International Journal of
Innovation in Mechanical Engineering & Advanced Materials (IJIMEAM)
https://publikasi.mercubuana.ac.id/index.php/ijimeam

Vol.5 (No.1). 2023. pp. 9-15
Published online: April 1, 2023
ISSN: 2477-541X

BIODIESEL PRODUCTION FROM WASTE FISH CANNING OIL USING COCOPEAT ASH CATALYST

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Abstract

Biodiesel is an environmentally friendly alternative fuel for diesel engines. The research studies the extraction process of biodiesel from waste produced by a fish canning factory using a heterogeneous catalyst known as cocopeat ash. The experiment was designed utilizing a completely randomized design with two factorial treatments and four repetitions. The primary factor under investigation was the composition of the catalyst, specifically 3%, 5%, and 7% w/v methanol. The second factor examined was the reaction time of either 60 minutes or 120 minutes. Data analysis revealed variations in density, viscosity, acid number, and Free Fatty Acids (FFA) in fish oil before and after refinement. The treatment that yielded the highest results was A3B2, featuring a catalyst composition of 7% and a reaction time duration of two hours, which achieved an impressive biodiesel yield of 81%. Moreover, several parameters tested for compliance with SNI-04-7182-2015 standards showed positive outcomes. These parameters include a density value measuring 876.3 kg/m³, the flash point around 160°C, iodine number reaching 16.36 g/100g, and heating value 47.47 MJ/Kg.

Keywords: Biodiesel, Composite Catalyst, Reaction Time, Cocopeat Ash, Waste Fish Oil

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DOI: 10.22441/ijimeam.v5i1.19936

1. Introduction

According to *Badan Pusat Statistik*, in 2015, the number of motorized vehicles in Indonesia reached 99 million units, an increase of around 9.48% from the previous year. Therefore, various efforts have been made to find alternative fuels that are renewable and environmentally friendly, one of which is biodiesel. Biodiesel is a renewable fuel that can be used as a substitute for diesel fuel, made from natural resources and used in diesel engines. In the Muncar sub-district of Banyuwangi, East Java, Indonesia, fish waste stands out as an up-and-coming natural resource with significant potential for biodiesel production. This factor holds advantages for villages in the area as it not only aids in waste reduction but also serves as an alternative fuel source. However, due to the high FFA (Free Fatty Acid) content in fish oil, further research is required to explore the purification and characterization methods when utilizing fish oil as a primary ingredient for biodiesel manufacturing.

In order to expedite the production of biodiesel, a catalyst is essential. This catalyst lowers the activation energy of the reaction, enabling it to occur at a faster rate. The catalyst is considered homogeneous when it is in the same phase (liquid) as the reactants.

Meanwhile, catalysts in a different phase from the reactants (can be solids, immiscible liquids, or gases) are referred to as heterogeneous catalysts[1]. The conventional method of biodiesel synthesis usually involves a homogeneous catalyst in the reaction, known to have negative environmental impacts. To address this issue, researchers have explored alternative options, such as heterogeneous catalysts derived from readily available sources like cocopeat. According to Benzon et al., cocopeat is a coconut coir that contains significant amounts of potassium (K) and chlorine (Cl). This material is an ideal natural catalyst for biodiesel production thanks to its high potassium content. By utilizing cocopeat as a substitute for traditional catalysts, we can contribute towards more sustainable and eco-friendly manufacturing processes.

Through the elemental analysis of coconut fiber ash, it has been determined that the compound composition in the form of potassium (K) amounts to 18.21%. Therefore, this research aims to evaluate the process of producing biodiesel from waste generated by fish canning factories using cocopeat ash as a natural catalyst. The goal is to achieve a high biodiesel yield while ensuring an efficient process that adheres to SNI-04-7182-2015 standards for diesel engine fuel[2]. The optimal results obtained from the biodiesel production process include a

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Published online: April 1, 2023
ISSN: 2477-541X

reaction time of 5 minutes, NaOH catalyst at 1% weight relative to Waste Cooking Oil (WCO), a molar ratio of WCO to MeOH at 1:6, an ultrasonic frequency of 20 KHz, and an output power for ultrasonic at 650 W. By reducing reaction time by 12-24 times compared to conventional methods and achieving ester content yield as high as 96.54% weight, this research ensures significant advancements in biodiesel production efficiency[3].

2. Experimental Method

2.1 Material Preparation

The research was conducted at the Renewable Energy Engineering Laboratory, Politeknik Negeri Jember. The study utilized waste oil from a fish canning factory in Muncar Banyuwangi, along with coconut (cocopeat), methanol (100%), ethanol (97%), H_2SO_4 (98%), distilled water, NaOH, and acetic acid as materials.

In this study, the process of esterification was conducted by heating a purified fish oil sample of 250 ml to a temperature range of 55-60°C. The heated oil was then combined with a methoxide solution consisting of 30% methanol and 5% H_2SO_4 catalyst (both in volume proportions to the oil). The mixture underwent stirring at a speed of 700 rpm for 60 minutes. The primary objective behind this esterification reaction was to decrease the amount of FFA present in the material by introducing a potent acid, H_2SO_4, as an effective catalyst.

When the composition of free fatty acids and oil is high, as in fish oil, simultaneous esterification and transesterification reactions through acid catalysts can potentially obtain almost complete biodiesel conversion due to the energy balance that will be reached within a few hours.

2.2 Fish Oil Pretreatment

The pretreatment of fish oil consists of two distinct phases: degumming and neutralization. Degumming is the initial step to eliminate dissolved substances from the fish oil. In this process, the fish oil is mixed with water at 60°C, comprising 20% of the total oil volume. The mixture is then agitated using a magnetic stirrer and operates at 400 rpm for 15 minutes. Following this agitation period, a separator divides the solution into three layers: oil, water, and gum. These layers are subsequently separated from one another. Afterward, hot water at 60°C was added and vigorously shaked until a neutral pH was reached. The neutralization phase aims to remove free fatty acids from the fish oil waste. This process involves adding heated NaOH (4.12 M) to the oil maintained at a temperature of 70°C while stirring for two minutes continuously. The resulting mixture is then transferred into a separatory funnel with an additional volume of 10% v/v hot water (at around 70°C). After allowing time for separation within the separatory funnel, three discernible layers form: soap, water, and refined oil. To calculate the NaOH volume required during the neutralization stages, as mentioned above, the equation would be utilized:

$$NaOH = \frac{Acid\ Number\ x\ 40\ x\ Oil\ (\ ml\ )x\ 1{,}3\ x\ \rho\ x\ 10}{56\ x\ 1000\ x\ 16{,}7} \quad (1)$$

2.3 Characterization of Fish Oil After Pretreatment

Once the oil has completed the pretreatment phase, it is crucial to evaluate its physicochemical properties just as we would with the original oil characterization. These properties include determining the content of FFA (as outlined by AOAC in 1984), utilizing the density pycnometer method (according to ASTM D1298) to assess density, measuring acid number (based on AOAC guidelines from 1984), and evaluating viscosity using the Otswald method (per ASTM 445).

2.4 Research Method Parameters

Following the washing process, various parameters are considered when determining the characteristics of biodiesel yield. These include:

1. Yield of Biodiesel

The calculation of biodiesel yield is determined using the formula below, considering the weight of Biodiesel oil after washing (BW) and Fish Oil Weight (FOW).

$$Yield(\%) = \frac{BW\ (g)}{FOW(g)}\ x\ 100\% \quad (2)$$

2. Characteristics of Biodiesel

The characterization was conducted according to SNI 7182:2015, including the parameters related to the character of biodiesel.

3. Transesterification Process

This stage is carried out if the fish oil already has more than 2% FFA. In the transesterification stage, 220 ml of the oil resulting from the separation in the esterification process is heated in a glass beaker until it reaches 55-60 Celsius, after which a mixture of 70% methanol v/v oil is prepared with a heterogeneous base catalyst (cocopeat ash). Variations in the composition of the catalyst in the reaction and methanol were mixed into the oil samples. The mixture was maintained constant at a temperature of 55-60 Celsius and stirred at 700 rpm using a magnetic stirrer. The reaction time was varied according to the

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ISSN: 2477-541X

treatment to be carried out, namely for 60 minutes and 120 minutes. After the time was reached, the mixture was allowed to stand until room temperature and separated in a separatory funnel that had been given filter paper where the product was crude biodiesel as the main product, which was located in the glass, and a brownish-white catalyst which lies on the filter paper.

This analysis used a completely randomized design with a factorial pattern of 2 factors with four repetitions in the transesterification reaction. The first factor was the catalyst composition of cocopeat ash (A1, A2, A3, where the composition of the catalyst base was heterogeneous at 3%, 5%, and 7% v/v methanol). The second factor is the transesterification reaction time (B1, B2 where 60 minutes and 120 minutes). The observed results were tested by analysis of variation Analysis of Variance (ANOVA) to test the effect of the amount of catalyst on the analytical parameters. If it had a significant effect, then the treatment of the amount of catalyst was significantly different from the amount of other catalysts tested by Duncan's Multiple Range Test with an actual level of error of 5%.

3. Results and Discussion

3.1 Results of Fish Oil Purification

The initial investigation assessed the effectiveness of a fish oil catalyst derived from waste generated by a fish canning facility. This catalyst would be utilized in the primary study to achieve optimal biodiesel production. Five samples were prepared, each containing different amounts (3% to 7%) of K_2O catalyst mixed with fish oil. The transesterification reaction was carried out for 60 minutes, yielding the most favorable results. Before processing, the waste oil was subjected to several initial tests, including free fatty acid content (AOAC, 1984), density test using the pycnometer method (ASTM D 1298), acid number (AOAC, 1984), and viscosity test using Ostwald method (ASTM 445). After the initial characteristics were carried out, the pretreatment/refining of fish oil was carried out[4].

Fish oil pretreatment here is carried out using two stages of purification, namely degumming and neutralization. In degumming, fish oil is heated to 80 degrees Celsius and mixed with water as much as 20% v/v oil to form 3 layers. This process is carried out to remove latex and dissolved substances in fish oil so as not to hinder further processing. Neutralization was done by adding NaOH to reduce FFA in fish oil[5].

The findings of the analysis conducted on the oil derived from waste produced by a fish canning facility, both prior to and after purification, are outlined below:

Table 1 illustrates the fluctuation in parameter values of fish oil before and after refinement. The initial value of free fatty acid (FFA) was 8.4%, which decreased by 6.56% to reach a final value of 1.84%. Similarly, the acid number decreased by 26.33% from its initial value of 33.72%. This decline in FFA and acid number can be attributed to adding NaOH during the neutralization process, effectively reducing the acidity levels in the fish oil.

On the other hand, there was an increase in both density and viscosity values after purification. The initial density value of 698 kg/m3 rose to a final value of 700 kg/m3, while viscosity increased from an initial reading of 35 cps to a final reading of 45 cps. During the purification process, a significant amount of water was employed, contributing to the increase in viscosity. The increase in viscosity can also be influenced by other factors such as pressure and temperature, the presence of other substances, size, molecular weight, and intermolecular forces.

In summary, refining fish oil causes some parameters to decrease (such as FFA and acid number) due to neutralization with NaOH, reducing acidity levels. Furthermore, this refinement process increases the fish oil's density and viscosity due to water usage and the influence of factors like temperature and pressure. These changes indicate that fish oil undergoes significant alterations during the refining process.

3.2 Result of Catalyst Composition

In the reaction of making biodiesel, a catalyst is needed because the reaction tends to run slowly. Catalysts lower the reaction's activation energy so that it can take place more quickly. Catalysts in different reactant phases (can be solids, liquids, or gases) are called heterogeneous catalysts[6]. There are two types of heterogeneous catalysts, namely acidic heterogeneous catalysts, and alkaline heterogeneous catalysts. Heterogeneous catalysts have several advantages, namely being easily separated from

Table 1. Characterization of fish canning factory waste oil before and after refining

Parameters	Oil Before Refining	Oil After Refining
FFA (%)	8.4	6.56
Acid Number (mg KOH/gr)	33.72	26.33
Density (kg/m^3)	698	700
Viscosity (Cps)	35	45

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ISSN: 2477-541X

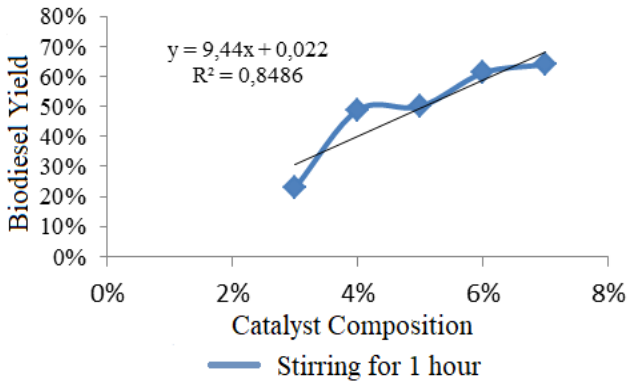

Fig. 1. Graph of preliminary research results

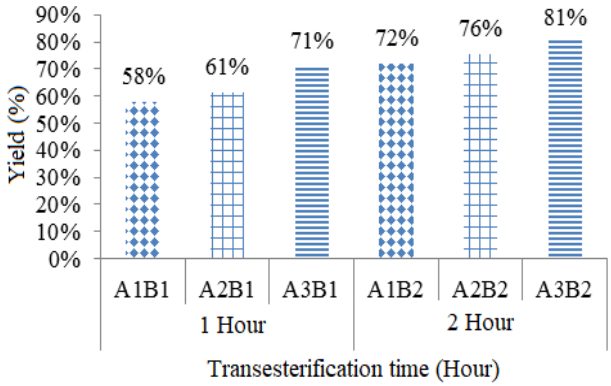

Fig. 2. Graph of catalyst composition to yield

reaction products and more resistant to free fatty acids contained in raw materials without going through a saponification reaction, making it possible to carry out transesterification and esterification reactions simultaneously with raw materials containing high levels of free fatty acids[7].

The determination of the catalyst amount is established through initial research. The catalyst employed is a solid base catalyst derived from fish oil canning residues. The results of the preliminary study will serve as a reference for the main investigation—the primary research involved using five samples of fish oil, each with a volume of 100 ml. Various composition variations were tested, including concentrations of 3%, 4%, 5%, 6%, and 7%. The optimal reaction time for transesterification was 60 minutes[4]. Below is a graph illustrating the findings from the preliminary research:

The relationship between catalyst composition and biodiesel yield is demonstrated in Figure 1, with a direct proportionality. The results show that a higher catalyst composition produces a more significant result. The highest yield was achieved when using a catalyst composition of 7%, similar to the yields obtained with a 6% catalyst composition of 61% and 64%. On the other hand, the lowest yield was observed with a 3% catalyst composition, resulting in only 23% yield.

The graph above indicates that the determination value equals 0.8486, suggesting a strong correlation between these two factors as it is close to R2 = 1. Based on these findings, catalyst compositions of either 3%, 5%, or 7%, along with transesterification times of either 60 minutes or 120 minutes, would be suitable for further experiments.

The appropriate amount of catalysts was needed for subsequent research activities, and attention has been given to selecting those compositions that produce yields closest to those obtained from preliminary investigations.

3.3 Result of Biodiesel Yield

Biodiesel production efficiency is determined by the biodiesel yield, calculated by dividing the quantity of biodiesel obtained by the initial oil used. Various factors influence the yield value in the transesterification process, including the catalyst type, reaction time, molar ratio of triglycerides to alcohol, water content, soap content, and free fatty acid content. Several samples were analyzed to determine the exact yield amount. These include A1B1 (3% catalyst composition with a 1-hour reaction time), A2B1 (5% catalyst composition with a 1-hour reaction time), A3B1 (7% catalyst composition with a 1-hour reaction time), A1B2 (3% catalyst composition with a 2-hour reaction time), A2B2 (5% catalyst composition with a 2-hour reaction time), and A3B2 (7% catalyst composition with a 2-hour reaction time)[8]. The graph below illustrates how different levels of catalyst compositions affect transesterification times.

In Figure 2, there is a correlation between the catalyst's composition and the reaction's duration. A higher catalyst composition and longer reaction time during transesterification result in a greater yield. Because when the reaction time increases, there is more mixing between the catalyst and methanol, leading to a highly homogeneous solution.

Combining the two treatments resulted in the highest yield, with a reaction time of 2 hours and a catalyst composition of 7% and 81%. In contrast, the lowest yield was achieved using the two treatments with a reaction time of 1 hour and a catalyst composition of 3%, yielding only 58%. The results demonstrate that both the reaction's duration and the catalyst's makeup impact increasing biodiesel yields. The collected data was then analyzed using variance (ANOVA) through an *F* test at 95% and 99% confidence levels.

Because the value of the long reaction time and catalyst composition significantly affects the

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Published online: April 1, 2023
ISSN: 2477-541X

ANOVA variance, further testing is needed on this interaction. The difference between the mean treatment pairs was further tested using the DMRT test (Duncan's Multiple Range Test). Based on the DMRT test, it showed that the highest yield value of biodiesel made from fish canning factory waste was obtained with a reaction time of 2 hours and a catalyst composition of 7%.

The analysis above showed no significant difference between the A1B1 treatment (1 hour reaction time, 3% catalyst composition) and A2B1 (1 hour reaction time, 5% catalyst composition). The decision was made based on the follow-up test on the A3B2 treatment, namely the long reaction time of 2 hours and the catalyst composition of 7% with an average yield of 81.02% because it significantly affected the result of biodiesel. Moreover, the longer the transesterification reaction time, the more homogeneous the solution and the more influential the catalyst that works and produces a high yield.

3.4 Result of Characterization Biodiesel

The A3B2 treatment was conducted to determine the biodiesel's characteristics, with a reaction time of 2 hours and a catalyst composition of 7%. The biodiesel quality assessment involves various parameters examination, including density, viscosity, flash point, acid number, water content, iodine number, saponification number, cetane number, and heating value. The results of these tests will now be presented and discussed, as summarized in Table 2.

The measure of the quantity of a substance within a given space is known as density. When density is higher, the mass contained in each unit of volume is also higher. This density value directly correlates with the calorific value and power output of a diesel engine per unit of fuel volume. Specifically, biodiesel derived from waste produced by fish canning factories has been found to have a density value of 876.3 kg/m3. The increase in density can be attributed to the presence and influence of residual catalysts that were not completely removed during the washing process, leading to an overall denser composition[8].

Table 2. Comparison of biodiesel from fish canning factory waste and biodiesel based on SNI 04-7182-2015

Parameters	Biodiesel Waste Fish Canning	Biodiesel Standard
Density40°C(kg/m³)	876.3	840-890
Kinematic viscosity 40°C(cps)	11.24	2.3-6.0
Cetane number	42.75	min.51
Flash point (°C)	160	min. 100
Water and sediment (%-vol)	0.06	max. 0.05
Acid Number (mg-KOH/g)	0.59	max. 0.5
Iodine Number (%-mass)	16.36	max. 115
Calorific Value (Mj/Kg)	47.47	min. 40

Viscosity is a characteristic that determines how resistant a fluid is to flow. When the fluid viscosity is high, the fuel is more difficult to be introduced into a diesel engine combustion chamber. The viscosity value of biodiesel derived from waste produced by fish canning factories does not meet the standards set by SNI 04-7182-2015, which states that it should be between 2.3 and 6.0 cSt. In this case, the measured viscosity value is 11.2431 cSt, exceeding the specified range. When biodiesel has a higher viscosity than required, it leads to incomplete combustion reactions, increases wear and tear on engine components, and produces more exhaust emissions. On another note, flash point refers to the lowest temperature at which fuel vaporizes and ignites when exposed to air and an ignition source like fire. Based on research findings, the flash point value of biodiesel obtained from waste generated by fish canning factories measures 160°C. This particular flash point value meets or exceeds SNI 04-7182-2015's minimum requirement of 100°C for safe handling and storage purposes[8].

The acid number serves as a significant indicator when assessing biodiesel. A lower acid number indicates reduced free fatty acids in the biodiesel. The results show that the biodiesel derived from fish canning factory waste fails to meet the SNI 04-7182-2015 standard, which sets a maximum limit of 0.5 mg-KOH/g, with an actual value of 0.59 mg-KOH/g. The high content of fatty acids in biodiesel leads to soot formation within engine injectors and contributes to engine corrosion [9]. To determine the acid value of the extracted fish oil, Demirbas et al. described a method based on titration using phenolphthalein as an indicator and KOH solution in ethanol to detect the endpoint[11, 12]. This method involves diluting with an ethanol-diethyl ether mixed solvent before titration.

As per the specifications outlined in SNI 04-7182-2015, biodiesel should not contain more than 0.05% of water. However, fish oil biodiesel has a water content of 0.06%, suggesting it fails to meet the established standards. This disparity can be attributed to residual water during the washing process of biodiesel derived from fish canning factory waste.

The iodine number indicates the level of unsaturation in the constituent compounds found in biodiesel. A higher number signifies a more significant presence of unsaturated compounds, which leads to increased iodine production costs and decreased CO2 exhaust gas emissions[13].

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According to industry standards, the maximum allowable iodine cost is 115% by mass (gl_2/100g), while fish oil biodiesel has an iodine value of 16.36 g/100g. The test results demonstrate that the iodine value of biodiesel derived from waste produced by fish canning factories meets the requirements set for biodiesel production.

The saponification number serves as a means to determine the cetane number value in biodiesel. It is worth noting that the SNI standard does not provide an explicit listing for this saponification number. In the case of biodiesel derived from waste from fish canning factories, it was found to have a notably high saponification number of 41.79 mg/g. As the transesterification reaction progresses for a longer duration, there is an observable decrease in both fatty acids and saponification numbers [2]. A higher cetane number results in easier combustion within the cylinder and avoids accumulation at lower temperatures [9]. Considering this approach, it has been determined that fish canning factory waste has a cetane index value of 42.75, indicating non-compliance with the SNI 04-7182-2015 biodiesel standard, which sets a maximum limit at 51. The calorific value or heating value refers to the amount of heat energy released per unit mass of fuel [11].

Biodiesel has the ability to burn quickly in the combustion cylinder due to its high cetane number. It also doesn't accumulate at lower temperatures [8]. The cetane index determines the value of biodiesel's cetane number. According to [13], the cetane number is typically 2% higher than the cetane index. In this study, an approximation method was used to determine the cetane index, which resulted in a value of 42.75. The result shows that the oil yield's cetane number was unable to meet the SNI 04-7182-2015 biodiesel standard, which requires a minimum value of 51.

According to USA No. 2 Distillate (Diesel) standard, the maximum limit for the calorific value is the HHV value is 40 MJ/Kg. The calorific value was calculated using an equation proposed by Demirbas utilizing saponification value and iodine number [8]. From the results of these calculations, the calorific value of the biodiesel from the fish canning factory waste is 47.47 MJ/kg, so the heating value of biodiesel can be categorized as compliant with the standard.

Analysis of variance (ANOVA), in which four replications showed that the influence of the magnitude of the composition and the length of time of the transesterification reaction, the interaction of the two treatments, had no significant (non-significant) effect on the increase in the value of the biodiesel acid number so it did not require further testing. The development of biodiesel based on oil is an alternative because it benefits both the environment and renewable natural resources [14]. According to a study [15], fish waste in biodiesel production faces challenges related to high levels of free fatty acids (FFAs), water content, and other impurities.

4. Conclusions

Based on the research findings, it was determined that the purification process for fish canning factory waste consisted of two main stages: degumming and neutralization. The purification process increased density and viscosity values while simultaneously decreasing FFA value and acid number. When using cocopeat ash catalyst as a biodiesel catalyst, the A3B2 treatment with a catalyst composition of 7% and 2 hours yielded the highest percentage at 81%. Numerous tests were performed on biodiesel standards derived from fish canning factory waste to ensure compliance with SNI-04-7182-2015. These tests revealed that the biodiesel had a density value of 876.3 kg/m3, a flash point of 160°C, an iodine number of 16.36 g/100g, and a heat value of 47.47 MJ/kg.

During this study, it was discovered that purifying fish canning factory waste involves two distinct steps: degumming and neutralization. By undergoing this purification process, specific parameters such as density and viscosity increased while others like FFA value and acid number decreased accordingly. Employing cocopeat ash catalyst as the biodiesel agent led to optimum results with an impressive yield rate reaching up to 81%, specifically regarding treatment A3B2, which used a mixture containing approximately seven percent biomass-derived fuel and two hours' duration.

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