to the conventional form of energy that originates from fossil fuel [6,7]. Biodiesel is a form of biofuel derived from various sources of plants or animal fats. First-generation biodiesel is often heavily criticized because it derives from edible vegetable oils, which prioritized to remedy world hunger. Hence, a lot of attention gave to second-generation feedstock, which consists of non-edible vegetable oils, as an effort to produce biodiesel.

In recent years, a lot of research alrady been conducted on non-edible feedstocks such as waste cooking oil [8], Pongamia pinnata [9], Ceiba pentandra [10], Jatropha curcas [11], Calophyllum inophyllum [12], and microalgae [13] for biodiesel production. Biodiesel originated from second-generation feedstocks are proven to be desirable due the positive characteristics such as good oxidation stability, excellent cold flow properties, and high production yield [14]. The non-edible Ceiba pentandra, which is also called kapok, is seen as a very promising alternative feedstock, especially when combined with cutting-edge production methods, which could reduce both the production cost and the pollutant emission [10]. Even though kapok originates from West Africa and America, the plant can now be abundantly found in Malaysia, Vietnam, India, and Pakistan [15]. The Mature kapok tree is typically around 60 - 70 m in height, producing 1000 – 4000 seed pods at a time [16]. In a pod, kapok seed surround by fluffy fibre. Normally, it uses to make mattresses and pillows. Regarding waste, kapok seeds contain around 22 wt% of oil, which can use to produce biodiesel [17]. Kapok regard as a highly potential candidate used in biodiesel production due to its capacity to produce an average of 1280 kg of oil per hectare annually [18]. Hence, the usage of kapok is in line with the need to use non-edible feedstock in producing biodiesel.

Ultrasound is considered one of the promising technologies in the production of biodiesel. Besides consuming less than one-third of the energy compared to the conventional method, it also gives a higher yield of the final product [19–21]. A collective of acoustic phenomena during an ultrasonic reaction resulted in what refers to as the cavitation effect. During reacted, micro bubbles are formed and continue to grow until they collapse (in a matter of nanoseconds), releasing tremendous energy, which leads to intense localized heating reaching the temperature and pressure of about 5000K and 1000atm, respectively [22,23]. Due to the nature of the ultrasonic reaction, it reported that the production time of biodiesel reduces efficiently, shortening the time by more than 63.3% [24].Besides, the usage of ultrasound is also known to have a better biodiesel yield compared to the conventional transesterification method [25,26]. Hence, ultrasound irradiation considered being both effective and efficient method for producing biodiesel.

The modes of sonication also have an essential factor in biodiesel yield. It is because of an ultrasound used in the transesterification reaction. A previous study showed direct sonication is superior in energysaving to indirect sonication. Direct sonication applies via horn or probe, while indirect sonication is like a sonicator bath [27]. The pulse sonication (5 sec ON, 1 sec OFF) led to a higher biodiesel yield in the amount of 98%. Meanwhile, continuous sonication only about 93.5% [28]. Even though there are numerous studies done on the effect of duty cycle to the final biodiesel yield in ultrasound-assisted transesterification reactions, none of the studies could come to the similar conclusions on the optimum pulse to be used in all reactions. It showed that the results depend on various factors, including the types of alcohol donors, feedstock, and used catalyst [29–31].

To the best of our knowledge, no research has been conducted on the effect of an ultrasound pulse in biodiesel production from Ceiba pentandra. This research work aim is to measure the transesterification reaction time, catalyst loading, ratio of methanol to oil, and duty cycle on the overall biodiesel yield. The properties of the biodiesel obtained from Ceiba pentandra oil are measured using ASTM D6751 or EN 14214 to ensure its compliance with the standards.

2. Material and Methods

2.1. Material and experimental set-up

The feedstock used in this study is Ceiba pentandra, where the readily available oil procured from Cilacap, Centre Java, Indonesia. All chemicals (methanol, sulfuric acid, and potassium hydroxide)

sourced from Sigma-Aldrich, Malaysia. The sonicator device used in this study (Qsonica Q500) has a power rating of 500W and 20 kHz frequency equipped with a0.5-inch solid titanium probe. A borosilicate glass beaker with a capacity of 100ml used as a reactor. Figure 1 shows the experimental setup used in this study.



Figure 1 Schematic diagram of experiment

2.2. Esterification and transesterification of Ceiba pentandra oil

Pre-treatment needs did on the Ceiba pentandra oil in the effort to lower the free fatty acid before the transesterification reaction. In general, it accepted that the free fatty acid value needs to be reduced to below 2% to avoid saponification process, which in turn will lead to a lower methyl ester yield [32]. The acid-catalyzed esterification process was done by following the procedure set by Silitonga et al. [33]. For this purpose, 1 liter of Ceiba pentandra oil was mixed with methanol at 8:1 methanol to oil molar ratio. 1% (v/v) sulfuric acid (H₂SO₄) added before the mixture was heated at 60°C for 2 hours while being stirred at 1200 rpm. Once the reaction was completed, the mixture poured into a separating funnel and left to settle under gravity for 4 hours. As a result, two distinct layers, which consist of methanol and esterified Ceiba pentrandra oil formed inside the funnel. The bottom layer, (esterified Ceiba pentrandra oilwill discharge from the funnel and then placed in a rotary evaporator for 60 minutes at 65°C to remove the left methanol from the oil.

For the transesterification process, 20g of the esterified oil was poured into a 100ml glass beaker, as a reactor. Potassium hydroxide (KOH) catalyst varying from 0.50, 0.75, 1.00, and 1.25 wt.% were diluted with methanol in the amount of 40, 50, 60 and 70 wt. % before being poured into the esterified oil. Different reaction times of 30, 40, 50, and 60 minutes were used in the transesterification reaction. After the reaction ended, the mixture was poured into a separating funnel and left for 8 hours. Two visible layers were formed, where the lower layer (glycerol) was removed, leaving the upper layer (methyl ester) inside the funnel. The methyl ester then washed several times using warm distilled water to remove the impurities. After discarding the water from the funnel, the product transfer to a rotary evaporator set at 60°C for 1 hour to further remove any water and methanol leftover. Finally, the methyl ester was filtered using Whatman filter paper. The biodiesel yield was quantified using Equation (1) [10]:

$$Biodiesel \ yield \ (\%) = \frac{weight \ of \ Ceiba \ pentandra \ methyl \ ester \ (g)}{weight \ of \ Ceiba \ pentandra \ oil \ (g)} \ x \ 100$$
(1)

2.3. Duty cycle

The Duty cycle plays an essential role in an ultrasound-assisted reaction. It defines as the ratio of the pulse duration (ON) to the total time in a cycle (ON+OFF) that set in a sonicator, as shown in Equation 2 [27]:

$$Duty cycle (\%) = \frac{time \ on \ (s)}{total \ time \ (on+off)} \ x \ 100$$
(2)

The Duty cycle is an ideal way to evaluate the overall energy requirement involved in an ultrasound-assisted reaction. It can further expand to gauge the overall economics of that reaction, leading to any possible saving while keeping the product yield in check. Meanwhile, the number of cycles is define as the number of ON pulse repetition during the entire process.

2.4. FTIR analysis

The functional groups of obtained product identified by using FTIR spectroscopy (Shimadzu IRPrestige -21), looking at the range wave number between 4000–650 cm–1. It is to identify the existance of methyl ester in the final product.

2.5. Properties of Ceiba pentandra methyl ester (CPME)

ASTM D6751 and EN 14214 standard test procedures, used to evaluate various properties of the CPME, such as acid value, density, calorific value, and kinematic viscosity. The acid value obtained using Mettler Toledo Titration Automation Rondo 20, while the density was determined using Mettler Toledo DM40 LiquiPhysics. Meanwhile, Parr 6200 Isoperibol Calorimeter utilized to evaluate the calorific value, and the kinematic viscosity of the methyl ester determines at 40 °C by using Anton Paar SVM3000.

3. Results and Discussions

3.1. Effect of Reaction Time

In a transesterification process, usually, reaction time plays a dominant factor that effects the overall biodiesel yield. The influence of reaction time, varied at 30, 40, 50, and 60 min accordingly, was assessed while keeping other parameters constant with ratio of methanol to oil at 50 (w/w)%, KOH catalyst concentration at 0.75 (w.t.)%, ultrasound amplitude at 30% with 5 sec ON and 2 sec OFF pulse. Figure 2 shows that the CPME yield steadily increases between the 30 - 50 min reaction time (peaking at a yield of 96.31%), after which it drops to 94.25 % at 60 min reaction time. It is because of the nature of the transesterification process, where the reversible reaction could happened at an extended period of reaction time [34].



Figure 2 Relationship between CPME yield and reaction time

3.2. Effect of KOH catalyst concentration

To investigate the effect of KOH catalyst concentration on the CPME yield, different catalyst concentrations (0.50, 0.75, 1.00, and 1.25 wt.%) were used while fixing the ratio of methanol to oil at 50(w/w)%, ultrasound amplitude at 30% with 5 sec ON and 2 sec OFF pulse. Previously, this investigation uses a determined optimum time of 50 min. From Figure 3, it observed that as the KOH catalyst concentration increases from 0. 50 to 1.00 wt.%, CPME yield also increases from 92.5 % to 98.2 %. After peaking at 1.00 wt.%, the CPME yield reduces to 96.85% when a catalyst uses a concentration of 1.25 wt.%. The excessive usage of the catalyst in a transesterification process can make saponification as a side reaction. Hence, leading to a lower methyl ester yield. From the graph, it determined that the optimum KOH catalyst concentration in this study to be at 1.00 wt.%, with a 98.2% CPME yield.



Figure 3 Relationship between CPME yield and the KOH catalyst concentration

3.3. Effect of methanol to oil ratio

Methanol to oil ratios in different percentages of 40, 50, 60 and 70 (w/w) % used to evaluate the effect of this parameter on this study. For this purpose, other parameters such as reaction time and KOH catalyst concentration are kept constant at 50 minutes and 1.00 wt.%, respectively. Again, t ultrasound's amplitude was fixed at 30%, with a pulse of 5 sec ON and 2 sec OFF. Figure 4 showed CPME yield increases from 90.42 to 98.28%, when the ratio of methanol to oil increases from 40 to 60 (w/w)%. Nevertheless, a lower CPME yield obtained when methanol's ratio to oil increases from 60 to 70 (w/w) %. It is explained by the high soluble glycerol that left in the biodiesel phase, which resulted in a reverse reaction or a more difficult separation process [14].



Figure 4 Relationship between CPME yield and the ratio of methanol to oil

3.4. Effect of duty cycle

The various duty cycle (50, 55, 60, 65, 70, 75 and 80 %) used to determine the effect of a duty cycle on the CPME yield. While reaction time, KOH catalyst loading, and ratio of methanol to oil were kept constant at 50 min, 1.00 wt.%, and 60 (w/w)%, respectively. Figure 5 showed CPME yield increased from 93.52 to 99.24% from duty cycles of 50 - 75%, respectively. Nevertheless, the CPME yield starts decreasing from 99.24 to 95. 48% when the duty cycle increases from 75 to 80 (w/w) %. It is because of the reaction's high temperature as a result of a high duty cycle; where a limited amount of methyl ester produces when the temperature inside is close to the methanol's boiling point. Similar occurrences reported by other researchers working on the effect of duty cycles to the methyl ester yields [27,35].



Figure 5 Effect of duty cycle to Ceiba pentandra methyl ester yield

3.5. Energy consumption

The energy consumption and the energy saved during the ultrasound-assisted production of CPME under optimized conditions shown in Table 1, which corresponds to the reaction time of 50 minutes used in this study. With the 500W sonicator used, the energy consumed during the reaction time is calculated via Equation (3).

Energy (Joule) = Power (Watt) x time (sec)
$$(3)$$

Besides, energy saved in during a pulse mode sonication can be quantified by calculating the total OFF time during the entire process. It can be observed that an energy saving of 50% are possible with yield of 97.22% using 66.67% duty cycle. The maximum yield in this study achieved by energy saving of 33.33% with 75% duty cycle with 99.24% yield biodiesel.

Condition		Duty	Time on	Time off	Consumed	Saved	Yield	
0]	N (s)	OFF (s)	Cycle (%)	(min)	(min)	Energy (kJ)	Energy (kJ)	(%)
	4	4	50.00	50.00	50.00	1500	1500	93.52
	5	4	55.56	50.00	40.00	1500	1200	94.65
	6	4	60.00	50.00	33.33	1500	1000	96.52
	4	2	66.67	50.00	25.00	1500	750	97.22
	7	3	70.00	50.00	21.43	1500	643	98.15
	6	2	75.00	50.00	16.67	1500	500	99.24
	4	1	80.00	50.00	12.50	1500	375	95.48

Table 1 Various duty cycles and the corresponding consumed and saved energy

3.6. FTIR analysis

Figure 6 showed the characteristics peaks of the CPME produced under optimum conditions found to be at 3008 cm-1, 2923 cm-1, 2854 cm-1, 1742 cm-1, 1436-1460 cm-1 and 1170 cm-1. These corresponds to the vigorous absorption intensity for the C–H stretching vibration, vigorous absorption intensity for the CH₂ asymmetric and symmetric vibration, vigorous absorption intensity for C=O stretching vibration, mild absorption intensity for the CH₂ shear-type vibration and mild absorption intensity for C–O–C symmetric stretching vibration, respectively.



Figure 6 FTIR spectrum of CPME produced using optimum conditions

3.7. Ceiba pentandra methyl ester properties

Table 2 showed the fuel properties of petrodiesel and CPME. It produced using optimum conditions via ultrasound-assisted transesterification process. Properties of the CPME such as kinematic viscosity at 40°C, density at 15°C, acid value, and calorific value found to comply with the criteria set by both ASTDM D6751 and EN 14214 standards. Hence, this shows that the obtained CPME can utilize as a substitute for petrodiesel.

Properties	Units	Standard test	ASTM	EN 14214	Petro	CPME
Ĩ		methods	D6751		diesel	
Kinematic viscosity at	mm ² /s	D 445	1.9-6.0	3.5-5.0	2.96	4.77
40°C						
Density at 15°C	kg/m ³	D 1298	860-880	860-900	846.1	879.6
Acid value	mg KOH/g	D 664	Max. 0.5	Max. 0.5	0.017	0.24
Calorific value	MJ/kg	D 975	Min. 35	Min. 35	45.361	40.385

Table 2 Physicochemical properties of biodiesel from CPME and petro diesel

4. Conclusions

To summarize, the duty cycle's effect on the transesterification of CPME production study. The result is that the optimum methyl ester yield was 99.24% for 75% duty cycle (6 sec ON and 2 sec OFF). It also found that a saving of 50% energy can obtain by using sonication pulse of 4 sec ON, 2 sec OFF, while giving a CPME yield of 97.22%. The obtained CPME in this study conforms to both ASTDM D6751 and EN 14214 standards.

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