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# Optimization of biodiesel production by microwave irradiation-assisted transesterification for waste cooking oil-*Calophyllum inophyllum* oil via response surface methodology

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ABSTRACT

In this study, microwave irradiation-assisted alkaline-catalysed transesterification was used to produce W70CI30 biodiesel from a mixture of waste cooking oil and *Calophyllum inophyllum* oil. The methanol/oil ratio, catalyst concentration, stirring speed, and reaction time were optimized using response surface methodology based on the Box-Behnken experimental design in order to maximize the biodiesel yield. The quadratic response surface regression model was used to predict the biodiesel yield. It is found that the optimum methanol/oil ratio, catalyst concentration, stirring speed, and reaction time are 59.60 (v/v)%, 0.774 (w/w)%, 600 rpm, and 7.15 min, respectively, and the predicted biodiesel yield is 97.40%. Experiments were conducted using the optimum process parameters and the average biodiesel yield is 97.65%, which is in excellent agreement with the predicted value. The physicochemical properties of the W70CI30 biodiesel produced using the optimum process parameters were measured and it is found that the biodiesel has significantly higher oxidation stability (18.03 h) compared with the waste cooking oil biodiesel (4.61 h). In addition, the physicochemical properties and cold flow properties of the biodiesel fulfil the fuel specifications stipulated in the ASTM D6751 and EN 14214 standards. It can be concluded that microwave irradiation-assisted transesterification is effective to boost the biodiesel yield and produce biodiesel of superior quality. In addition, this method significantly reduces the reaction time of the transesterification process to 9.15 min and the process is energy-efficient. It is believed that the findings of this study will be beneficial for microwave irradiation-assisted biodiesel synthesis on the industrial scale.

## 1. Introduction

It is known that global energy demands are mainly fulfilled by fossil fuels. However, global energy demands seem to increase at a slower pace in recent years due to unprecedented efficiencies created by novel renewable energy technologies as well as the enforcement of stringent energy policies and environmental legislations. There is a dramatic shift in the energy pattern where the demand for energy harvesting from fossil fuels has declined since year 2014. The current state of global energy is called 'The Grand Transition' [1]. In this state, there is a strong demand for renewable energy due to the emergence of new technologies, greater environmental challenges, and swiftness in economics and geopolitical power [2]. It is well-known that the burning of fossil fuels such as coal and oil leads to environmental problems and decarbonization of energy systems (increasing the utilization of low-carbon energy sources such as renewable energy) to address environmental issues such as climate change is one of the toughest challenges that require full commitment from all relevant parties [3,4]. The carbon intensity contributed by the transportation sector in particular, plays a crucial role on environmental health [5–7].

Hence, efforts are being made to diversify the fuels used by vehicles and it is believed that this approach is one of the efficient solutions to address environmental problems resulting from fossil fuel combustion [8,9]. Biodiesels play an important role to fulfil the demand for alternative fuels, which will help reduce carbon emissions [10,11] due to the fact that biodiesels are biodegradable and environmentally friendly

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compared with fossil fuels. First-generation biodiesels are produced from edible vegetable oils such as sunflower, soybean, and palm oils. However, using edible vegetable oils for biodiesel production is not a feasible solution in the long term because this approach leads to global food insecurity and fluctuations in food prices [12–14]. For this reason, second-generation biodiesels come into play and these fuels are produced from non-edible plant-based oils such as *Calophyllum inophyllum* [15,16], *Ceiba pentandra* [17], *Schleichera triguga* [18], and *Pongamia pinnata* [19] oils. In addition, second-generation biodiesels are proven to have high biodiesel yields, good oxidation stabilities, and favourable cold flow properties. Furthermore, second-generation biodiesels fulfil the fuel specifications given in the ASTM D6751 and EN 14214 standards, rendering these fuels promising as diesel substitutes. However, the main disadvantage of second-generation biodiesels is their high acidity since these fuels are produced from non-edible feedstocks with high free fatty acid (FFA) content.

While improving the physicochemical properties of biodiesels is one of the key areas in biodiesel research, the cost of biodiesel production remains the primary interest in the biodiesel industry in order to achieve energy and environmental sustainability [12,20–22]. In this regard, waste cooking oils are among the low-cost feedstocks that can be easily collected from restaurants in the food and beverage industry. It is a great idea to recycle waste cooking oils because these oils can be used as a continuous supply in order to create useful products (biodiesels), which will help to fulfil the ever-increasing global energy demands. Converting waste cooking oils into biodiesels is a practical approach since the oils are recycled rather than being disposed into the landfill. In addition, this approach will help minimize environmental pollution caused by illegal handling of waste cooking oils. However, it is particularly challenging to convert waste cooking oils into biodiesels with high biodiesel yields, good oxidation stabilities, favourable cold flow properties, as well as low FFA content, trace elements, and acid values [23,24]. In order to address this issue, waste cooking oil can be blended with non-edible plant-based oil in order to improve the physicochemical properties of the biodiesel. In this study, waste cooking oil is blended with non-edible *Calophyllum inophyllum* oil in order to improve the physicochemical properties of the biodiesel which will fulfil the fuel specifications given in the ASTM D6751 and EN 14214 standards [25,26].

Besides the prices of the raw materials, the technology used for biodiesel production is equally important in order produce biodiesel with competitive prices as those for diesel [27,28]. Biodiesels produced from conventional alkaline-catalysed transesterification requires long reaction times (typically more than 60 min) due to the heat transfer from the heating surface to the oil by conduction, convention, and radiation [29]. The mode of heat transfer between the surface and interior of the material is thermal conduction. The chemical reaction is dependent on the heat transfer efficiency, which is why conventional heating results in long reaction time in order to achieve a high conversion of crude oil into biodiesel. Therefore, it is crucial to use the appropriate technology for biodiesel production. In this regard, microwave irradiation-assisted alkaline-catalysed transesterification process helps improve the heat transfer efficiency, which results in shorter chemical reactions [30–32]. Microwave irradiation creates a magnetic field in the oil which forces the original random thermal motion of the reactants to follow the orientation of the electric field in order to generate heat. The heat transfer efficiency is dependent on the dielectric properties of the material used. The heating characteristics of a material subject to microwave irradiation are dependent on the ability of a specific substance to convert the electromagnetic energy into heat. Therefore, synthesis of organic materials such as oil with high ionic liquids such as potassium hydroxide (KOH)-methanol reagent mixture results in strong polarity, which will increase the efficiency of microwave heating. In addition, microwave irradiation-assisted alkaline-catalysed transesterification is a feasible biodiesel production method because the results are repeatable and reproducible at extreme temperatures and pressures.

Naor et al. [33] adopted microwave irradiation-assisted transesterification to produce biodiesel from *Nannochloropsis* microalgae oil in 2 min. Strontium oxide/silicon dioxide ($SrO/SiO_2$) nanopowders were used in the biodiesel synthesis. El Sherbiny et al. [34] produced *Jatropha* biodiesel by using both conventional and microwave irradiation-assisted transesterification methods. The results showed that the reaction rate was significantly reduced from 150 min (conventional transesterification) to 2 min (microwave irradiation-assisted transesterification). Xiang et al. [35] produced biodiesel from waste cooking oil using microwave irradiation-assisted transesterification with a fixed reaction time of 6 min. The optimum process parameters used in their study were (1) methanol/oil molar ratio: 9.67:1, (2) modified coal fly ash: 3.99%, and (3) reaction temperature: 66.2 °C. The corresponding biodiesel yield was 94.5%. Hong et al. [36] produced biodiesel from waste cooking oil using microwave irradiation-assisted transesterification and the fatty acid methyl ester (FAME) content was 96.8 (w/w)%. The following process parameters were used in their study: (1) reaction time: 6 min, (2) methanol/oil molar ratio: 8:1, (3) microwave power: 500 W, and (4) KOH catalyst concentration: 1 (w/w)%. In general, the results of these studies indicate that microwave irradiation-assisted transesterification is one of the promising methods for biodiesel production. Microwave irradiation provides unique thermal effects that are beneficial to chemical synthesis. Microwave irradiation boosts biodiesel yields, reduces the time of chemical reaction, improves the separation process, and reduces the net energy to produce biodiesel.

Response surface methodology (RSM) is an effective statistical tool used to examine the effects of various independent variables on the dependent variable. This tool greatly facilitates researchers in determining the optimum parameters by reducing the large number of experiments [37,38] that supposedly need to be performed with conventional experimental methods. Thus, RSM helps boost productivity and minimizes the time, material, and cost consumption required for optimization [39,40].

Due to the advantages of microwave irradiation-assisted transesterification, this method is adopted in this study to produce biodiesel from waste cooking oil blended with *Calophyllum inophyllum* oil. RSM based on the Box-Behnken experimental design is used to optimize the methanol/oil ratio, KOH catalyst concentration, stirring speed, and reaction time in order to maximize the biodiesel yield. RSM is also used to examine the interaction effects of the aforementioned process parameters on the biodiesel yield. The procedure used for biodiesel production using microwave irradiation-assisted transesterification and conventional transesterification is described in detail in this paper. The procedure used to determine the FAME content, linolenic methyl ester content, as well as glyceride and glycerol composition is also described in detail. The physicochemical properties of the biodiesel are measured and the effect of blending waste cooking oil with *Calophyllum inophyllum* oil on the physicochemical properties of the biodiesel is discussed. The results obtained from microwave irradiation-assisted transesterification and conventional transesterification are compared and discussed. The novelty of this work lies in the optimization of the process parameters for microwave irradiation-assisted alkaline-catalysed transesterification using RSM in order to maximize the yield of biodiesel produced from waste cooking oil blended with *Calophyllum inophyllum* oil. The use of a microwave reactor significantly reduces the reaction time and produces biodiesel of superior quality, as shown in the results of this study.

## 2. Material and methods

### 2.1. Materials

Crude *Calophyllum inophyllum* crude oil was purchased from Kebumen, Central Java, Indonesia, whereas the waste cooking oil was collected from various restaurants in the food and beverage industry. The following chemicals were used for biodiesel production: (1)