

# Transesterification of Kapok (*Ceiba pentandra*) Oil Using Sonication

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**Abstract.** The high fatty acid content and non-edible nature of kapok (*Ceiba Pentandra*) oil make it a promising raw material for biodiesel production. However, the traditional transesterification process involves long durations and high energy consumption, necessitating a more efficient method. Sonication, using ultrasonic waves at 20-40kHz, is a leading method that reduces transesterification duration from hours to minutes. It induces cavitation, the formation and collapse of microbubbles, raising the temperature of the reaction environment and accelerating the transesterification reaction. The purpose of this research is to study the transesterification of kapok oil into biodiesel using sonication. The results showed that the biodiesel produced had a viscosity value of 5.6-5.9 cSt, which is still below the maximum value of SNI. The density value is in the range of 913.533 to 951.43 Kg/m<sup>3</sup>, this value still exceeds the SNI value of 890 Kg.m<sup>3</sup>. Both parameters were measured at 25°C which should be 40°C based on SNI. The viscosity and density values will decrease as the measurement temperature increases. The highest yield value was produced at 2 min sonication time, 65.7%, while the lowest yield value was produced at 6 min, 53.3%.

## 1. Introduction

The global need for fossil fuels continues to grow progressively. However, due to their non-renewable nature, alternative sources are necessary to meet fuel requirements. Biodiesel presents a viable solution to this issue because it is produced from plentiful renewable energy sources [1]. Additionally, biodiesel offers the benefit of being a low-emission and environmentally friendly fuel, emitting 32% less CO<sub>2</sub> per liter compared to diesel derived from petroleum [2].

Biodiesel can be derived from both vegetable and animal oils, but it is commonly produced from palm oil. However, using palm oil for biodiesel production is not ideal since it is a crucial food resource, leading to high production costs. Therefore, there is a need for vegetable oils that do not impact food supply chains, resulting in lower production costs [3]. Non-edible vegetable oils suitable for biodiesel production include Karanja seed oil (*Pongamia pinnata*), Mahua seed oil (*Madhuca longifolia* (L.) J. F. Mac), and Castor seed oil (*Jatropha Carcus*) [4], *Madhuca indica* [5], *Moringa oleifera* [6], and *Havea brasiliensis* [7]. Anwar et al [8] conducted experiments to produce biodiesel from Karanja seed oil, achieving a 95.4% yield in 120 minutes using mechanical heating and stirring. Biodiesel from Mahua seed oil can yield between 90-98% with a reaction time of 80-90 minutes using similar methods [9].

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The Indonesian National Standard (SNI) and the American Society for Testing and Materials (ASTM) define quality criteria for biodiesel, which include parameters such as density and viscosity to ensure its performance and quality. Density, which ranges from 860-900 kg/m<sup>3</sup> (SNI) and 850-900 kg/m<sup>3</sup> (ASTM) at 15°C, and kinematic viscosity, ranging from 3.5-5.0 mm<sup>2</sup>/s at 40°C in both standards, are critical indicators. Density affects the energy content and combustion characteristics of biodiesel, influencing its efficiency in diesel engines [10]. Viscosity, on the other hand, impacts the flow properties and lubricity of biodiesel, which are vital for engine operation and longevity [11]. Adherence to these standards is crucial for maintaining biodiesel's reliability, sustainability, and compatibility with existing diesel infrastructure worldwide [12].

The kapok tree serves multiple industries, including wood, fiber, and oil production. The fiber obtained from kapok fruit shows promise for use as a biopolymer [13]. The kapok tree's wood is of low density, which limits its use in the wood industry primarily to board production [14]. Despite the advantages and disadvantages of the kapok tree, its seeds have the potential to be processed into biodiesel. Retrieved from Silitonga et al [15] kapok seed oil can be utilized as a raw material for biodiesel production because it contains quite a lot of fatty acids as shown in Table 1. The potential of kapok oil in general is 2850 kg/ha of kapok land [16]. Kapok seed oil is extracted by pressing with a screw press machine continuously, then the results of the oil are filtered and heated at 120°C to remove impurities [17]. Kusumo et al [18] conducted research on biodiesel production from *Ceiba pentandra* oil using a 1 wt% KOH catalyst with the stirring method at 60°C for 108 minutes, resulting in a biodiesel yield of 99.80%.

**Table 1.** Characteristic of *Ceiba pentandra* oil

<b>Characteristics</b>	<b><i>Ceiba pentandra</i></b>
Density at 15°C	905.2 (kg/m <sup>3</sup> )
Kinematic viscosity at 40°C	34.45 (mm <sup>2</sup> /s)
Lauric acid	0.1 %
Myristic acid	0.1 %
Palmitic acid	19.2 %
Palmitoleic acid	0.3 %
Stearic acid	2.6 %
Oleic acid	17.4 %
Linoleic acid	39.7 %
Linolenic acid	1.5 %
Arachidic acid	0.6 %
Malvalic acid	18.5 %

Biodiesel production includes both esterification and transesterification reactions, which depend on the quality of the vegetable oil used [19] Esterification becomes essential when the oil's free fatty acid content exceeds 2%, as elevated levels of free fatty acids can cause saponification, thereby hindering the transesterification process [20]. This saponification process occurs due to the reaction of fatty acids with the catalyst, resulting in the formation of soap. Subsequently, the soap acts to prevent the methyl groups in the alcohol from effectively reacting with the glyceride groups present in the vegetable oil. Esterification is performed with an acid catalyst, whereas transesterification uses a base catalyst.

The predominant method employed for biodiesel production is heating with mechanical stirring, which is both time-consuming and inefficient. Alternative methods include sonication and supercritical transesterification [21]. However, the supercritical method has drawbacks, such as not reducing production costs and being challenging to scale up for large-

scale biodiesel production [22]. In contrast, sonication can significantly reduce reaction times from hours to minutes [23]. Sonication techniques have proven to be effective methods in various chemical processes, including the acetylation of crude glycerol into glyceryl triacetate [24]. Currently, sonication is a leading method in biodiesel production because it decreases the catalyst requirement for esterification and transesterification processes [25]. Sonication induces vigorous localized turbulence by creating and collapsing microbubbles in the reactants, a process known as cavitation, enhancing heat and mass transfer [21].

Thus, the objective of this paper is to investigate biodiesel transesterification from *C. pentandra* oil using sonication.

## 2. Experimental Method

### 2.1 Biodiesel Transesterification

In biodiesel production, methanol is used as a reactant due to its light specific gravity, constituting 15-20% of the oil used. Kapok randu oil undergoes pretreatment in the form of esterification with an acid catalyst,  $H_2SO_4$ , to decrease the free fatty acid content in the oil. Elevated concentrations of free fatty acids can induce saponification reactions. Transesterification is then performed using a 1% KOH catalyst, with a molar ratio of kapok oil to methanol of 1:6, over five different reaction times: 2, 3, 4, 5, and 6 minutes. This transesterification is conducted using the sonication method, employing a Mikuni ultrasound generator and a 100-watt transducer.

### 2.2 Characterization of Biodiesel

#### 2.2.1 Yield Analysis

Biodiesel yield refers to the percentage of oil that is converted into pure biodiesel. Yield measurement is conducted after the biodiesel purification process. The produced biodiesel volume is quantified and then compared to the volume of oil used.

$$\text{Yield (\%)} = \frac{\text{Weight of Biodiesel}}{\text{Weight of Oil}} \times 100\% \quad (1)$$

#### 2.2.2 Density Analysis

Biodiesel density is determined using a pycnometer. First, the pycnometer is prepared and weighed while empty. Biodiesel is then added to the pycnometer, which is sealed tightly to ensure no bubbles remain. The pycnometer containing the biodiesel is reweighed, and the density is determined by calculating the mass per unit volume of the sample.

$$\rho = \frac{W_t - W}{V} \quad (2)$$

$\rho$  : Density (gr/ml)

$W_t$  : Mass of Pycnometer with Sample

$W$  : Mass of Pycnometer

$V$  : Volume of Biodiesel

#### 2.2.3 Viscosity Analysis

Viscosity was measured using a Brookfield viscometer, which determines the viscosity of biodiesel through rotational methods. As the rotation speed increases, the viscosity decreases. To measure, the biodiesel sample is placed in a glass beaker, and the viscometer probe is immersed in the sample. Once the viscometer is activated, the viscosity value is displayed on the viscometer's screen.

### 3. Result and Discussion

#### 3.1 Transesterification Process

Sonication generates cavitation waves that facilitate the mixing and reaction between kapok oil and methanol to form methyl esters, as shown in Figure 1. The collapse of cavitation bubbles creates an environment with extremely high temperatures and pressures, producing rapid heating and cooling rates that result in high-energy chemistry, similar to photochemistry. This process introduces substantial energy in a brief period (within microseconds), creating hot spots with temperatures around 4726.8°C [26]. The continuous formation and collapse of cavitation waves significantly increase the microscale temperature [23]. This temperature increase accumulates, leading to a rise in the reaction temperature during sonication. As shown in Figure 2, the longer the transesterification process, the higher the reaction temperature. Elevated temperatures in the sonication transesterification process help reduce the viscosity of oils or fats, leading to faster reaction rates and shorter reaction times. However, if the temperature exceeds the alcohol's vapor point, the yield decreases because the alcohol evaporates before it can react with the vegetable oil to form biodiesel. This is supported by Cintas et al [27] who found that transesterification near the boiling point of alcohol dramatically reduces cavitation and reaction efficiency.

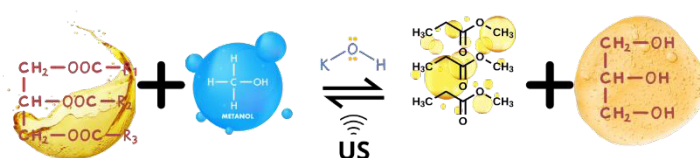
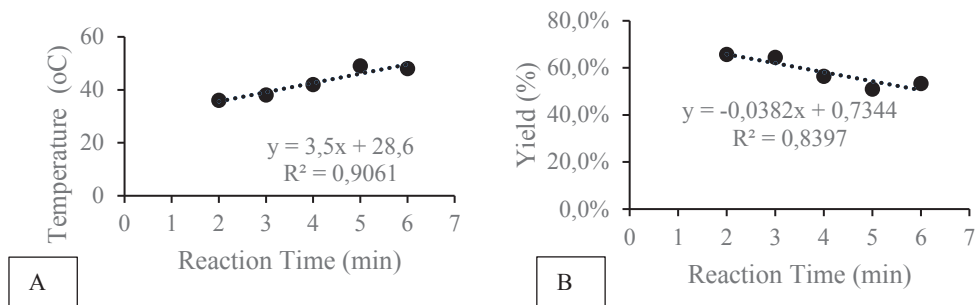


Fig 1. Transesterification scheme by sonication

#### 3.2 Yield

According to Figure 2, the duration of sonication has an adverse effect on biodiesel yield, with longer sonication times leading to lower yields. The highest yield, 65.7%, was achieved with 2 minutes of sonication, while the lowest yield, 51%, was observed at 5 minutes. Prolonged sonication can lead to increased ester hydrolysis, where the breakdown of esters into fatty acids is accelerated due to excessive exposure to ultrasonic waves. This phenomenon occurs because prolonged sonication can generate excessive heat and mechanical stress, promoting unwanted side reactions that degrade the biodiesel yield [16].

Conversely, insufficient sonication time may result in incomplete transesterification, where the conversion of triglycerides to biodiesel is not fully achieved due to inadequate mixing and reaction kinetics. This incomplete conversion can leave behind unreacted components, reducing overall yield and quality. Therefore, optimizing sonication duration is critical not only to maximize biodiesel yield but also to ensure efficient utilization of resources and minimize the formation of by-products like soap.



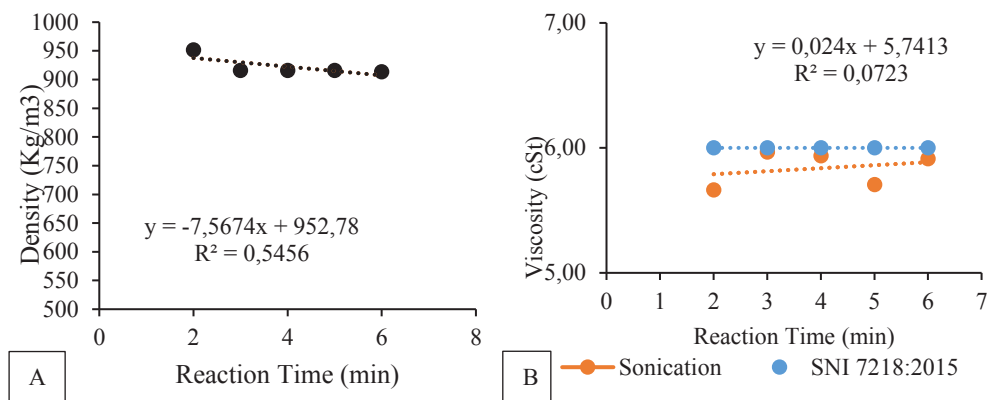
**Fig. 2.** (A) Impact of reaction duration on temperature elevation, (B) Influence of reaction duration on biodiesel yield

### 3.3 Density

As shown in Figure 3, the density of biodiesel is affected by the temperature during the transesterification process, with higher temperatures typically leading to lower density values. This relationship occurs because increased temperatures cause the volume of the reactants to expand while maintaining constant mass, thereby reducing density. As depicted in Figure 3, longer reaction times indirectly lead to higher process temperatures, which consequently decrease the density of biodiesel.

In relation to the Indonesian National Standard (SNI), biodiesel must meet specific density criteria to ensure its quality and compatibility with conventional diesel fuels. If the density values obtained from the study do not meet these standards, several methods can be explored to adjust them accordingly. For instance, optimizing the molar ratio of reactants, adjusting reaction temperatures, or selecting alternative catalysts can potentially affect biodiesel density. Furthermore, modifying feedstock compositions or refining purification processes could also impact density outcomes.

Regarding the impact of unsaturation levels in Fatty Acid Methyl Esters (FAMES) on density, previous studies by Pratas et al. [10] have demonstrated that higher unsaturation levels result in lower density values. This is due to the increased presence of double bonds in FAMES, which lead to looser molecular packing and reduced density.



**Fig. 3.** (A) Impact of reaction duration on biodiesel density, (B) Influence of reaction duration on biodiesel viscosity

### 3.4 Viscosity

The viscosity of biodiesel produced through sonication complies with SNI 7218:2015, not exceeding 6 cSt. Viscosity affects the amount of biodiesel injected during combustion; higher viscosity leads to greater volume variability or lower consistency in the fuel injected [28]. This low consistency can reduce combustion efficiency. According to Figure 3, the viscosity of the biodiesel tends to increase but remains within the SNI limit. In this study, viscosity was measured at 25°C, although SNI recommends measurement at 40°C. The temperature during viscosity measurement affects the value, with higher temperatures resulting in lower viscosity. Robertson et al [29] found that at 16.85°C, biodiesel from rose oil had a viscosity of 5 cSt, while at 6.85°C, the viscosity increased to 7.5 cSt

## 4. Conclusion

Biodiesel produced through sonication-assisted transesterification of kapok oil meets the viscosity requirements stipulated by the Indonesian national standard but falls short of meeting the density standard. The decline in yield observed with extended reaction times is likely attributable to increased ester hydrolysis, which is the reverse reaction of transesterification. This process leads to the breakdown of esters and the formation of extra fatty acids, which then undergo saponification to produce soap.

Further investigations into the sonication of kapok oil for transesterification should focus on optimizing key factors, including the ratio of oil to alcohol and the amount of catalyst used. This investigation aims to optimize the formulation and ensure that biodiesel meets the quality standards outlined in SNI. By studying these parameters, researchers can enhance the transesterification process, improve biodiesel yield, and align with regulatory requirements for biodiesel quality and performance. In making biodiesel can be studied along with studies on the transesterification method.

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