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Synthesis of *Ceiba pentandra* biodiesel using ultrasound and infrared radiation: Comparison and fuel characterisation.

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Abstract. The continuous expending of the economy and population in modern society has caused an increase in energy usage. Currently, fossil fuels and renewable energy are used to generate energy, contributing to greenhouse gas emissions. A significant effort has been made globally to address the issue of rising emissions by boosting the usage of renewable energy. In comparison to fossil fuels, biodiesel has many benefits, including the ability to be produced from a wide range of feedstocks, the ability to be renewable, and the reduction of atmospheric pollution emissions. Besides, advanced technologies can help the biodiesel sector meet the energy demand while producing high-quality biodiesel. The *Ceiba pentandra* was used for biodiesel production using ultrasound-infrared applications in the present research work. The study aims to produce biodiesel for a better conversion rate and improve fuel properties. Comparisons were conducted using a combination of infrared ultrasound versus ultrasound irradiation. The results show that ultrasound produced the highest yield of 98.76% when the conditions were as follows: methanol/oil ratio: 60%, KOH: 1%, reaction time: 50 minutes. Yet, the addition of infrared on ultrasound has also produced a high conversion yield in a shorter time than ultrasound. A 98.42% biodiesel yield option when using infrared-ultrasound irradiation with conditions as follows: methanol/oil ratio: 60%, KOH: 1%, reaction time: 30 minutes. As both applications were examined, the ultrasound-infrared application was preferable in saving time and energy constraints for biodiesel production. The fuel properties were found to be equivalent to ASTM D6751 and EN 14214 biodiesel standards.

Keywords: Biofuel, Energy, ESG, Sustainable feedstock, Non-edible oil

1. Introduction

Biodiesel is gaining attention as a promising fuel due to its potential to reduce greenhouse gas emissions and its compatibility with existing diesel engines. Biodiesel has several key characteristics that make it a desirable alternative to conventional diesel fuel. One of the main characteristics of biodiesel is its biodegradability, which means that it breaks down naturally in the environment, reducing its impact on the ecosystem. Additionally, biodiesel has a higher lubricity compared to conventional diesel, which can help extend the lifespan of engines and reduce maintenance costs. Another important characteristic is its lower emissions of sulphur and other pollutants, making it a cleaner option for the environment. Furthermore, their higher flash points make biodiesels more convenient for storage and transportation. Biodiesels exhibit superior combustion, engine performance, and exhaust emission qualities in comparison to diesel, featuring lower levels of carbon monoxide (CO), particulate matter (PM), and unburned hydrocarbons (UHC) [1]. The growing energy requirements from industrialization have contributed to a substantial rise in crude oil imports, particularly in developing countries. Apprehensions about food security have prompted the formulation and implementation of policies that prioritize the production of biofuels from non-agricultural origins. To address this demand, scientists and researchers are actively exploring methods to generate biodiesels from non-edible feedstocks, as well as macroalgae and microalgae [2].

Selecting a non-edible oil for biodiesel production involves several key factors, as the choice of feedstock significantly impacts the biodiesel's properties, yield, and environmental sustainability. To seek sustainable and renewable sources that are abundant and not in direct competition with food production [3]. Higher oil content is preferable as it leads to higher biodiesel yield. Hence, *ceiba pentandra* can be considered a suitable candidate for biodiesel production [4]. The processed ceiba pentandra biodiesel can be distributed and used as a renewable and sustainable alternative to traditional fossil diesel fuels. It can be used in diesel engines with little or no modification and can help reduce greenhouse gas emissions and dependence on fossil fuels [5]. The produced biodiesel undergoes quality control tests to ensure it meets specifications and standards required for biodiesel [6, 7]. The technology used plays a significant role in improving biodiesel production processes [8, 9]. It is known that ultrasound technology enhances biodiesel production as ultrasound promotes the rapid and efficient mixing of reactants by generating acoustic cavitation bubbles [10]. This cavitation effect enhances mass transfer and increases the rate of the transesterification reaction, reducing reaction times and improving biodiesel yield. Ultrasound assists in uniform temperature distribution within the reaction mixture, minimizing temperature gradients and preventing localized hotspots, which can be problematic in conventional heating methods. The limitation of the ultrasound is preheating the reaction mixture to be at a certain temperature typically around 60-70 °C for the esterification and transesterification reactions to occur efficiently. Therefore, to mitigate this limitation, we are exploring strategies to improve the energy efficiency of ultrasound-assisted biodiesel production by adding the infrared bulb to the system [11]. Infrared coupled with ultrasound works more efficient as preheating the mixture to the required temperature of 60 °C has help to reduce the preheating time. This study explores the synthesis and analysis of biodiesel derived from *Ceiba pentandra* through the integration of an ultrasound system with infrared technology.

2. Materials and methods

In order to produce ceiba pentandra biodiesel, the following materials and methods were employed.

2.1 Materials

The following substances were utilized in the production of biodiesel: CH_3OH, (2) H_2SO_4, (3) Na_2SO_4, (4) KOH, (5) reference material of carboxylic acid (6) nonadecanoic acid methyl ester, and (7) phenolphthalein solution (1% in ethanol). The properties of crude *Ceiba pentandra* and their fatty acid composition are presented in Table 1 and Table 2, and C*eiba pentandra* was procured from Indonesia.

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Table 1. Properties of crude *Ceiba pentandra*.

Properties	Unit	Test method		Crude Ceiba Pentandra
		ASTM D6751	EN 14214	
Kinematic viscosity at 40 °C	mm^2/s	D 445	EN ISO 3104	18.74
Density at 15 °C	kg/m^3	D 127	EN ISO 3675	906.50
Acidity	mgKOH/g	D 664	EN 14104	16.20
Iodine number	$1_2/100g$	–	EN 14111	116.70
Heating value	MJ/kg	D 240	–	38.67
Flash point	°C	D 93	EN ISO 22719	186.50
Water content	% (v/v)	D6304	EN ISO 12937	0.05

Table 2. *Ceiba pentandra*'s fatty acid composition.

Name	Composition (%(w/w))
$C_{12}H_{24}O_2$ (dodecanoic acid)	0.1
$C_{14}H_{28}O_2$ (tetradecanoic acid)	0.1
$C_{16}H_{32}O_2$ (hexadecanoic acid)	20.8
$C_{16}H_{30}O_2$ (cis-9-hexadecenoic acid)	0.5
$C_{18}H_{36}O_2$ (octadecenoic acid)	4.2
$C_{18}H_{34}O_2$ (cis-9-octadecenoic acid)	17
$C_{18}H_{32}O_2$ (linolic acid)	38.2
$C_{18}H_{30}O_2$ (alpha-linolenic acid)	1.3
$C_{20}H_{40}O_2$ (icosanoic acid)	0.8
$C_{18}H_{32}O_2$ (Halphen acid)	16.8
Saturated	26
Unsaturated	73.8

2.2 Experiment set-up

The biodiesel production reactor setup employed a 250 mL borosilicate beaker, metal foil, and a temperature indicator. An ultrasonic cavitation was generated within the mixture using a Qsonica (Q500-20) sonicator with a 500 W power rating and a 20 kHz frequency. The sonicator was equipped with a 1-inch probe, and its settings were established with an amplitude fixed at 35% and a pulse configuration of 6 seconds ON followed by 2 seconds OFF. To expedite the transesterification process, the reactor was equipped with 300 W infrared lamps operating at 220 V, emitting heat radiation. This infrared radiation was applied with the specific purpose of accelerating the chemical reaction. The set-up box is made from wood and the surrounding is enclosed with aluminium foil. The biodiesel synthesis set-up that combines ultrasound with infrared radiation is depicted in figure 1.

2.3 Esterification process

Acid-catalysed esterification is necessary for crude *Ceiba pentandra* oil as the free fatty acid (FFAs) content is high ~ 8.1mg KOH/g. High FFAs content will cause saponification during the base catalyst conversion process, greatly reducing the yield and quality of the target product. The parameters for the acid-catalysed esterification process are a CH_3OH/oil ratio of 70 vol.%, heat radiation maintained at 60 °C, an H_2SO_4 catalyst of 1.8%, and a reaction time of 30 minutes using ultrasound equipped with the infrared radiation system. Upon completion, the mixture is transferred to a separating funnel and allowed to settle for a duration of 6 hours. Distinguishable layers can be observed: the lower layer comprises esterified oil, while the upper layer consists of methanol and any excess H_2SO_4. The acidity of esterified oil is 1.4 mg KOH/g, which is reduced to below 2 mg KOH/g, enabling the esterified oil to undergo the second process (transesterification).

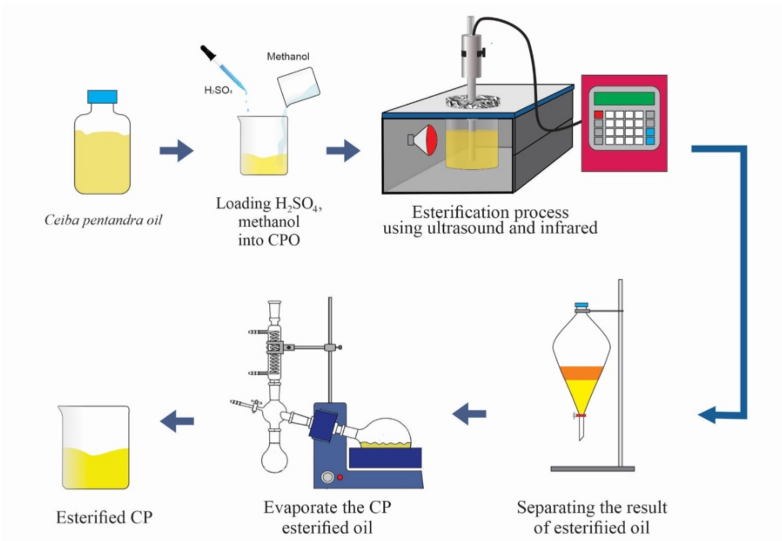

Figure 1. Esterification process of ceiba pentandra using infrared bulb coupled with ultrasound.

2.4 Base catalytic process

The product from esterification is used for the base catalytic process. The oil was initially heated to 60 °C using an infrared bulb; upon reaching the required temperature, the ultrasound was turned on. The usage of the infrared bulb gradually decreased. The factors for the base catalytic process are a CH_3OH/oil ratio of 60 vol.%, heat radiation maintained at 60 °C, a KOH catalyst of 1%, and duration maintained for 30 minutes using ultrasound equipped with the infrared radiation system. Upon completion, the mixture is transferred to a separating funnel and allowed to settle for a duration of 6 hours. Distinguishable layers can be observed: the lower layer is the methanol, by-products and excess KOH catalyst, while the upper layer is the targeted product. The targeted product was collected for a purification process to remove impurities and dried using a rotovap set at 60°C for 45 minutes. Dried sodium sulphate was used to remove any moisture content in the biodiesel. Then, the filtration was performed to remove the clumped anhydrous sodium sulphate and impurities. The processes of transesterification are summarised in figure 2.

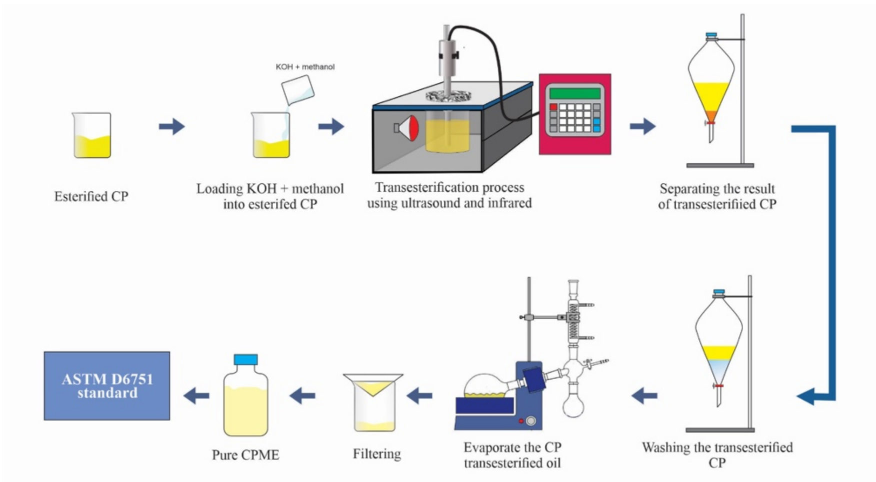

Figure 2. The transesterification process of *Ceiba pentandra* uses an infrared bulb coupled with ultrasound.

2.5 Measurement of physicochemical properties

The physicochemical properties of the *ceiba pentandra* biodiesels produced through infrared coupled with ultrasound irradiation were measured and the results were assessed with those for Ceiba pentandra biodiesel and diesel. Key properties, including kinematic viscosity at 40°C, density at 15°C, acid value, calorific value, flash point, and oxidation stability at 110°C. The measurements were conducted following the guidelines outlined in ASTM D6751 and EN 14214 standards. The converted biodiesel is measured using the equation (1).

$$Biodiesel\ (\%) = \frac{Weight\ of\ methy\ ester\ (g)}{Weight\ of\ oil\ (g)} \times 100\ (1)$$

3. Results and discussion

The analysis of the properties of *ceiba pentandra* biodiesel, according to ASTM D6751 and EN 14214 standards, was summarized in table 3. The main crucial properties that influences fuel fluidity and spray characteristics during combustion is the kinematic viscosity. Elevated kinematic viscosity can result in inadequate fuel atomization, leading to soot formation and engine deposits. The fluidity of the *ceiba pentandra* for this investigation (4.77 mm^2/s) corresponds with the findings documented by Ong et al. [12]. Based on Table 3, shows that *ceiba pentandra* biodiesel possesses the appropriate physicochemical qualities and the properties are comparable with *ceiba pentandra* produced by Ong et al [12]. The acid value of *ceiba pentandra* biodiesel is much lower (0.2 mg KOH/g) compared with others, and this indicates that *ceiba pentandra* biodiesel produced using infrared coupled with ultrasound has a high potential that allows for a high blending ratio to achieve the desired fuel properties. The best ultrasound-assisted transesterification process for *ceiba pentandra* with a methanol/oil ratio of 60 %, KOH: 1%, and reaction time of 50 minutes produced approximately 98.76%. This method required an extra 20 minutes of reaction time compared with the infrared ultrasound-assisted transesterification process, which only required a reaction time of 30 minutes, which is more time-saving and energy-saving than ultrasound alone.

Table 3. Physicochemical properties of *ceiba pentandra* biodiesel.

Properties	Units	Standard test methods	ASTM D6751	EN 14214	Diesel	CPME	CPME [12]
Kinematic viscosity at 40°C	mm^2/s	D 445	1.9-6.0	3.5-5.0	2.96	4.77	4.62

Density at 15°C	kg/m^3	D 1298	860-880	860-900	846.1	864.2	880.5
Acid Value	mgKOHg	D 664	Max. 0.5	Max. 0.5	0.017	0.2	0.38
Calorific value	MJ/kg	D 975	Min. 35	Min. 35	45.361	40.228	39.78
Flash point	(°C)	D 93	100-170 min	>120	71.5	175.5	125.5
Oxidation stability at 110°C	h	EN 14112	3 min	6 min	23.7	12.63	5.22

4. Conclusions
The physicochemical properties of *ceiba pentandr*a biodiesel were evaluated after infrared coupled with ultrasound irradiation-assisted transesterification. Our study obtained 98.42% biodiesel yield using infrared-ultrasound irradiation with the following conditions: methanol/oil ratio: 60%, KOH: 1%, temperature reaction: 60 °C, and duration: 30 minutes. By opting out of the infrared function, we need a longer reaction time, ~50 minutes, to obtain 98.76% biodiesel conversion yield, while other conditions remained the same.

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