

Calcined Biowaste Durian Peel as a Heterogeneous Catalyst for Room-Temperature Biodiesel Production Using a Homogenizer Device

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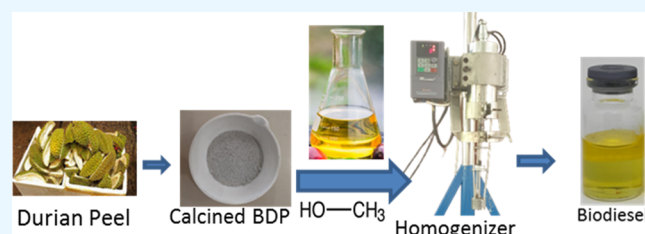
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ABSTRACT: Calcined biowaste durian peel (BDP) contains 86% potassium element as the main compound and has successfully catalyzed the transesterification of palm oil to biodiesel at room temperature. The effect of catalyst weight, molar ratio of palm oil to methanol, reaction time, and rotational speed of the homogenizer device was investigated on biodiesel conversion and yield. The highest biodiesel conversion of $97.4 \pm 0.3\%$ was achieved using the following reaction conditions: a catalyst weight of 5 wt %, a molar ratio of palm oil to methanol of 1:15, a reaction time of 10 min, and a rotational speed of 6000 rpm. Unfortunately, calcined BDP could not hold its catalytic activity in the reusability study. The biodiesel conversion was decreased in the second cycle due to the decrease of both catalyst weight and concentration of potassium ions after the first cycle. However, the calcined BDP paired with a homogenizer device could produce biodiesel in a short reaction time and at room temperature.



INTRODUCTION

Currently, biodiesel production uses a homogeneous substance as a catalyst due to its high activity to catalyze transesterification reactions in a short reaction time.¹ Even though it is widely available and relatively cheap,² the after-reaction treatment, such as neutralization of the catalyst and purification product, is required. Another problem arises related to the provision of a large volume of water for the postproduction processes and pretreatment of the wastewater before release to the environment.^{3,4} Therefore, the total biodiesel production cost is increased, which could reduce its competitiveness in the fuel market toward petro-diesel fuel. In addition, oil/lipid raw materials have to contain free fatty acids (FFAs) of less than 2% to avoid saponification,⁵ hence limiting its ability to transesterify low-cost feedstock such as waste cooking oil, jatropha oil, rubber seed oil, microalgae oil, and other nonedible oils.^{1,6}

Heterogeneous substances have been developed to overcome homogeneous catalyst disadvantages.² Heterogeneous catalysts offer some advantages such as ease of recycling and reuse, tolerance to moisture and FFAs, noncorrosive, and environmentally friendly.^{2,7,8} However, the catalytic activity of a heterogeneous substance is limited by mass transfer due to different phases with reactants.⁹ Recently, transforming heterogeneous catalysts to form nanoparticles has reduced the mass transfer limitation. Nanocatalysts provide a large active area and better surface energy, which could increase the reaction rate.² However, an excessive volume of methanol is

required to enhance the transesterification reaction. In addition, more energy is indispensable to separate nanocatalysts from the products through centrifugation separation.⁷

Presently, heterogeneous catalysts derived from agricultural waste biomass such as banana peel, banana peduncle/trunk, cocoa pod husk, walnut shell, passion fruit peel, coconut husk, palm bunch ash, and more^{3,10–16} have been introduced and proven to have catalytic activity comparable with a homogeneous substance.¹⁷ For instance, our previous research showed that calcined waste passion fruit peel could transesterify palm oil to biodiesel in a reaction time of 30 min. A biodiesel conversion of $95.4 \pm 2.8\%$ could be achieved at room temperature.¹⁰ Interestingly, the reaction time could be reduced when reaction-intensified devices such as microwaves or homogenizers were used. Falowo et al. (2019) in microwave-intensified biodiesel production using elephant-ear tree pod husk showed that conversion of $98.77 \pm 0.16\%$ was achieved in a reaction time of 5.88 min.¹⁸ In our previous work, we successfully demonstrated that biodiesel could be produced in 10 min using a homogenizer and a palm bunch

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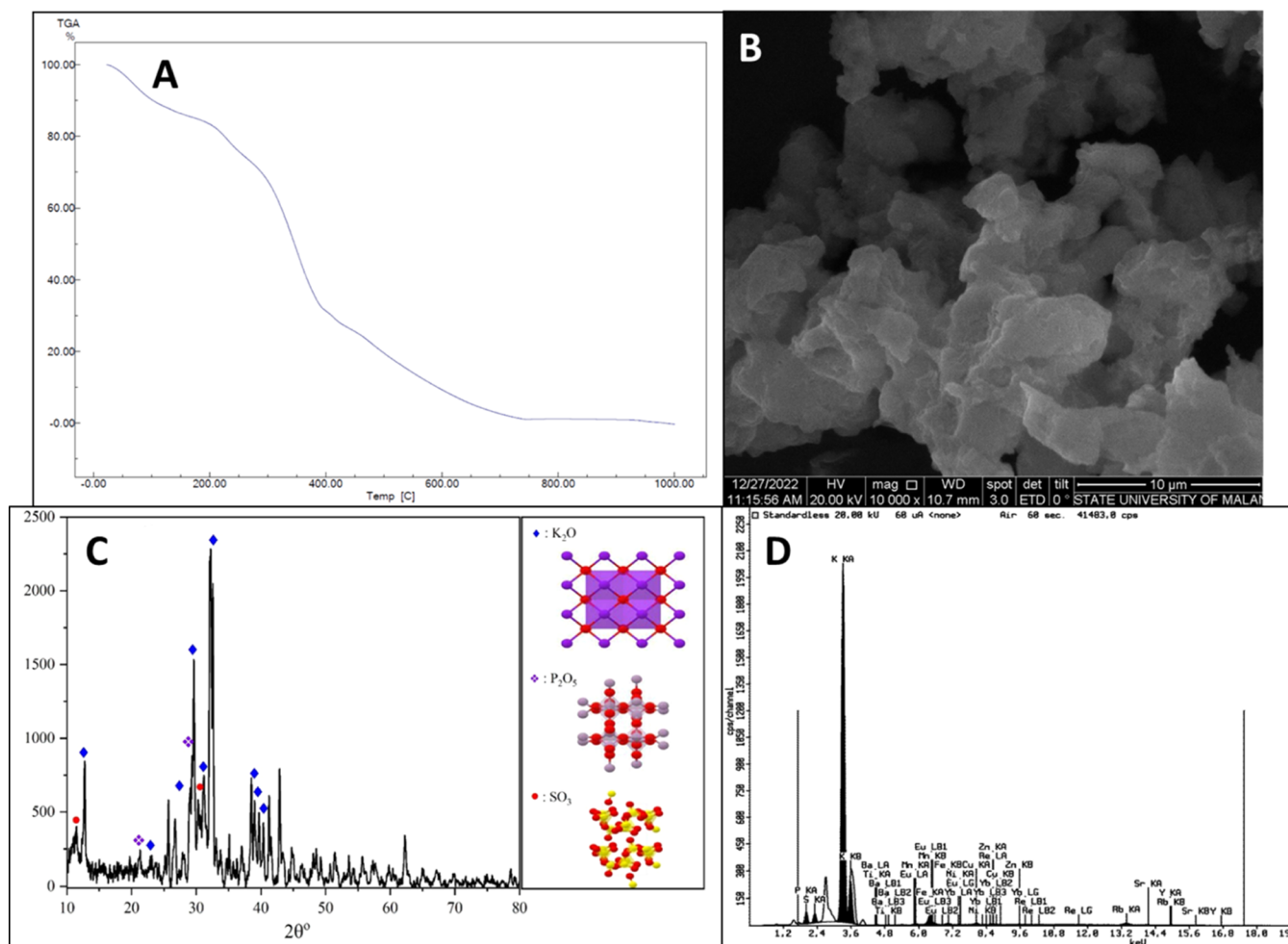


Figure 1. BDP characterization: (A) TGA thermogram, (B) SEM image, (C) XRD spectrum, and (D) XRF spectrum.

ash as a heterogeneous catalyst.¹¹ These biowaste-derived catalysts contain potassium ions as the main element in the form of potassium carbonate or potassium oxide. However, no literature has been discovered related to the utilization of BDP as a heterogeneous catalyst in biodiesel production using a homogenizer device.

Durian is a renowned and prominent fruit in the South Asia region. The durian fruit is composed of pulp/flesh, seeds, and rind or shell. Approximately 70% of durian fruit is a nonedible part and 40% among them is durian peel.¹⁹ The production of durian in Indonesia reached 1.58 million tons in 2022, with 0.632 million tons attributed to the peel.²⁰ Some researchers have utilized BDP as an adsorbent for the removal dyes^{21,22} and biocomposite.¹⁹ However, usually, the BDP was thrown away without any pretreatment which could harm the environment. Hence, this study reported the utilization of BDP as a catalyst in the transesterification of palm oil to biodiesel using a homogenizer. The BDP was calcined and characterized by X-ray diffraction (XRD), X-ray fluorescence (XRF), thermogravimetric analysis (TGA), scanning electron microscopy coupled with energy-dispersive spectrometry (SEM-EDS) and Fourier transform infrared (FT-IR). The single factor of catalyst weight (wt %), molar ratio of palm oil to methanol, reaction time (minutes), and rotational speed (rpm) was determined to establish the maximum biodiesel conversion and yield. In addition, the reusability and leaching catalysts were also determined.

EXPERIMENTAL SECTION

Materials. The BDP was collected from the local durian fruit seller in Medan, Sumatera Utara, Indonesia. The BDP was washed, dried in an oven at 110 °C overnight, crushed using a household grinder, and sieved. The BDP powder was calcined in a muffle furnace at 600 °C for 4 h and stored in a desiccator before use. All chemicals were brought from a local chemical shop and were used as received.

BDP Catalyst Characterization. The BDP powder was characterized by TGA recording using a Shimadzu DTG-60 instrument to study the decomposition and thermal stability of the catalyst. Based on the TGA data, the BDP powder was calcined at 600 °C for 4 h. The calcined BDP was characterized using XRD, XRF, SEM-EDS, and FT-IR. The XRD and XRF patterns were determined using a Shimadzu XRD-700 and a Rigaku Supermini-200, respectively. The SEM equipped with a JEOL JSM-6510LA energy-dispersive X-ray (EDS) spectrometer was used to study the surface morphology of the catalyst and the components in the calcined BDP. The FT-IR spectrum of the calcined catalyst was recorded in a PerkinElmer 100 spectrophotometer with wavelength registered from 4000 to 600 cm^{-1} .

Homogenizer-Intensified Biodiesel Production Using Calcined BDP as a Catalyst. The homogenizer consists of a rotor-stator (diameters of 25 and 35 mm, respectively) and a controllable motor with a speed range from 100 to 12,000 rpm

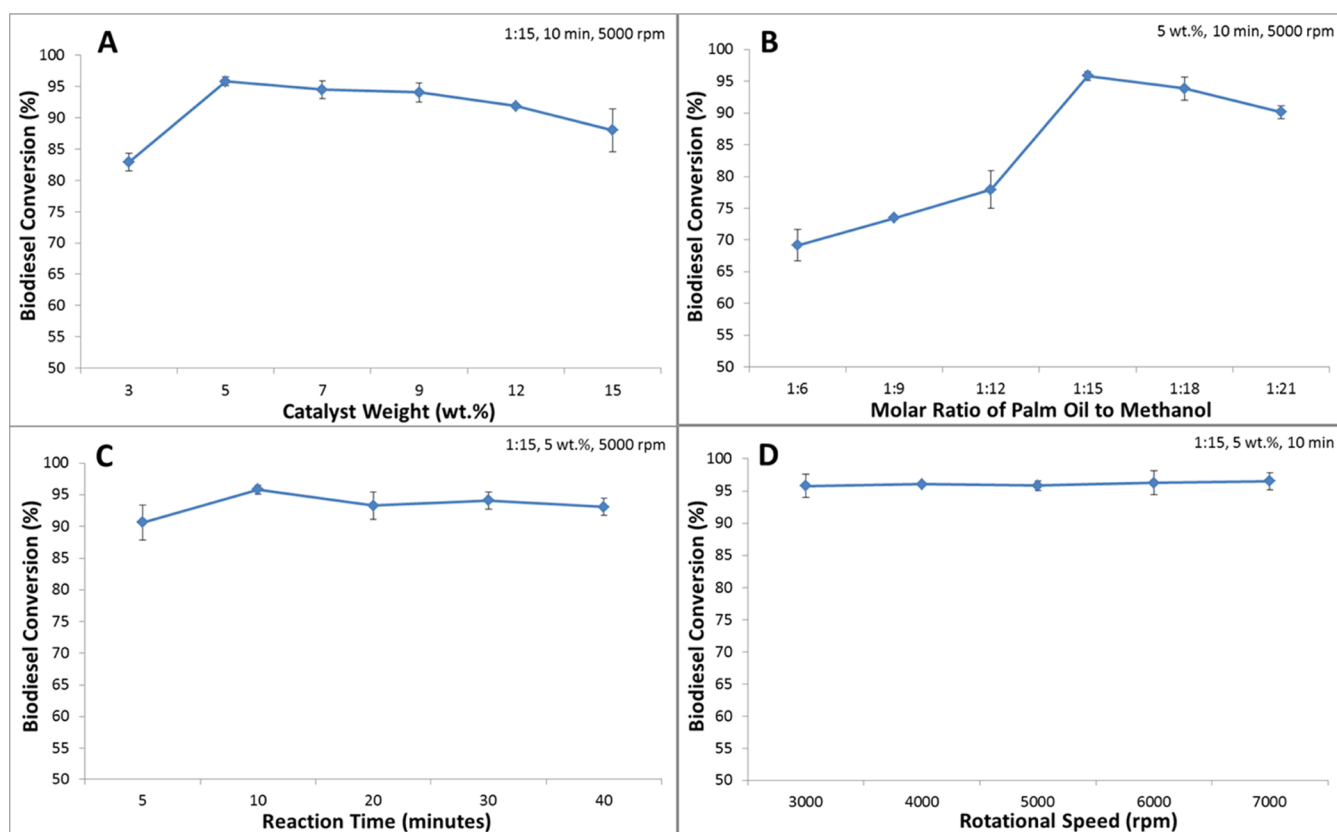


Figure 2. Effect of (A) catalyst weight, (B) molar ratio of palm oil to methanol, (C) reaction time, and (D) rotational speed on biodiesel conversion.

was used to intensify the transesterification reaction of palm oil with methanol using calcined BDP as a heterogeneous catalyst. The parameters studied were a catalyst weight of 5–12 wt % based on palm oil, a molar ratio of palm oil to methanol of 1:6–1:21 with an increment of 3 molars, a reaction time of 5–40 min, and a rotational speed of 3000–7000 rpm with an increase of 1000 rpm. 45 mL of palm oil, 10.92 mL of methanol (molar ratio of 1:15), and 1.9192 g of calcined BDP were dispersed in a cavitation reactor for 10 min at a rotational speed of 5000 rpm. The product was separated through centrifugation at 7000 rpm for 5 min. The biodiesel layer was collected and stored in a desiccator for GC analysis.

Biodiesel Conversion Analysis and Yield Determination. The biodiesel conversion was determined using GC (Shimadzu type 2010) analysis following the EN 14103 standard method, which was used and reported elsewhere.^{3,10,11,13} Methyl heptadecanoate with a known concentration (C_s) and volume (V_s) was used as an internal standard. The biodiesel conversion was calculated based on the difference peak area between all fatty acid methyl esters ($\sum A$) with an internal standard (A_s) using eq 1. The mass of biodiesel was calculated using eq 2, while the biodiesel yield was projected using eq 3.

$$C = \frac{\sum A - A_s}{A_s} \times \frac{C_s V_s}{m} \times 100\% \quad (1)$$

$$\text{Mass of methylesters} = C \times w \quad (2)$$

$$\text{Yield} = \frac{\text{mass of methylesters}}{\text{mass of palmoil}} \times 100\% \quad (3)$$

Reusability and Leaching Study. The reusability study was conducted under reaction conditions of a molar ratio of palm oil to methanol of 1:15, a catalyst weight of 5 wt %, a reaction time of 10 min, and a rotational speed of 6000 rpm. The calcined BDP catalyst was separated from the reaction mixture and was directly used for the next reaction. The biodiesel product for each cycle was collected for GC analysis. The leaching study of the catalyst was performed under the reaction condition of 4 g of calcined BDP dispersed in 80 mL of methanol at a rotational speed of 6000 rpm for 10 min reaction time. The remaining catalyst was separated through centrifugation, dried in an oven at 40 °C overnight, and stored in a desiccator. The EDS analysis was used to determine the concentration of potassium elements in the catalyst after and before the dispersion.

Statistical Analysis. All of the experiments were conducted three times, and the statistical differences between parameters were confirmed using analysis of variance (ANOVA) and Tukey's post hoc test using Statistica v14.

RESULTS AND DISCUSSION

Catalyst Characterization. The TGA analysis of the BDP catalyst was conducted first to determine the calcination temperature based on the weight losses. As shown in Figure 1A, three stages of weight loss were observed. The first stage was noticed at a temperature of 200 °C, which is water evaporation, followed by the decomposition of the organic compound at 400 °C, reducing the catalyst weight by 68.32%. The final stage was observed at a temperature of 600 °C, reducing BDP weight by 90.31% due to the decomposition of the carbonaceous material releasing carbon dioxide and carbon

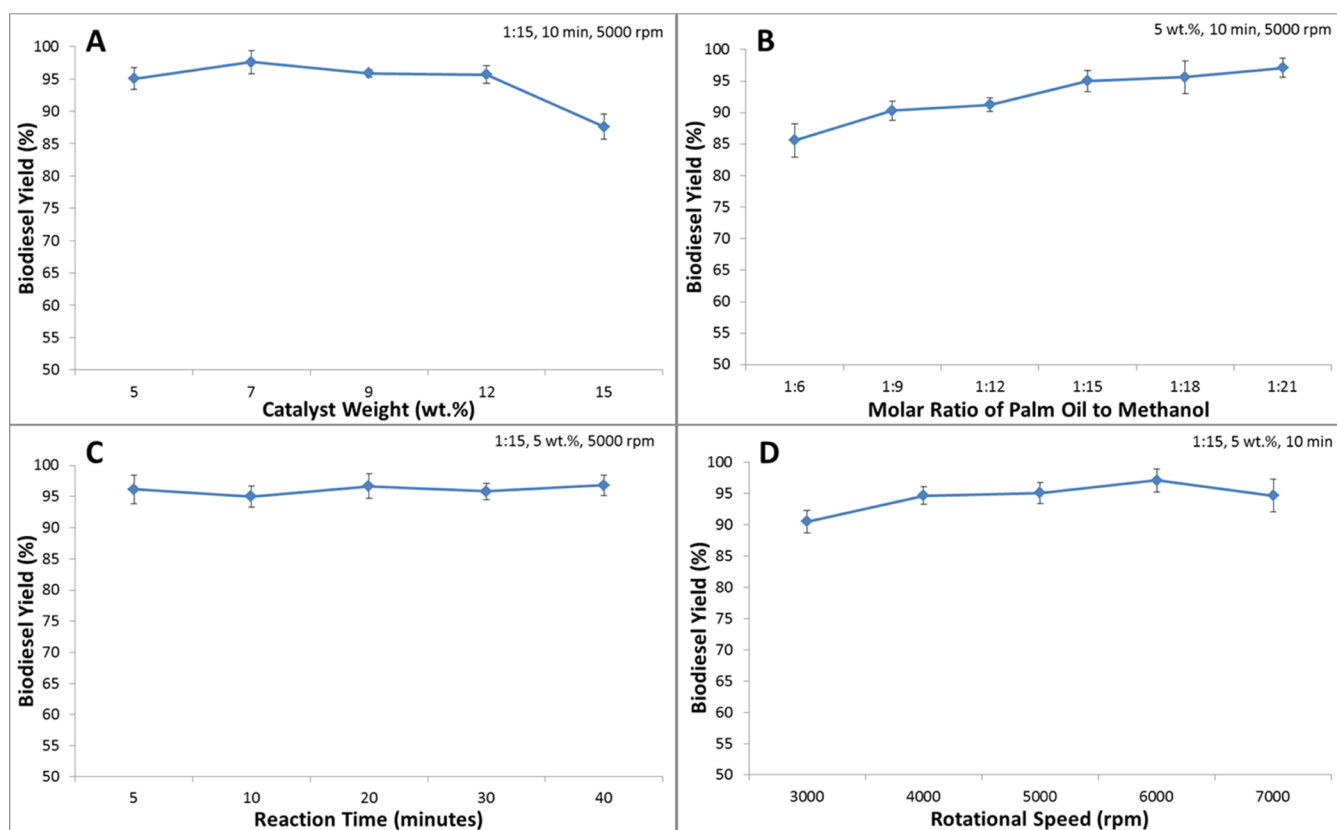


Figure 3. Effect of (A) catalyst weight, (B) molar ratio of palm oil to methanol, (C) reaction time, and (D) rotational speed on biodiesel yield.

monoxide.¹⁰ Hence the calcination temperature for waste BDP is set to 600 °C for 4 h. Figure 1B depicts that the surface morphology of the calcined BDP is not homogeneous and forms an irregular particle and lumpy structure with several pores. This is due to agglomeration during the calcination process of the BDP powder.¹¹ The agglomeration could decrease the surface area, hence reducing the catalytic activity.²³ The XRD analysis of calcined BDP showed that K_2O , SO_3 , and P_2O_5 were the main components (Figure 1C). The presence of a peak in the diffraction of 2θ at 23.01, 39.63, 12.74, 28.02, 29.48, 31.27, 32.28, 38.70, and 40.35° is a K_2O crystal that has a cubic structure (JCPDS K_2O #PDF 00–023–0493). The peak of other diffraction was also found in 2θ at 21.28 and 29.70° for P_2O_5 compounds with orthorhombic phases based on JCPDS P_2O_5 #PDF 96–231–0557 and at 10.97 and 30.41°, which showed SO_3 (JCPDS SO_3 #PDF 01–072–1664). The low intensity of the two crystals in the diffractogram indicates a small concentration of calcined BDP. This also explains that the K_2O compound is the main compound in this catalyst. Further, XRF analysis emphasizes the XRD result that K_2O is a dominant compound with a concentration of 86%, followed by P_2O_5 and SO_3 at 6.98 and 3.25%, respectively.

Effect of Calcined BDP Catalyst Weight. Catalyst concentration or weight is an important parameter to achieve a high biodiesel conversion or yield in transesterification/esterification reactions.^{24,25} The biodiesel conversion or yield tends to increase with the increase of catalyst weight up to the maximum peak and gradually decrease.^{3,10} Hence, the effect of catalyst weight on either biodiesel conversion or yield was examined at reaction conditions of a molar ratio of palm oil to methanol of 1:15, a reaction time of 10 min, and a rotational

speed of 5000 rpm by changing the catalyst weight from 5 to 15 wt %. As predicted, an increased biodiesel conversion from 82.9 ± 1.4 to $95.8 \pm 0.7\%$ was noticed when the amount of calcined BDP was increased from 3 wt % to 5 wt % (Figure 2A). The highest conversion was achieved using a catalyst concentration of 5 wt %, presumably due to the high availability of catalytic sites.²⁶ However, no increase in biodiesel conversion was detected in increasing catalyst concentration above 5 wt %. Increasing catalyst concentration could increase the viscosity of the reaction mixture, causing a decrease in biodiesel conversion.⁵ This result is in agreement with Laskar et al. (2020) in the utilization of *Mangifera indica* peel as a heterogeneous catalyst in biodiesel production from soybean. In contrast, our previous study in the transesterification of palm oil to biodiesel using palm bunch ash as a catalyst did not observe decreasing biodiesel conversion in increasing catalyst weight.¹¹

Similarly, as shown in Figure 3B, the biodiesel yield also depicted a similar pattern. The maximum biodiesel yield of $97.6 \pm 1.8\%$ was achieved using a catalyst weight of 7 wt % and tends to decrease with increasing catalyst concentration. Further, the ANOVA test revealed that catalyst weight has a significant effect either on biodiesel conversion or yield, which was driven by low conversion and yield at a catalyst concentration of 15 wt %.

Effect of the Molar Ratio of Palm Oil to Methanol. Theoretically, three moles of methanol and one mole of triglyceride are required to produce three moles of fatty acid methyl ester (FAME).¹¹ However, practically, an addition of moles is necessitated to drive the reaction to the biodiesel production.²⁴ Experiments were performed by varying the molar ratio between 1:6 and 1:12 and keeping the catalyst

weight at 5 wt %, a reaction time of 10 min, and a rotational speed of 5000 rpm. The biodiesel conversion and yield both increase with the molar ratio of palm oil to methanol. The maximum biodiesel conversion of $95.8 \pm 0.7\%$ was achieved using a molar ratio of 1:15 (Figure 2B), while the highest biodiesel yield of $97.1 \pm 1.5\%$ was achieved in a molar ratio of 1:21 (Figure 3B). The addition of methanol in the reaction system can create more chances for the reactant molecules to collide with each other. Hence, it could shift the equilibrium of the reversible transesterification toward the formation of biodiesel.²⁷ However, further increasing the methanol volume could dilute the catalyst concentration, decreasing both biodiesel conversion and yield. Ma'arof et al. (2021) in their study of the valorization of banana peel as a renewable heterogeneous catalyst in transesterification soybean oil observed a similar phenomenon and concluded that an excessive volume of methanol could increase the solubility of glycerol, affecting the separation of the biodiesel product. The significant effect of changing the molar ratio on either biodiesel conversion or yield was revealed using analysis of variance. Tukey's post hoc test showed that all parameters significantly affected the biodiesel conversion, and a low yield of $85.6 \pm 2.6\%$ at a molar ratio of 1:6 affected the biodiesel yield.

Effect of Reaction Time. The transesterification reaction of triglyceride with methanol involves three stages with each step generating one molecule of FAME.²⁷ Hence, it is important to select an appropriate reaction time to guarantee the completion of the biodiesel production. Therefore, the effect of reaction time in homogenizer-intensified biodiesel production from palm oil using calcined BDP as a heterogeneous catalyst was determined at different reaction times of 5, 10, 20, 30, and 40 min under reaction conditions of a catalyst weight of 5 wt %, a molar ratio of 1:15, and a rotational speed of 5000 rpm. The biodiesel conversion and yield were routinely achieved at $>90\%$ for all reaction time parameters with averages of 93.4 and 96.1%, respectively (Figures 2C and 3C). The biodiesel conversion is slightly increased in addition to the reaction time and forms a plateau conversion with increasing reaction time. The maximum biodiesel conversion of $95.8 \pm 0.7\%$ was achieved at a reaction time of 10 min. In contrast, the biodiesel yield did not change significantly with reaction time, and a maximum yield of $96.8 \pm 1.6\%$ was achieved at 40 min of reaction time. Our previous studies in biodiesel production using waste passion fruit peel as a heterogeneous catalyst showed a similar graphic trend.¹⁰ Similarly, the study in the utilization of palm bunch ash as a catalyst in homogenizer-intensified biodiesel production from palm oil depicted a plateau conversion.¹¹ The analysis of variance confirms the significance of the reaction time on both biodiesel conversion and yield.

Effect of Rotational Speed. One important factor in the transesterification reaction is the degree of mixing between the reactants. Triglyceride and methanol are not miscible and hence require an intense mixing to increase the contact between them.²⁸ Figures 2D and 3D show the effect of the rotational speed on biodiesel conversion and yield, respectively, in which the mixing speed is varied from 3000 to 7000 rpm in increments of 1000 rpm. Interestingly, a biodiesel conversion of $95.8 \pm 1.8\%$ was achieved at a rotational speed of 3000 rpm, and the conversion was relatively similar for all rotational speeds tested, with the highest conversion of $97.4 \pm 0.2\%$ occurring at a rotational speed of 6000 rpm. The biodiesel yield also showed a similar pattern, with the highest

yield of $97.0 \pm 1.8\%$ obtained also at 6000 rpm. The plateau graph of both biodiesel conversion and yield presumably due to the transesterification reaction have reached equilibrium at a speed of 3000 rpm; hence, either biodiesel conversion or yield was constant in increasing mixing speed.²⁹ This result is in agreement with some previously published results, which also showed that the conversion remained constant as the mixing speed increased.^{30,31} In addition, our previous study in transesterification of palm oil to biodiesel using a homogenizer and palm bunch ash as a catalyst also showed a similar graphical trend.¹¹ Further, the ANOVA test showed that the rotational speed had a significant effect on both biodiesel conversion and yield.

Reusability and Leaching Test. The reusability test was conducted to determine whether calcined BDP could be used several times without losing its catalytic activity or leaching, decreasing the biodiesel conversion in the following reaction cycle. The reaction conditions of a molar ratio of palm oil to methanol of 1:15, a catalyst weight of 5 wt %, a reaction time of 10 min, and a rotational speed of 6000 rpm, producing maximum biodiesel conversion, were used. The biodiesel conversion drastically dropped from 97.4 ± 0.3 to $36.4 \pm 2.9\%$ in the second cycle, as shown in Figure 4. Further subsequent

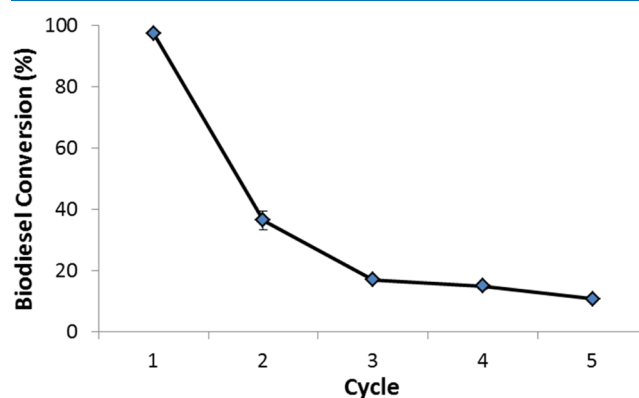


Figure 4. Reusability study of calcined BDP.

cycles showed a conversion of $10.9 \pm 0.9\%$ in the fifth cycle. To understand the cause of this decline in conversion, the leaching study was executed under the reaction condition of 4 g of calcined BDP dispersed in 80 mL of methanol at a rotational speed of 6000 rpm for 10 min of reaction time. As predicted, both catalyst weight and concentration of K_2O as the main components in calcined BDP were alleviated. The catalyst weight was reduced by 77% after the first cycle, while the concentration of potassium ions was decreased from 86 to 18%. The hydrolysis of K_2O with methanol to form KOH was presumably the main reason either the weight or concentration was decreased.^{11,32} To overcome this problem, impregnating the biobased catalyst into supported materials such as zeolite-based and graphene oxide could reduce the leaching of the catalyst.^{2,33}

Comparison of Room-Temperature Biodiesel Production Using a Biobased Catalyst. Reaction temperature is an important parameter in biodiesel production that has a linear relation with reaction rate.²⁷ The dispersion of methanol in triglyceride is increased with the increasing temperature.² However, the electricity required to operate the reactor at boiling point methanol consumes 9% of the total biodiesel production cost.^{10,34} Therefore, conducting a transesterifica-

Table 1. Summary of Room-Temperature Biodiesel Production

biomass/intensified-device	potassium concentration (wt %)	reaction condition (ratio molar oil: methanol, catalyst weight, reaction time, stirring speed)	biodiesel conversion (%)	refs
passion fruit peel magnetic stirrer	44.4	1:15; 7 wt %; 30 min; NA	95.4	10
coconut husk magnetic stirrer	55.8	1:12; 5 wt %; 180 min; NA	99.3	35
banana trunk magnetic stirrer	23.45	1:6; 7 wt %; 360 min; NA	98.4	36
mangifera indica peel magnetic stirrer	59.14	1:6; 6 wt %; 240 min; NA	98	16
banana peel homogenizer	47.01	1:15; 7 wt %; 30 min; 6000 rpm	97.7	3
palm bunch ash homogenizer	35.9	1:15; 18 wt %; 10 min; 4000 rpm	98.9	11
durian peel homogenizer	86	1:15; 5 wt %; 10 min; 6000 rpm	97.4	this study

tion reaction at room temperature is necessary to reduce the biodiesel price. Table 1 recaps the room-temperature biodiesel production using biomass-derived heterogeneous catalysts, particularly those that have potassium elements as the main compounds. A biodiesel conversion of >95% was achieved for all studies listed, even when using banana trunks, which have the lowest potassium ions. However, the reaction takes place over a prolonged reaction time. In terms of reaction time, homogenizer devices could facilitate transesterification faster than the traditional agitation method. Either palm bunch ash or calcined BDP could produce a high biodiesel conversion in a reaction time of 10 min. However, the catalyst concentration used in homogenizer-intensified biodiesel production using palm bunch ash as a heterogeneous catalyst was 2.4 larger than using calcined BDP. Hence, it can be concluded that the concentration of potassium element has a positive effect on biodiesel conversion. The molar ratio of oil to methanol varied from 1:6 to 1:15. The homogenizer-intensified method requires an excessive amount of methanol to increase the diffusion rate between reactants.³

CONCLUSIONS

This study presents a heterogeneous catalyst derived from the calcination of durian peel for the transesterification reaction of palm oil to biodiesel. Characterization of calcined BDP showed that K₂O was the main component. The biodiesel conversion routinely achieved >90 for all experiments, with the highest of 97.4 ± 0.3% obtained under reaction conditions of a catalyst weight of 5 wt %, a molar ratio of palm oil to methanol of 1:15, a reaction time of 10 min, and a rotational speed of 6000 rpm. Even though the calcined BDP was categorized as a heterogeneous catalyst, the reusability study showed that the biodiesel conversion decreased sharply after the first cycle. Further, the leaching study revealed that the potassium element was reduced by 79%. However, impregnating the calcined BDP catalyst on the solid and highly porous materials could increase the reusability of the catalyst. In addition, the calcined BDP could catalyze the transesterification reaction in short reaction time and at room temperature conditions.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.3c09642>.

Analysis of variance and Tukey's post hoc test for biodiesel conversion and yield of the homogenizer-intensified transesterification of palm oil using calcined BDP as a heterogeneous catalyst (PDF)

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Notes

The authors declare no competing financial interest.

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