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Production of biodiesel from non-edible waste palm oil and *sterculia foetida* using microwave irradiation

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Abstract. The environmental damage stemming from traditional diesel begins during crude oil extraction and persists throughout its usage. The burning of fossil fuels has further deteriorate the environmental effect and added to global warming by emitting harmful substances. Moreover, the reduction of finite fossil fuel reserves due to widespread extraction has made the adoption of renewable resources essential. Given these considerations, biodiesel emerges as a highly promising alternative to conventional diesel due to its environmentally beneficial nature, renewable source, and economic feasibility. In this study, biodiesel was prepared by a microwave reactor in the presence of potassium methoxide using blended waste palm oil and *sterculia foetida*. The effects of raw materials characteristics on transesterification products were studied. The studied process parameters were methanol/oil ratio, microwave temperature, catalyst concentration, reaction time, and stirring speed. The optimal yield with 98.5% FAME content was obtained at a methanol/oil ratio of 60 vol. %, microwave temperature of 120 °C, catalyst concentration of 0.3 wt.%, and 3 min reaction time, and stirring speed of 500 rpm. The potassium methoxide was used to catalyse the transesterification process. The physicochemical properties and the fatty acid methyl ester composition were discussed thoroughly. The flash point of biodiesel, at 157.5°C, exceeds that of diesel fuel by more than two times. The cetane index is 59.5 which is higher than diesel (49.6). The biodiesel's fuel properties conformed to the requirements of both ASTM D6751 and EN 14214. High biodiesel conversion and low sulphur content show that waste palm oil and *sterculia foetida* are sustainable and economical feedstocks that produce clean fuel to aid the feasibility of the energy transition of the global energy sector. In addition, the selection of synthesis approaches can be further explored for potential catalysts to ensure eco-green biodiesel's sustainability with minimised.

Keywords: Biofuel, Energy, Hybrid feedstock, Sustainability, Waste management

1. Introduction

Biodiesel, a renewable and environmentally friendly alternative to conventional fossil fuels, has gained significant attention in recent years. As the global demand for cleaner energy sources continues to grow, there is an increasing focus on improving the properties and sustainability of biodiesel. One promising approach in this endeavour involves blending waste cooking oil (WCO) with non-edible oils, an innovative strategy that enhances biodiesel's properties and addresses sustainability concerns. WCO, a readily available by-product of culinary activities and food processing, has become a valuable feedstock for biodiesel production [1, 2]. Recycling WCO not only reduces waste but also offers economic and environmental advantages. However, WCO alone may not always meet the desired biodiesel specifications due to variations in quality and composition, more advancement is needed such as improving the process of conversion [3-6], the addition of other feedstocks [7, 8], alternative catalyst [9-11] and many more [12]. Non-edible oils derived from plants that are not part of the human food chain, such as *Jatropha curcas* [13], *Calophyllum inophyllum* [14], *Ceiba pentandra* [15], Nagkesar [16], *Cichorium intybus* [17], and others have emerged as complementary sources for biodiesel production. These non-edible oils often exhibit favourable properties for biodiesel, including high oil content, good cold properties, long oxidation stability, and low competition with food crops. *Sterculia foetida* can be a valuable feedstock due to its relatively high oil content and favourable fatty acid composition [18].

The synergy between WCO and non-edible oils offers a unique opportunity to produce biodiesel with improved properties [7]. By blending these feedstocks, it becomes possible to balance the shortcomings of WCO, such as higher levels of free fatty acids and lower cold flow characteristics, with the strengths of non-edible oils, including high energy content and good oxidative stability. This results in biodiesel with enhanced overall quality, meeting or exceeding industry standards and specifications. Besides, blending feedstocks can help reduce reliance on any single feedstock and enhance the stability of the biodiesel production process [19].

Besides the sustainability of feedstocks, specific processes and methods for biodiesel production may vary based on the technology and equipment available. Microwave irradiation-assisted transesterification is an innovative and efficient method for biodiesel production, offering several advantages over traditional transesterification processes [20]. In this method, the reaction mixture is exposed to microwave radiation, resulting in rapid heating, lower relative energy consumption, and reduced reaction time. The process also enables the production of biodiesel at milder conditions, which can be cost-effective and environmentally friendly. Microwave irradiation-assisted transesterification has been shown to increase biodiesel yield and reduce product degradation, making it a promising alternative to conventional transesterification methods. Microwaves are a form of electromagnetic radiation that generates heat by causing polar molecules (like those in the reaction mixture) to rotate and generate thermal energy [21]. Microwaves selectively and rapidly heat the reaction mixture, focusing energy directly on the reactants. This results in faster and more uniform heating than conventional heating methods, reducing reaction times, and energy consumption and minimising heat loss [22].

This study explores the promising prospects of blending WCO with *Sterculia foetida* for biodiesel production. We found that W70SF30 impact its performance and applicability. Additionally, by blending 70 vol. % of WCO with 30 vol.% of *Sterculia foetida*, we will assess the sustainability of this approach and the reliability of the biodiesel properties, emphasising the investigation of the properties of biodiesel and its potential to reduce waste and reliance on food-based feedstocks. Microwave irradiation transesterification of W70SF30 offers advantages such as shorter reaction times, reduced energy consumption, improved biodiesel yield, and enhanced reaction control. Besides, the high-quality biodiesel can be used as an eco-friendly and renewable fuel source for diesel engines, providing a cleaner alternative to traditional diesel.

2. Materials and methods

The following materials and methods was employed to produce a blend biodiesel from WCO with sterculia foetida at ratio of 7:3 using energy efficiency equipment (monowave reactor).

2.1. Materials
Crude *Sterculia foetida* crude oil was procured from Indonesia, whereas the waste cooking oil was collected from local food industry. The chemicals utilized in biodiesel production included CH_3OH (99.9% purity, ACS reagent grade), H_2SO_4 (>98.9% purity), H_3PO_4 (85% purity), Na_2SO_4(99% purity), $NaHCO_3$ (>99% purity), KOH pellets (99% purity), Carboxylic ester mixture (8-24), Nonadecanoic acid methyl ester, 19 (brand: Sigma-Aldrich, purity:>99.5%), 1,2,4-butanetriol, glyceryl monononadecanoate mono C19, glyceryl dinonadecanoate di C38, glyceryl trinonadecanoate tri C57, and Fluka® Analytical phenolphthalein solution (1% in ethanol).

2.2. Experiment set up
Waste cooking oil was initially purified using a filter to eliminate solid impurities and then subjected to heat treatment to eliminate any remaining water content. Subsequently, the purified waste cooking oil was blended with Sterculia foetida oil in a 7:3 volume ratio, resulting in the creation of the W70SF30 oil blend. Analysis revealed that the FFA of crude oil mixture exceeded the acceptable threshold for transesterification, measuring at 4.6% compared to the permissible limit of 2%. The oil mixture was then esterified with 50 (v/v)% of methanol and 1 (v/v)% of sulphur acid in a 500 mL double-jacketed reactor set at a temperature of 60 °C and stirring speed of 1500 rpm for 2 h. The properties of the waste cooking oil, *Sterculia foetida* oil, and W70SF30 blend are tabulated in table 1. FFA content of the esterified W70SF30 oil is reduced to less than 2%, indicating that the oil is ready for further process. The monowave reactor with 24 automatic samplers was selected for its suitability in small-scale microwave synthesis, making it an ideal choice for preliminary studies. This equipment is specifically designed for low volume microwave synthesis applications in R&D laboratories, offering features like unattended sequential processing of 24 experiments and precise temperature control up to 300°C.

Table 1. Properties of waste cooking oil, *Sterculia foetida* , and W70SF30.

Properties	Unit	Crude oils		Crude oil mixture
		WC	SF	W70SF30
Kinematic viscidity at 40°C	mm^2/s	49.05	38.64	47.60
Dynamic Viscidity at 40°C	mPas	44.27	34.99	43.03
Denseness at 40°C	kg/m^3	902.70	905.70	904.00
Denseness at 15°C	kg/m^3	901.00	904.00	902.30
Acid Value	mgKOH/g	2.19	26.30	9.20
Heating capacity	MJ/kg	38.59	38.30	37.52
Oxidation Value	hr	4.61	3.07	3.28

2.3. Microwave irradiation-assisted alkaline-catalysed transesterification
AP Monowave reactor with 24 samplers was utilized for the conversion process. This equipment is integrated with thermal sensors to accurately control the temperature, pressure value indicator to observe reactions, and an integrated magnetic stirrer for optimal mixing. Throughout the experiments, the temperature and pressure profiles were automatically recorded by the reactor. The heating process in the microwave reactor involves three stages: heating the vessel to the desired temperature, maintaining a constant temperature for a specific duration, and then cooling the vessel to stop the reaction. Adjusting the power voltage by refering to the chosen temperature every experiment was crucial to prevent any exothermic reactions during the transesterification process.

2.4. Measurement of physicochemical properties
W70SF30 biodiesels' properties were compared with those of diesel fuel and Jatropha-ceiba biodiesel. From the findings, W70SF30 biodiesels exhibited similar properties to Jatropha-ceiba biodiesel and

diesel, with some variations when compared. The W70SF30's physicochemical properties produced from microwave-assisted transesterification were analysed according to the standard shown in table 2. These properties included kinematic viscosity at 40 °C, density at 15 °C, acid value, calorific value, oxidation value, flash point, copper strip corrosion, Conradson carbon residue, cetane index, cold flow properties, sulphur content, water content, methanol content, FAME content, glycerides (single, double and triple bond), free glycerol, and total glycerol.

3. Results and discussion

3.1. Biodiesel yield of W70SF30 from the high-performance microwave reactor

The microwave reactor offers efficient in-core heating directly to the glass vial, ensuring energy-efficient processing. In figure 1(left), the glass vial is partially filled with esterified W70SF30 oil in the bottom layer and a KOH catalyst dissolved in methanol in the top layer. A magnetic stirring bar is gently placed inside the glass vial to guarantee thorough mixing of the reaction mixture during the transesterification process in the microwave reactor. The KOH catalyst-methanol mixture is crucial in expediting the transesterification reaction, ensuring the experiments remain unbiased as all processes are completed within a few minutes. Figure 1(right) illustrates the production of W70SF30 methyl ester through the microwave irradiation-assisted alkaline-catalysed transesterification process. The transparent glass vial clearly shows the completion of the transesterification reaction, with W70SF30 methyl ester formed in the upper layer, while the lower layer contains methanol, glycerol, KOH catalyst, and other impurities. As anticipated, the transesterification reaction is finalised in less than 5 minutes with the microwave reactor's assistance, in contrast to the conventional transesterification method, which typically requires over 60 minutes to reach completion. Additionally, the microwave reactor's parameters (infrared temperature, fibre optic temperature, operating pressure, and power) can be accurately measured and monitored throughout the experiment, ensuring precise control of the parameters. The best yield conversion of W70SF30 of 96.5 wt. % by controlling the factor likes CH_3OH/Triglycerides ratio, microwave temperature, accelerator concentration, conversion time, and mixer speed at 60 vol. %, 120 °C, 0.3 wt.%, 3 min. and 500 rpm, respectively.

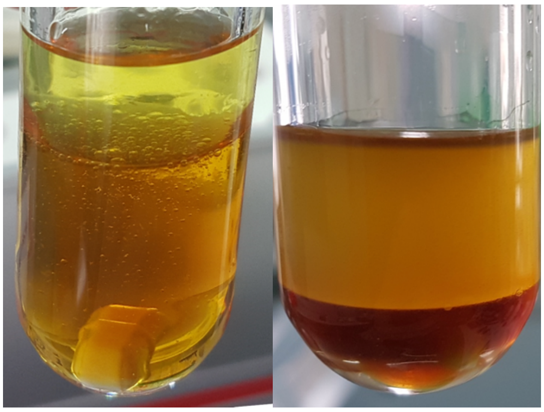

Figure 1. (Left) Esterified W70SF30 oil before inserting into the microwave reactor (Right) W70SF30 methyl ester after the glass vial is removed from the microwave reactor.

3.2. Physicochemical properties of the W70SF30 biodiesel

W70SF30 methyl ester's properties was evaluated and compared with other fuels, as shown in table 1. W70SF30 methyl esters produced from the microwave irradiation-assisted transesterification methods fulfil the fuel specifications given in the ASTM D6751 and EN 14214 standards. The optimized W70SF30 methyl ester exhibits lower kinematic viscosity at 40°C (4.54 mm²/s) and density at 15°C

(876.1 kg/m³), indicating favorable lubrication characteristics. Additionally, the acid value of the optimized methyl ester is 0.3 mg KOH/g, below the permissible limit of 0.5 mg KOH/g set by ASTM D6751 and EN 14214 standards. The calorific value of the methyl ester is 40.51 MJ/kg, slightly lower than diesel (45.36 MJ/kg), suggesting comparable energy content. However, the oxidation stability at 110°C is 5.3 h, meeting ASTM D6751 but falling short of EN 14214's requirement of 6 h. Oxidation stability holds significant importance as it serves as a key indicator of fuel deterioration, which has a pivotal impact on fuel storage, handling, and transportation. Undesirable fuel oxidation results in adverse effects on the physical and chemical properties of the fuel, leading to an increase in acid value, which, in turn, can cause corrosion of engine components. Additionally, it leads to an elevation in kinematic viscosity, potentially causing fuel injector clogging. The flashpoint of the W70SF30 methyl ester is 157.5 °C, which is significantly higher than that for diesel (75.5 °C) but lower than biodiesel Jatropha50Ceiba50 (196 °C). The flash point of the W70SF30 meets the criteria set by both ASTM D6751 and EN 14214 standards. These standards stipulate that the fuel's flash point should fall within the 100–170 °C range and exceed 101 °C, respectively. The elevated flash point of the optimized W70SF30 methyl ester serves to mitigate the risk of fire hazards when the fuel comes into contact with potential ignition sources, such as flames or sparks. The cold flow properties are also for the W70SF30 methyl ester where the PP, CP, and CFPP are 3, 3.2, and 3 °C, respectively, indicating that the methyl ester is suitable for use in cold-climate countries. The copper strip corrosion test indicates that the W70SF30 biodiesel is less corrosive as the test value is 1a. The cetane index of W70SF30 biodiesel (59.5) is slightly higher than diesel (49.6) but lower than Jatropha50Ceiba50 biodiesel (58.0). The FAME content and linolenic methyl ester content of the W70SF30 biodiesel 98.50 wt.% and 0.51 wt.%. The FAME content is above the minimum requirement of 90 wt.% and the linolenic methyl ester content of the W70SF30 biodiesel which is significantly lower than the value stipulated in the EN 14103:2011 standard (15 wt.%). The methanol content (0.02 wt.%) of the W70SF30 biodiesel shows that the purification process was done perfectly and the stability of the biodiesel favours the forward transesterification process. The total monoglyceride content (0.35 wt.%), total diglyceride content (0.04 wt.%), and total triglyceride content (0.12 wt.%) of the W70SF30 biodiesel fulfils the requirements of the EN 14214 standard. In addition, the total glycerol (0.12 wt.%) and free glycerol (0.02 wt.%) fulfils the specifications since the values are less than 0.25 wt.% and 0.02 wt.%, respectively. The notably low levels of monoglycerides, diglycerides, and triglycerides point to the successful conversion of most glycerides into methyl ester. Most glycerol has been eliminated from the methyl ester during the subsequent separation and purification processes. A comparison of the physicochemical properties of W70SF30 biodiesel, Jatropha50Ceiba50 biodiesel, and diesel reveals that W70SF30 biodiesel complies with the specified standards outlined in ASTM D6751 and EN 14214. This underscores W70SF30's potential as a biodiesel with a substantial impact on the sustainability, cost-effectiveness, and overall characteristics of the biodiesel it yields. Moreover, it's well-suited for use in both tropical and cold climates.

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Table 2. The properties of diesel, W70SF30 biodiesel and Jatropha50Ceiba50 biodiesel.

Properties	Unit	Biodiesel			Diesel	Biodiesel W70SF30	Biodiesel Jatropha50Ceiba50 [23]
		ASTM D6751	Test method	EN14214			
Kinematic viscosity at 40°C	mm^2/s	1.9 to 6.0	D 445	3.5 to 5.0	2.96	4.5400	3.95
Density at 15°C	kg/m^3	880	D 127	860 to 900	846.1	876.1	831.20
Acid value	mgKOH/g	0.5max	D 664	0.5max	0.017	0.30	0.25
Calorific value	MJ/kg	-	D 240	-	45.36	40.51	40.92
Oxidation value at 110°C	hr	3 min	EN 14112	6 min	15.2	5.3	10.01
Flash point	°C	100 to 170	D 93	101 min	75.5	157.5	196.00
Pour point	°C	-15 to 16	D 2500	-	3.0	3	0.50
Cloud point	°C	-3 to 12	D 2500	-	2.0	3.2	0.50
Cold filter plugging point	°C	19 max	D 6371	0 max	0	3	-2.00
Copper strip corrosion	-	3 max	D 130	-	1a	1a	1a
Cetane index	-	47 min	D 613	51 min	49.6	59.5	58.00
Sulphur content (S 500 grade)	ppm	500 max	D 874	10 max	449.7	-	-
Sulphur ash content	wt.%	0.02	D 874	0.02	449.7	0.004	-
Conradson carbon residue	wt.%	-	-	-	0.187	0.03	-
Water content	vol.%	0.05 max	ASTM D6304	500 ppm	-	0.035	-
FAME content	wt.%	-	-	90 min	-	98.50	99.10
Linolenic-AME content	wt.%	-	-	1-15	-	0.51	-
Methanol	wt.%	0.2	EN 14110	0.2	-	0.02	-
Single-glycerides	wt.%	-	-	0.8	-	0.35	-
Double-glycerides	wt.%	-	-	0.2	-	0.04	-
Triple-glycerides	wt.%	-	-	0.2	-	0.12	-
Free glycerol	wt.%	0.02 max	D 6584	0.02	-	0.02	-
Total glycerol	wt.%	0.24 max	D 6584	0.25	-	0.12	-
Carbon content	wt.%	77	-	-	88.5	79	-
Hydrogen content	wt.%	12	-	-	13.5	13	-
Oxygen content	wt.%	11	-	-	0	12.5	-

4. Conclusions

The research produces biodiesel from a blend of waste cooking oil and *Sterculia foetida* oil at a volume ratio of 7:3 (W70SF30) using microwave irradiation. Initially, the W70SF30 mixture had an acid value of 9.20 mg KOH/g and a kinematic viscosity at 40 °C of 47.60 mm^2/s. Through acid-catalyzed esterification followed by microwave irradiation-assisted alkaline-catalyzed transesterification, these values were reduced to 0.30 mg KOH/g and 4.54 mm^2/s, respectively. Optimal conditions for biodiesel production were determined, including a methanol/oil molar ratio of 60 vol.%, microwave temperature of 120 °C, KOH catalyst concentration of 0.3 wt.%, stirring speed of 500 rpm, and a reaction time of 3 minutes. Under these conditions, a biodiesel conversion yield of 96.5% with a FAME content of 98.50 wt% was achieved. The physicochemical properties of W70SF30 biodiesel met the fuel specifications outlined in both ASTM D6751 and EN 14214 standards. Microwave irradiation played a crucial role in accelerating the alkaline-catalyzed transesterification process, significantly reducing the total reaction time to just 5 minutes (including a 2-minute preheat to reach 120°C and a 3-minute reaction time). This approach resulted in biodiesel with competitive advantages in terms of time and energy efficiency. Determining the optimal blend ratio and processing conditions for the amalgamation of waste cooking oil (WCO) with *Sterculia foetida* is essential for enhancing the efficiency and viability of biodiesel production. This approach can yield biodiesel with enhanced characteristics while minimizing its environmental impact, thus contributing to a more sustainable and streamlined biodiesel industry. It's crucial to underscore the importance of sustainability and the adoption of responsible harvesting practices to mitigate the environmental impact of utilizing *Sterculia foetida* as a biodiesel feedstock. As the global quest for sustainable alternatives to traditional fossil fuels gains momentum, blending waste cooking oil with non-edible oils emerges as a compelling solution to enhance biodiesel properties and reduce its environmental footprint. Besides, using waste cooking oil and *Sterculia foetida*, similar method can be applied to other biomass feedstocks such as *Jatropha curcas*, *ceiba pentandra*, rubber seed oil, *Millettia pinnata* and other. This research contributes meaningfully to advancing a more efficient, eco-friendly, and economically viable biodiesel production process, which can be pivotal in the global transition toward cleaner and more sustainable energy sources.

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