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Trenggalek Durian Peel Ash A K_2o Catalyst in Ultrasonic Biodiesel Production

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ABSTRACT

Global population growth has driven increased demand for energy, particularly fossil fuels. A sustainable alternative to address this issue is the production of biodiesel from renewable sources, such as waste cooking oil. This study utilized a heterogeneous potassium oxide (K_2O) catalyst derived from local Trenggalek durian peel ash to enhance the efficiency of the transesterification reaction. The catalyst was synthesized via calcination at 400°C, 500°C, and 600°C for 5 hours. The transesterification reaction was carried out with a methanol-to-oil molar ratio of 12:1 and catalyst concentrations of 1%, 3%, and 5% (w/w) at 65°C. The optimum condition was achieved using durian peel ash catalyst calcined at 600°C at a concentration of 3% (w/w), yielding a biodiesel conversion of 96.24% via ultrasonic-assisted transesterification. The physicochemical characterization of the produced biodiesel showed results that met standard specifications, including analyses of yield, density, viscosity, refractive index, acid value, saponification value, and iodine value. Furthermore, FT-IR and GC-MS analyses were performed to confirm the functional groups and components of the biodiesel product, in accordance with the expected specifications.

Keywords: Biomass waste, heterogeneous catalyst, renewable energy, transesterification, waste cooking oil.

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INTRODUCTION

Indonesia's rapid population growth has driven increased energy demand, particularly for diesel. This situation calls for energy diversification efforts to achieve national energy independence and security through the development of alternative fuels that are renewable, environmentally friendly, and economical (Fisika, P., F., U., & A., 2023; Suryatini & Milati, 2023). A promising approach is the use of biodiesel, an alternative fuel synthesized by transesterifying triglycerides with short-chain alcohols in the presence of a catalyst. The Indonesian government continues to increase its mandatory biodiesel program from a 15% blend (B15) in 2015 to 35% (B35) since 2023 as a strategic step to reduce diesel imports and greenhouse gas emissions (Kholishah, Mulyaningsih, & Firyanto, 2021).

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However, dependence on vegetable oils such as palm oil (CPO) as the main raw material for biodiesel raises new issues related to land conflicts, environmental degradation, and food security (Suryatini & Milati, 2023). Therefore, alternative raw materials that do not compete with food sources and are abundantly available are needed. Waste cooking oil is an ideal choice because it is a cheap, readily available household waste product with great potential as a biodiesel feedstock. In addition, waste cooking oil that is disposed of carelessly can pollute the soil and waterways, and is carcinogenic due to the formation of free radicals during the repeated heating process (Alamsyah, Kalla, & La Ifa, 2017). According to the International Council on Clean Transportation (ICCT), the potential for waste cooking oil in Indonesia reaches 715 kilotons per year, which theoretically can be converted into approximately 651 kilotons of biodiesel (Kristiana, Baldino, & Searle, 2022).

The conversion process of waste oil into biodiesel is carried out through transesterification, the efficiency of which can be improved with the aid of ultrasonic waves. The cavitation effect of ultrasonic waves produces microbubbles with high local pressure and temperature, thereby accelerating the homogenization of reactants and increasing the rate of the reaction (Putri et al., 2014; Santoso, Fantusi, Marfu'ah, & Sumari, 2021). In this process, the catalyst serves a crucial function by lowering the activation energy required for the reaction to proceed. (Wahyuni, Kadarwati, Wahyuni, & Kadarwati, 2011). Catalysts can be categorized by phase of operation into homogeneous and heterogeneous types.

Homogeneous catalysts have disadvantages such as being difficult to separate from the product and being corrosive, while heterogeneous catalysts are superior because they are easy to separate, non-corrosive, environmentally friendly, and can be reused (Ikhsan & Nizar, 2020).

One type of heterogeneous catalyst that is widely used is potassium oxide (K_2O)-based, because it has strong base sites and high catalytic activity (Sinaga, Pandia, Tarigan, & Tiffani, 2018). K_2O can be obtained from biomass waste, including fruit peels. Various studies have reported the utilization of fruit peel ash, such as avocado (Afandi, Riani, Syamsuddin, & Zuhra, 2021), banana (Basumatary, Basumatary, Das, Nath, & Kalita, 2021) , and cocoa (Sinaga et al., 2018) as a heterogeneous base catalyst in transesterification reactions. Durian peel is a type of biomass waste with high K_2O content and abundant availability, accounting for around 60% of the total fruit mass (Tiaradewi, 2016). Trenggalek, as a national producer of high-quality durians and home to the largest durian forest area in Southeast Asia, generates a high volume of durian peel waste, making it a potential raw material for catalysts (Nurfitri & Yahya, 2023).

Previous research by Tiaradewi (2016) showed that durian peel ash can be used as a catalyst in methanolysis reactions to produce methyl ester (Tiaradewi, 2016). Fitriana (2020). It was also reported that durian peel ash calcined at 600°C for 8 hours effectively catalyzed the transesterification of rubber seed oil, achieving optimum performance at 65°C, a reaction time of 1 hour, and a catalyst loading of 10% (w/w) (Fitriani et al., 2020). Sitepu et al. (2024) reported comparable findings, indicating that durian peel ash calcined at 600°C exhibits effective catalytic activity in the transesterification of palm oil under ambient conditions (Eko. K. Sitepu,* Reni P. A. Sinaga, Bryan E. N. Sitepu, Aman Santoso, Bambang Susilo & Sabarmin Perangin-angin, 2024)

Most previous studies used calcination temperatures of 600–1000°C. Therefore, it is important to study the catalytic activity of durian peel ash at calcination temperatures below 600°C to improve energy efficiency and broaden our understanding of the relationship between calcination temperature and catalytic activity. Based on this background, the present study focuses on optimizing biodiesel production from waste cooking oil using a K_2O-based heterogeneous catalyst

derived from Trenggalek durian peel ash under ultrasonic-assisted transesterification conditions. Optimization was carried out to determine the best reaction conditions that produce the highest biodiesel yield. Furthermore, the physical and chemical characteristics of the biodiesel obtained were analyzed, including testing of physical and chemical properties in accordance with SNI 7182:2015 to ensure compliance with national biodiesel quality criteria. Additionally, this study also evaluated *the reusability* of the K_2O durian peel ash catalyst under optimal conditions to assess its stability and catalytic efficiency in repeated use.

METHOD

Synthesis and Characterization of K_2O Catalyst in Trenggalek Durian Peel Ash

The durian peel samples were first washed, cut into smaller pieces, and then dried in an oven at 200°C for 2 hours. Following the drying process, the samples were finely milled using a household grinder and subsequently calcined in a furnace for 5 hours at 400°C, 500°C, and 600°C. This process produced a heterogeneous basic catalyst in the form of durian peel ash.

Subsequently, the catalyst was cooled in a desiccator for 24 hours to prevent direct exposure to air before being stored in a sealed container. The catalyst derived from calcined Trenggalek durian peel ash was subsequently characterized through X-ray fluorescence (XRF) analysis.

Waste Cooking Oil Pretreatment

The pretreatment of waste cooking oil involved a refining process utilizing natural zeolite at 30% of the total oil volume. The mixture was subjected to heating and stirring for 1 hour, followed by centrifugation to remove the zeolite and subsequent reheating at 100°C (Santoso, Sumari, Asrori, & Wele, 2024).

Subsequently, an esterification process was performed using a reflux system to lower the acid value. In this step, 100 grams of refined oil were heated to 65°C, after which methanol was added at a 5:1 molar ratio (methanol to oil) along with concentrated sulfuric acid (H_2SO_4) serving as a catalyst at 1% of the oil mass. The reaction was conducted at 65°C for 3 hours under continuous stirring.

The esterified mixture was transferred to a separating funnel and left to settle until two distinct layers were observed. The lower phase, consisting of methyl esters, was separated, washed several times with warm distilled water (50–60°C), and then heated to 100°C to remove residual moisture. The acid value of the purified ester was then determined to confirm that the FFA content was below 2% prior to transesterification.

Biodiesel Production

A total of 25 grams of esterified oil was introduced into a 100 mL Erlenmeyer flask, followed by the addition of methanol at a molar ratio of 12:1 (methanol to oil) and a heterogeneous K_2O catalyst derived from durian peel ash calcined at 400°C, 500°C, and 600°C with catalyst loadings of 1%, 3%, and 5% (w/w), respectively. The mixture was sealed with aluminum foil and subjected to transesterification using an ultrasonic cleaning bath at a frequency of 37 kHz and a temperature of 65°C for 1 hour. After the reaction, the mixture was centrifuged to separate the solid catalyst, and the supernatant was transferred into a separating funnel to facilitate the formation of two distinct layers. The upper biodiesel layer was then collected and washed repeatedly with warm distilled water (50–60°C), and subsequently heated at 100°C in the presence of anhydrous $MgSO_4$ to remove residual moisture.

Physicochemical Properties of Biodiesel

The characterization of methyl ester was carried out through compositional analysis of biodiesel using FT-IR and GC-MS, followed by physicochemical evaluations including density, viscosity, refractive index, acid value, saponification value, and iodine value.

Reusability of Trenggalek Durian Peel Ash Catalyst

The catalyst's reusability was examined to determine its stability and catalytic efficiency across multiple reaction cycles. After each transesterification, the catalyst was collected and sequentially cleaned with *n*-hexane and acetone under vacuum filtration using a Büchner apparatus to remove residual organic impurities. The cleaned catalyst was then dried in an oven at 100°C for 4 hours and subsequently reused under the previously determined optimal transesterification conditions for biodiesel production.

RESULTS AND DISCUSSION

Characterization of the Catalyst

The durian peel ash–derived heterogeneous catalyst was analyzed by X-ray fluorescence (XRF) to identify its elemental composition and associated metal oxide composition. This analysis confirmed the suitability of the raw material and the presence of key active components essential for catalytic activity. The XRF results, as presented in Table 1, revealed that the Trenggalek durian peel ash contained several metal oxides, with potassium (K) as the predominant element and potassium oxide (K_2O) as the major oxide compound.

This finding is consistent with previous studies reporting that durian peel ash is naturally rich in potassium [27]. During calcination, potassium is oxidized to form basic, active K_2O sites that play an essential role in enhancing transesterification.

Table 1. Oxide compound composition of the Trenggalek durian peel ash catalyst based on XRF analysis

Calcination Temperature	K_2O (%)	P_2O_5 (%)	SO_3 (%)	Dll (%)
400°C	87.90	5.34	4.52	2.24
500°C	88.00	4.95	4.87	2.19
600°C	88.06	5.14	5.09	1.71

Based on XRF results (Table 1), the lowest K_2O content was observed at 400°C and increased progressively with increasing calcination temperature up to 600°C. This improvement is associated with the thermal decomposition of organic components, which reduces the overall organic mass while enriching the inorganic K_2O fraction. Consequently, higher calcination temperatures result in a greater concentration of active K_2O species, thereby improving the catalytic potential of the material for biodiesel transesterification.

Effect of Calcination Temperature of Trenggalek Durian Peel Ash on Biodiesel Production

The effect of calcination temperature on the catalytic activity was investigated to identify the optimal conditions for biodiesel

production. Figure 1 shows that an increase in the calcination temperature of Trenggalek durian peel ash had a significant effect on biodiesel yield.

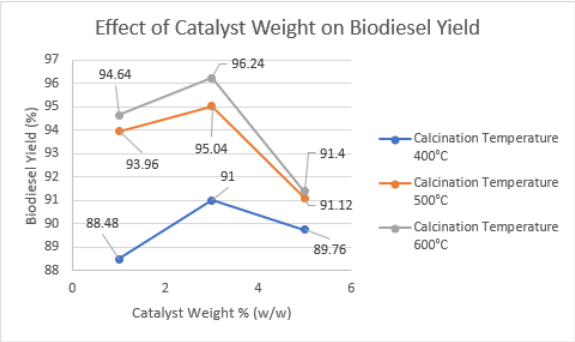

Figure 1. Influence of calcination temperature on biodiesel yield and production efficiency

At 600°C, the catalyst exhibited the highest activity, yielding 96.64% biodiesel. This enhancement is attributed to thermal decomposition, which promotes the formation of metal oxides at the catalyst's active sites, thereby improving biodiesel production efficiency. The generation of K_2O at elevated calcination temperatures significantly enhances the catalyst's basicity, directly accelerating the transesterification process. The strong basic sites provided by K_2O facilitate methanol deprotonation to form methoxide ions, which serve as reactive species that convert triglycerides into methyl esters. An increase in K_2O concentration is directly proportional to catalytic efficiency and biodiesel yield (Suhartini, Nurika, Paul, & Melville, 2021).

Meanwhile, the results at 400°C and 500°C were 95.04% and 91%, respectively. At lower temperatures, the ash still contained a lot of organic material, and potassium elements or compounds had not been completely decomposed or converted into K_2O (Sarkar et al., 2023).

Effect of Catalyst Loading on Biodiesel Production Yield

Figure 2 shows that using Trenggalek durian peel ash catalyst at 1% and 3% increases biodiesel yield, whereas 5% results in a decrease in yield. This is due to the adsorption of synthesis products by the catalyst pores as a result of the addition of excess solid catalyst (Oladipo, Ojumu, Latinwo, & Betiku, 2020).

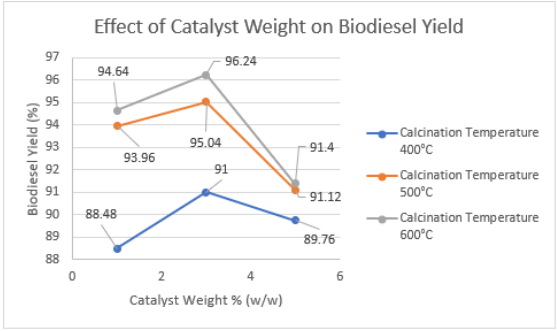

Figure 2. Effect of catalyst weight on biodiesel yield

In addition, excessive use of catalysts can also cause saponification, which affects biodiesel purification and additional glycerol production, thereby reducing biodiesel yield (Zhao, Chen, Wu, & Shan, 2023).

Physicochemical Characteristics of Biodiesel Produced from Waste Cooking Oil

The FT-IR spectra of both samples are shown in Figure 3. In waste oil, there are absorption peaks at 1747.51 cm^{-1} and 1165 cm^{-1}, which indicate the stretching vibrations of the C=O and C–O bonds of the triglyceride ester group. The bands at 2952.72 cm^{-1} and 2852.71 cm^{-1} indicate the stretching vibrations of C–H alkane bonds, while the band at 3007.02 cm^{-1} indicates C–H alkene bonds. The absorption at 723.31 cm^{-1} indicates the presence of CH_2 groups.

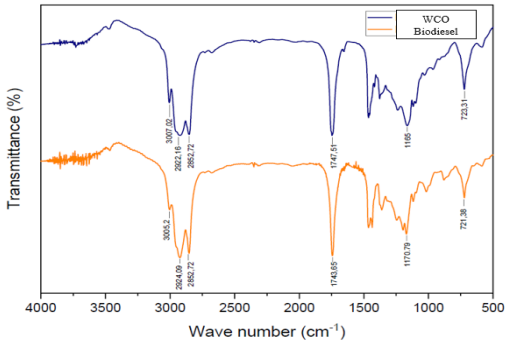

Figure 3. FT-IR spectrum of waste cooking oil and biodiesel produced

Figure 4 shows the results of GC-MS analysis, displaying six peaks with different retention times and percentage area, indicating the presence of six main components of methyl ester.

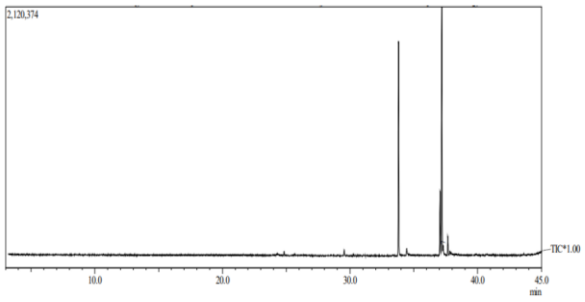

Figure 4. GC-MS chromatogram of biodiesel produced using Trenggalek durian peel ash catalyst

Based on fragmentation pattern analysis, the dominant components in biodiesel produced by transesterification of used cooking oil assisted by ultrasonic waves with K_2O heterogeneous catalyst from Trenggalek durian peel ash (600°C, 3% w/w) include methyl laurate (0.78%), methyl myristate (1.14%), methyl palmitate (37.67%), methyl linoleate (12.02%), methyl oleate (44.89%), and methyl stearate (3.50%). This composition has been verified with the distribution of methyl esters commonly reported in other biodiesels (Emmanouilidou, Lazaridou, Mitkidou, & Kokkinos, 2023; Mulkan et al., 2023).

The physicochemical properties shown in Table 2 play an important role in standardizing the quality of biodiesel produced. Fuel density affects the atomization process, combustion efficiency, and fuel requirements to produce optimum engine power. Kinematic viscosity determines the flow behavior of biodiesel at low temperatures, while the acid number indicates the level of degradation and potential corrosion in the fuel system, so it must be low. The refractive index is an optical and chemical parameter that reflects the stability and combustion quality of biodiesel, although it does not directly affect engine performance. The saponification number is used to predict the chemical character of biodiesel and its tendency to form soap, while the iodine number indicates the degree of unsaturation associated with susceptibility to oxidation, which ultimately affects the stability and shelf life of biodiesel (Suhartini et al., 2021).

Table 2. Physicochemical properties of biodiesel produced

Calcination Temperature (°C)	Catalyst Weight % (w/w)	Parameter					
		Density (kg/cm^2)	Viscosity (mm^2/s)	Refractive Index	Acid Value (mg KOH/gr)	Saponification Value (mg KOH/gr)	Iodine Value (mg KOH/gr)
SNI 7182-2015		850-860	2.3-6	1.45-1.475	<0,5	-	<115
400ºC	1	920	9.53	1.474	1.55	181.81	38.52
400ºC	3	912.3	8.29	1.467	0,.15	203.98	35.35
400ºC	5	897.2	5.08	1.463	0.308	207.19	39.98
500ºC	1	890.0	4.07	1.468	0.602	206.16	39.04
500ºC	3	887.4	5.45	1.463	0.308	202.97	31.12
500ºC	5	888.2	5.08	1.465	0.619	203.98	40.70
600ºC	1	889	4.80	1.465	0.622	204.39	38.52
600ºC	3	884.6	4.45	1.457	0.615	202.77	33.52
600ºC	5	886.2	4.72	1.455	0.619	206.36	33.39

Reusability of Trenggalek Durian Peel Ash Catalyst

This study covers the synthesis of biodiesel and the evaluation of *the* reusability of K_2O heterogeneous base catalysts derived from Trenggalek durian peel ash. Testing was carried out on the catalyst with the best performance, namely the catalyst calcined at 600°C with a concentration of 3% (w/w), which produced the highest yield. The test was carried out in three cycles to assess the decrease in catalyst activity due to agglomeration and the covering of active sites by impurities on the catalyst surface. Before reuse, the catalyst was washed with n-hexane and acetone. Washing with n-hexane was intended to remove oil residues and non-polar compounds, while acetone was used to clean the remaining polar contaminants.

Table 3. Biodiesel yield in reusability tests

Cycle	Oil (gram)	Biodiesel (gram)	Yield (%)
1	25.00	24.06	96.24
2	25.00	20.26	81.07
3	25.00	15.30	61.20

Test results (Table 3) show that biodiesel yield decreased in each cycle. The first cycle yielded 96.24%, indicating that the catalyst remained active. The yield decreased to 81.07% in the second cycle due to a reduction in K_2O content, and decreased significantly to 61.20% in the third cycle, indicating a drastic decrease in catalytic efficiency. This reduction is attributed to the loss of K_2O basic active sites responsible for facilitating the transesterification reaction (Santoso et al., 2024).

CONCLUSIONS

Trenggalek durian peel has the potential to serve as a heterogeneous catalyst in the transesterification of used cooking oil due to its K_2O content. Optimum conditions were obtained at a catalyst loading of 3% (w/w) and a calcination temperature of 600°C, resulting in the highest biodiesel yield of 96.24%. It exhibits physicochemical properties that comply with the SNI 7182:2015 standard. The catalyst demonstrates *reusability* for up to three cycles with a gradual decrease in activity.

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