

RESPONSE SURFACE METHODOLOGY (RSM) FOR GREEN ULTRASOUND-ASSISTED TRANS-ESTERIFICATION OF WASTE PALM COOKING OIL USING HETEROGENEOUS CATALYSTS FROM CALCINED DURIAN PEEL ASH

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ABSTRACT

Waste cooking oil is abundant and a source of environmental pollution, even though it has great potential as a raw material for biodiesel. The ash of Durian peel waste contains metal oxides that have the potential as base catalysts. The purpose of this study was to optimize the production of environmentally friendly biodiesel from waste cooking oil with Durian peel ash as a catalyst with Response Surface Methodology (RSM). Synthesis of heterogeneous catalysts (K_2O) of Durian peel ash through calcination with temperature variations of 400, 500, and 600 °C for 5 hours. The transesterification reaction was carried out under ultrasonic waves using a molar ratio of methanol and oil of 12:1 with variations in catalyst concentration of 1, 3, and 5% (w/w), reaction temperature of 65 °C. The optimal conditions for transesterification of waste cooking oil after RSM analysis were the calcined catalyst at 600°C, the concentration of catalyst of 3% w/w, with a yield of 96.24 %. The results of biodiesel, including density, viscosity, refractive index, acid number, saponification number, and iodine number, meet SNI 7182:2015 for biodiesel. The results of IR and GC-MS analysis of the main components of the methyl esters consist of methyl 9-octadecenoate (44.89%), methyl hexadecanoate (37.67%), methyl 9,12-octadecadienoate (12.02%), methyl octadecanoate (3.50%), and methyl tetradecanoate (1.14%). This study offers a green process utilizing Durian peel ash as a local waste.

Keywords: Biodiesel, Durian Peel Ash, Ultrasonic, Transesterification, RSM

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INTRODUCTION

High fuel demand globally has raised concerns, especially in Indonesia. Indonesia's dependence on fossil fuels is still very high, while oil reserves continue to decline. It is important to explore a fuel that is renewable, environmentally friendly (biodegradable), and non-toxic, for example, biodiesel.¹ Biodiesel, as an alternative green diesel fuel, is produced through transesterification and esterification reactions of triglycerides with short-chain alcohols.² One of the economic raw materials for biodiesel is waste cooking oil resulting from repeated frying of food.³ Waste cooking oil is not good to be reused in food processing because it is carcinogenic as a result of the content of radical compounds such as peroxide and hydroperoxide.⁴ Waste cooking oil can also potentially become a pollutant because it can pollute the soil, water, and drains, and disrupt the ecosystem if disposed of directly without processing.⁵ Biodiesel can be produced from triglycerides through transesterification and esterification reactions using short-chain alcohol reactants with the addition of catalysts.⁶ Biodiesel has a structure in the form of a long carbon chain of 12 to 20 bound to one ester group containing two oxygen atoms.⁷ The advantages of biodiesel include being renewable, environmentally friendly, non-toxic, and having low carbon emission levels.⁸ The government has determined the quality of biodiesel products with reference to SNI 7182-2015. Biodiesel production technology with transesterification reactions continues to develop, starting from conventional methods accompanied by mechanical stirring, then using supercritical alcohol, until the

reaction rate can be accelerated using ultrasonic waves. Good quality biodiesel is resistant to oxidants (air).⁹ In the biodiesel transesterification reaction, a catalyst is also used to accelerate the reaction by reducing the activation energy.¹⁰ In general, two types of catalysts can be used in biodiesel synthesis, namely homogeneous and heterogeneous catalysts. Homogeneous catalysts have several disadvantages, such as being difficult to separate from the reaction products and not being environmentally friendly because they are corrosive.¹¹ The use of heterogeneous base catalysts is considered more efficient because they have several advantages, including being non-corrosive, environmentally friendly, easily separated from liquid products, and having relatively high activity and selectivity. One of the strong base groups as a heterogeneous catalyst is Potassium Oxide (K_2O).¹² Potassium (K) in the K_2O compound can increase the active phase and has good catalytic activity.¹³ K_2O can be made using biomass waste such as fruit peels. Several studies have reported the use of fruit peel ash as a heterogeneous base catalyst (K_2O) in transesterification reactions, such as ash from avocado peel waste¹⁴, banana peel^{15,16}, cocoa peel¹⁷, etc. One type of fruit peel as a source of the alkali is Durian peel. Durian peel is a waste with a relatively large amount of around 86% K_2O .¹⁸ The K_2O content in Durian peel is the largest component compared to other compounds such as P_2O_5 , MgO , CaO , SiO_2 , etc. One of the superior national Durian varieties that has been certified is the local Durian from Trenggalek. Trenggalek is one of the largest durian-producing areas in Indonesia, which also has a tourist village and the largest international Durian forest area in Southeast Asia. Therefore, local Durian peel waste in Trenggalek has the potential as a raw material developed into a heterogeneous base catalyst in biodiesel synthesis, and there is no report study. Durian peel ash is useful in methanolysis reactions to produce biodiesel. The previous results showed that Durian peel ash with a calcination temperature of 600 °C for 8 hours proved to be effective in the transesterification of rubber seed oil, with the optimum reaction conditions obtained at a temperature of 65 °C for 1 hour with a catalyst concentration of 10% w/w.¹⁹ Likewise, the other results stated that Durian peel ash calcined at a temperature of 600 °C successfully became a catalyst in the transesterification reaction at room temperature using a homogenizer from palm oil with a catalyst concentration of 5% w/w.¹⁸ Therefore, it can be seen that several previous studies have studied the catalytic activity of Durian peel ash with a calcination temperature range of 600 – 1000 °C. By considering energy efficiency, it is necessary to conduct a study on Trenggalek Durian peel ash catalyst with a calcination temperature below 600 °C. Furthermore, the purposes of this study are (1) to determine the optimal conditions for the transesterification of waste cooking oil with a heterogeneous catalyst (K_2O) from local Durian peel ash from Trenggalek, assisted by ultrasonic waves, and (2) to determine the characteristics of biodiesel resulting from the transesterification by testing the physico-chemical properties according to the SNI 7182:2015.

EXPERIMENTAL

Material and Methods

The materials used in this study were samples of Trenggalek Durian peel waste, waste cooking oil from Malang market (Indonesia), anhydrous magnesium sulfate ($MgSO_4$), active natural zeolite from Malang, technical ethanol, technical concentrated sulfuric acid (H_2SO_4), potassium hydroxide (KOH, p.a.), sodium hydroxide (NaOH, p.a.), technical methanol, technical acetone, technical n-hexane, silica gel TLC plate, and distilled water.

General Procedure

The synthesis of biodiesel was carried out as follows: (1) synthesis of K_2O , (2) refining waste cooking oil, and (3) esterification-transesterification.

Synthesis of (K_2O) from Durian Peel Waste

The synthesis of the K_2O catalyst from Durian peel waste was carried out through a calcination process. First, the Durian peel sample was cleaned and cut into smaller pieces. It was then ovened at 200 °C for 2 hours. It was then smoothed using a blender and calcined for 5 hours at 400 °C, 500 °C, and 600 °C as independent variables. So, it resulted in a heterogeneous base catalyst in the form of Durian peel ash. The catalyst was then cooled in a desiccator for 24 hours. After that, the catalyst was placed in a closed container.

Refining Waste Cooking Oil

The initial treatment of waste cooking oil was carried out by refining using natural zeolite. Refining was carried out by adding natural zeolite with a concentration of 30% into waste cooking oil, then it was heated with stirring for 1 hour. It was then centrifuged to separate the zeolite and reheated at 100 °C.

Synthesizing Biodiesel from Waste Cooking Oil with the Synthesized Catalyst

Biodiesel synthesis was carried out in two stages, namely the esterification and transesterification stages. The esterification stage was carried out using the reflux method. The stage was started by heating 100 grams of the refined oil at 65 °C. Methanol was then added based on a ratio of methanol: waste cooking oil, namely 5, 1 and sulfuric acid (H_2SO_4) (1%) of the oil mass. The reflux process was carried out with stirring at 65 °C for 3 hours. Furthermore, the esterification results were transferred into a separating funnel and left for a while until two layers were formed. The bottom layer was separated (suspected to be methyl ester), it was then washed using warm distilled water (50 – 60 °C) and then heated at 100 °C to reduce the water content. The esterification results were tested for acid number again, and it was ensured that the free fatty acid content was less than 2% before proceeding to the transesterification stage. The transesterification stage using reflux was carried out by: 25 grams of esterified oil sample was put into a three-necked flask. Methanol was then added based on a ratio of methanol: waste oil of 12:1 and a heterogeneous catalyst (K_2O) of Durian peel ash with a calcination temperature of 400, 500, and 600 °C, respectively, as much as 1, 3, and 5% of the amount of raw material as an independent variable. Furthermore, it was covered using aluminum foil and the transesterification process was carried out by inserting an Erlenmeyer flask containing the sample into an ultrasonic cleaning bath at an ultrasonic frequency of 37 kHz with of 65 °C for 1 hour. Furthermore, the transesterification results were centrifuged to precipitate the catalyst. Then the centrifuge results were poured into a separating funnel and left to stand until 2 layers were formed. The top layer (biodiesel) was then washed with warm distilled water (50 – 60 °C) several times and heated at 100 °C with anhydrous MgSO_4 to remove water.

Detection Method

The characteristics of the K_2O catalyst was analyzed using X-Ray Fluorescence (XRF) (PANalytical, Minipal 4). The characteristics of methyl esters (biodiesel) composition was analyzed using Fourier Transform Infra-Red (FT-IR) Spectroscopy (Shimadzu, IR Prestige 21) and Gas Chromatography Mass Spectrometry (GC-MS) (Shimadzu, QP2010 Plus). The biodiesel was tested for physicochemical properties: density, viscosity, refractive index, acid number, saponification number, and iodine number.

RESULTS AND DISCUSSION

Characterization of K_2O Catalyst from Durian Peel Waste

A heterogeneous base K_2O catalyst sourced from local Durian peel waste was obtained through the calcination method. After the furnace, the Durian peel samples become ash. The results can be seen in Fig.-1 and Table-1.

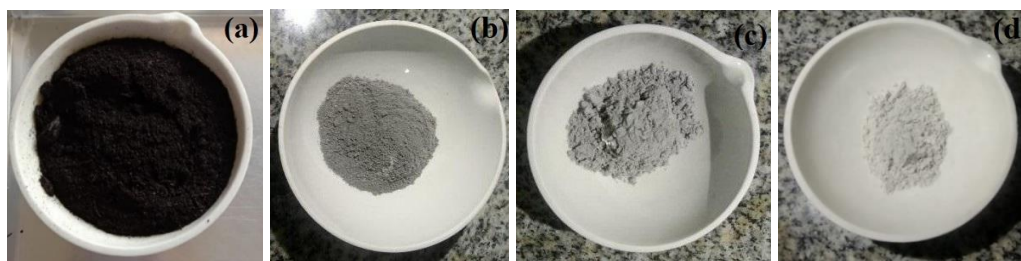


Fig.-1: Color Changes in Durian Peel Ash: (a) before Calcination and after Calcination at: (b) 400 °C, (c) 500 °C, and (d) 600 °C

Based on the data in Table-1 and Fig.-1, it can be seen that the relationship between calcination temperature and Durian peel ash yield is inversely proportional. The higher the calcination temperature, the lower the yield value obtained. Organoleptically, the characteristics of the Durian peel ash also have clear differences. Where the higher the calcination temperature, the whiter the ash produced will be. Furthermore, based on the XRF result (Fig.-2), it can be ensured that the catalyst has been made with the right raw materials.

Table-1: Yield of Durian Peel Ash

Calcination (°C)	Yield (%)
400	78.25
500	39.45
600	24.15

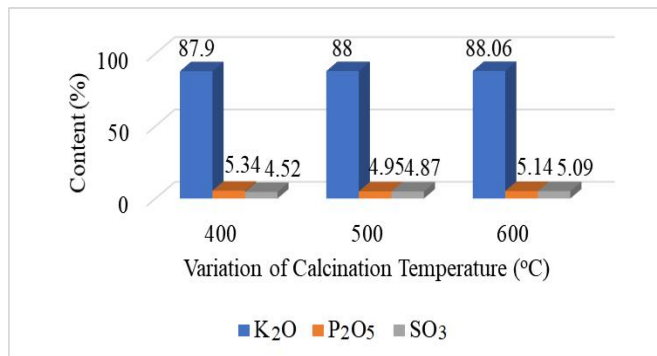


Fig.-2: XRF Test Results of Durian Peel Ash with Variation of Calcination Temperature

Subsequently, Durian peel ash has the main element component being potassium (K) and the largest oxide compound being potassium oxide (K₂O). This is in accordance with the theory, which states that the main content of Durian peel ash is the potassium element (K).¹⁹ Based on the results of the XRF test of Durian peel ash in Fig.-2, the increase in the calcination temperature removes the organic material contained and increases the concentration of K₂O. The released organic material removes the organic mass, while the potassium element or compound contained undergoes decomposition into K₂O.²⁰

Biodiesel Results of Waste Cooking Oil using Durian Peel Ash (K₂O) Catalyst

Before transesterification, the esterification must be carried out to ensure the free fatty acid is less than 2%. After esterification, a free fatty acid level test showed that the free fatty acid level of the esterified oil was 0.16%. This value indicates that the esterification reaction can reduce the free fatty acid content contained in waste cooking oil from the original 2.22%. The transesterification was carried out using the reflux method. Furthermore, the determination of optimum conditions for biodiesel synthesis at various catalyst calcination temperatures and catalyst concentrations can be analyzed statistically using response surface methods (RSM). RSM was used in equation (1).

$$z = ax + by + cx^2 + dy^2 + fxy$$

(1)

The results can be seen more clearly in Fig.-3.

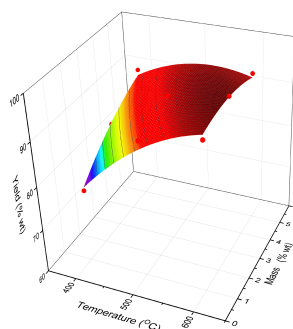


Fig.-3: Effect of Calcination Temperature and Catalyst Concentration on Transesterification Results Using Durian Peel Ash Catalyst

Based on the Fig.-3, it can be seen that the transesterification reaction with variations in calcination temperature (400, 500, and 600 °C) and variations in catalyst concentration used (1, 3, and 5 %) will produce different yields. Using a catalyst with a calcination temperature of 400 °C, the transesterification result is the lowest yield, due to the low catalytic activity of the catalyst. While using a catalyst with a

calcination temperature of 500 °C and 600 °C, the highest yield was obtained using a catalyst concentration of 3% w/w, which then decreased at a concentration of 5% w/w. This is due to the adsorption of the synthesis product by the catalyst pores due to the addition of excess solid catalyst. Subsequently, optimum conditions based on yield achieved in the area indicated by dark red color were achieved at a temperature of 600 °C, a concentration of 3% and a yield of 96.24%. From the products, the physical and chemical properties were tested, and then their potential was compared with the SNI 7182-2015 in Table-2.

Table-2: Characteristics of Biodiesel of Waste Cooking Oil using K₂O Durian Peel Ash

Calcination set of K ₂ O (°C)	[Catalyst] (b/b)	Density (kg/cm ²)	Viscosity (mm ² /s)	Refractive index	Acid value (mg KOH/gr)	Saponification value (mg KOH/gr)	Iod value (mg KOH/gr)
SNI 7182-2015		850 – 860	2.3 – 6	1.45 – 1.475	< 0.5	< 500	< 115
400	1	923.4	19.270	1.475	0.620	174.82	38.30
400	3	909.8	13.070	1.469	0.617	189.07	43.93
400	5	890.6	2.600	1.461	0.309	206.16	39.95
500	1	911.4	7.208	1.462	0.310	202.97	37.19
500	3	888.0	2.452	1.462	0.308	202.57	37.19
500	5	899.8	2.500	1.467	0.308	200.59	37.60
600	1	890.8	3.210	1.456	0.310	203.78	40.70
600	3	890.8	3.080	1.457	0.307	203.58	43.92
600	5	890.6	2.810	1.461	0.309	206.36	42.30

Based on Table-2, the values obtained in the density test on all methyl esters resulting from the transesterification generally meet the biodiesel quality according to SNI 7182:2015. This study revealed that using Durian peel ash can produce good biodiesel. On the other hand, the Durian peel ash can be promoted to be a green catalyst. Furthermore, analysis using FT-IR (Fig.-4) shows that the IR of the waste cooking oil and biodiesel from transesterification is almost similar, namely showing the first absorption band at a wave number of 3005.2 cm⁻¹, which appears with strong and sharp intensity, indicating the presence of stretching vibration of C-H alkene bonds. The second absorption band at a wave number of 2924.09 cm⁻¹ with strong and sharp intensity indicates the presence of a stretching vibration. The third absorption band at a wave number of 1743.65 cm⁻¹ with strong and sharp intensity indicates the presence of stretching vibration of typical C=O bonds as esters. The fourth absorption band at a wave number of 1170.79 cm⁻¹ with weak and sharp intensity indicates the presence of the stretching vibration of C-O bonds. The fifth absorption band at a wave number of 721.38 cm⁻¹ with medium sharp intensity. Based on the results of the IR spectrum analysis, it can be seen that the waste cooking oil and biodiesel products from transesterification have similar functional groups (ester groups). This indicates that triglycerides in the waste cooking oil have been successfully converted into methyl esters, which are the main components of biodiesel.²¹ GC-MS analysis was conducted to determine the compounds and ester content contained in biodiesel as the product of transesterification. Identification was carried out based on the peaks that appeared at each retention time in the GC-MS chromatogram.²² The GC-MS chromatogram of the methyl esters is shown in Fig.-5.

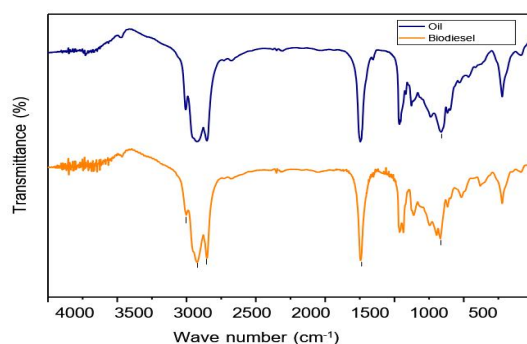


Fig.-4: IR Spectrum of Waste Cooking Oil and the Synthesized Methyl Ester by Durian Peel Ash Catalyst

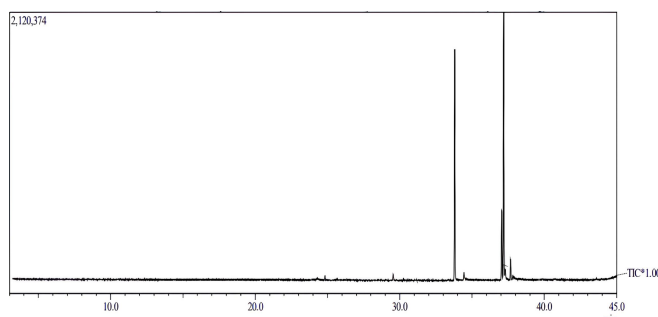


Fig.-5: GC-MS Chromatogram of the Methyl Ester of Waste Cooking Oil using K_2O Durian Peel Ash Catalyst

The example of the mass spectrum analysis of methyl ester synthesis result with a retention time (t_R) of 37.191 minutes is shown in Fig.-6.

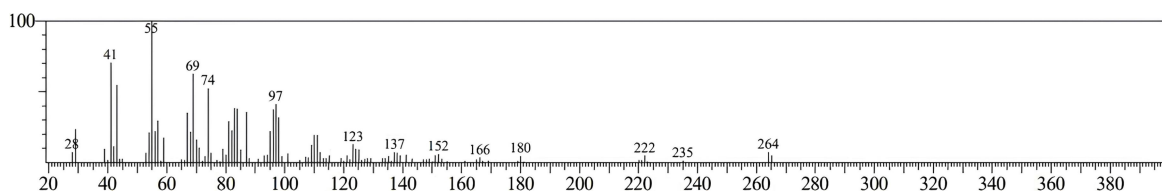


Fig.-6: Mass Spectrum of the Methyl Ester Chromatogram at $t_R = 37.191$ Minutes

The mass spectrum in Fig.-6 is then compared to the peaks that are relatively similar to the mass spectrum listed in the NIST62.LIB library. The compound in Fig.-6 has a 96% similarity to the NIST62.LIB library reference number of 42154 as methyl oleate. Furthermore, based on the analysis of the fragmentation pattern of the peaks at the retention time, the methyl ester compounds are listed in Table-3.

Table-3: Composition of the Biodiesel Product of Waste Cooking Oil using K_2O Durian Peel Ash

Compound Name	Molecular Formula	Content (%)
Methyl dodecanoate	$C_{13}H_{26}O_2$	0.78
Methyl tetradecanoate	$C_{15}H_{30}O_2$	1.14
Methyl hexadecanoate	$C_{17}H_{34}O_2$	37.67
Methyl 9,12-octadecadienoate (Z, Z)	$C_{19}H_{34}O_2$	12.02
Methyl 9-octadecenoate	$C_{19}H_{36}O_2$	44.89
Methyl octadecanoate	$C_{19}H_{38}O_2$	3.50
Total		100

Reusability of the Synthesized Catalyst

In this study, the results of the XRF analysis of the K_2O content in the catalyst after being used for transesterification decreased from around 88% to around 67% of K_2O content. The results of the reusability test with 3 replications. There was a significant decrease in yield. In the first cycle of transesterification, a yield of 96.24% was obtained, indicating that the catalyst was still in good condition and effective. Then, there was a decrease in the second cycle transesterification with a yield of 81.07% indicating a decrease in catalyst activity due to the loss of K_2O , which affected its efficiency. A further decrease occurred in the third cycle, indicating a significant decrease in catalyst effectiveness with a yield of 61.20%. This large decrease in yield was due to the insufficiency of the amount of active K_2O to facilitate the reaction.¹⁸

CONCLUSION

Based on the presented data, it can be summarized that local Durian peel has the potential as a heterogeneous catalyst for ultrasound-assisted transesterification reactions in waste cooking oil by utilizing the K_2O content contained therein, and works optimally at a concentration of 3% w/w with the calcined ash (600 °C), producing the highest yield of 96.24%. The results of the physical (density, viscosity, refractive index, acid value, saponification value, and iod value) and chemical properties of the biodiesel products obtained from the transesterification of waste cooking oil have met the standards

referring to SNI 7182:2015. Furthermore, the reusability of the catalyst can be used for transesterification up to 3 cycles.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-7: Affordable and Clean Energy*. All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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