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Utilization of Crude Glycerol Waste from Biodiesel Production as an Additive to Improve the Quality of Tea Dreg Biopellet

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ABSTRACT

Biodiesel production using the transesterification method generates a large amount of crude glycerol as by-products. Crude glycerol waste can be reused and utilized as an additive in the formulation of biopellet. Tea dregs are a waste produced by the beverages industry that can be used as green fuel in the form of biopellet as an environmentally friendly energy source. This study aims to analyze and characterize biopellet from tea dregs with crude glycerol as an additive to increase the quality. The biopellet formulation contains six levels of crude glycerol composition percentage: 0% (as control), 5%, 10%, 15%, 20%, and 25%. The parameters for the quality assessment of biopellet refer to the SNI 8675:2018 standard. The best formulation of biopellet was obtained in the 10% treatment with properties of density value of 0.93 g/cm3, pellet durability of 98.09%, moisture content of 8.10%, volatile matter content of 73.37%, ash content of 6.08% and calorific value of 16.38 MJ/kg. The addition of up to 10% crude glycerol as an additive has been shown to improve the quality of tea dregs biopellet.

1. INTRODUCTION

The biodiesel reaction process will produce a by-product of crude glycerol, which still contains unreacted oil, excess methanol, and a catalyst. Glycerol is a major component of all fats and oils with the chemical formula of $C_3H_8O_3$. Crude glycerol from biodiesel waste also has flammability properties with a high calorific value as a fuel additive composition (Ilham *et al*., 2016). With increased biodiesel production in various countries, including Indonesia, the supply of biodiesel waste will grow, while the global demand for glycerol remains limited and needs to be utilized (Afrozi, 2018). The use of crude glycerol as a material for increasing the energy value of combustion is an alternative to utilizing biodiesel waste without purification.

Tea dregs produced by the beverage industry from extraction were typically discarded or used as organic fertilizer. According to the Director General of Plantations, tea production in Indonesia in 2017 is estimated to reach 146 thousand tonnes (Hartati & Kusumaningrum, 2019). Tea dregs can be used as an alternative fuel in the form of biopellets for environmentally friendly alternative energy. Tea dregs can potentially be used as a biopellet because it contains high crude fiber, such as 34.16% cellulose and 29 % lignin (Ariwidyanata *et al*., 2019).

Presidential Regulation of Indonesia concerning national energy policy encourages efforts to develop renewable energy sources as an alternative to fuel oil, natural gas, and coal. One of the steps that can be taken is to utilize biomass energy sources. Biopellets are solid biomass-based fuels in the form of tubes or pellets. Biopellets produced by pelletizing bulky biomass materials are widely used in combustion applications such as sawdust, agricultural residues, biomass waste, and can be used as alternative energy. Biopellets are formed by pressing biomass in a cylindrical shape using the densification method and have a length of 6 - 25 mm with a diameter of 3 - 12 mm (Rusdianto *et al*., 2014).

The utilization of crude glycerol as an additive to increase the calorific value and quality of combustion is an alternative to utilizing biodiesel waste. Various types of vegetable oil and glycerol as additives up to 7.5% in biopellet formulations have been carried out with positive responses to increasing energy content (Mišljenović *et al*., 2015). As a by-product, biodiesel waste consists of crude glycerol, which still contains oil, methanol, catalyst, and other types of salt. Using crude glycerol directly in biopellet production helps solve the problem of biodiesel by-product disposal while lowering the cost of biodiesel production (Demir *et al*., 2016).

This study aims to analyze and characterize the ratio composition of tea dreg biopellets with crude glycerol as an additive to increase quality and obtain optimal formulations. Several aspects for assessing biopellet quality as a renewable energy fuel, such as density, durability, moisture content, calorific value, ash content, and volatile matter, refer to the SNI 8675:2018 standard (biomass pellet as a fuel). The results were expected to provide information on the characteristics of various formulations of tea dregs biopellet with the addition of crude glycerol as an environmentally friendly alternative fuel energy.

2. MATERIALS AND METHODS

2.1. Material and Tools

The materials used in this study were tea dregs, crude glycerol, and starch for adhesives. The raw material for tea dregs was obtained from the beverage industry in Sukabumi, West Java. Crude glycerol was a by-product of biodiesel production at the Renewable Energy Laboratory, Department of Mechanical and Biosystem Engineering, IPB University. The tools used were a fixed die pelletizer model TET-01 (3-phase 10 HP motor) , stirrer, measuring cup, drying oven, furnace, biopellet stove, desiccator, bomb calorimeter, solar dryer, thermocouple type K, data recorder Graptech GL840, digital balance, and standard sieve.

2.2. Experimental Design

The research followed by several steps, including the material composition formula of tea dregs and crude glycerol, pelletizing (densification), biopellet drying, and proximate -ultimate analysis. The research uses one factor of composition formula, 6 levels of treatment (A0 as control, A1, A2, A3, A4, A5), and 3 repetitions of a sample. The data analysis was conducted by the statistical method of one way ANOVA that observed variance data with a Completely Randomized Design (CRD) non-factorial model

(Selvamuthu & Das, 2018). The level of significance was chosen as 0.05 (p value). When the results had a significantly different effect, then continue with Duncan's Multiple Range test (DMRT) test to show the relationship between all treatment levels. Statistical calculations were performed using SPSS Ver.26 software.

2.2.1 Biopellet Formulation

The initial proximate analysis shows the tea dregs material used has a moisture content of 9.28 - 10.14% (wet basis), average density of 0.32 g/cm^3, average particle size of 40 mesh. The treatment of tea dregs was by mixing with water in a ratio of 2:1. The addition of water to an optimum moisture content of up to 40% was necessary for pelletizing process depending on the characteristic of the machine. The adhesive was a 1:10 mixture of starch and water that was heated until it thickened. The crude glycerol used has a density of 1.08 g/cm^3, a moisture content of 10 - 15%, and a dark yellow color. The calorific value of the crude glycerol from transesterified biodiesel was 25.18 MJ/kg (Litwiniak & Radomiak, 2019). The ingredients were combined into a dough with 10% optimal adhesive (Lamanda *et al*., 2015). However, the lignin content in the tea dregs was relatively low and the temperature in the pelletizing process was not high. Consequently, the lignin did not function optimally as a natural adhesive for biopellet. The formulation level in percent of weight per weight of the biopellet formulation is shown in Table 1.

Table 1. The composition level of the biopellet tea dregs and crude glycerol formulation

Level treatment	Formulation (% weight/weight)		
	Tea dregs (%)	Crude glycerol (%)	Adhesive (%)
A0 (control)	90	0	10
A1	85	5	10
A2	80	10	10
A3	75	15	10
A4	70	20	10
A5	65	25	10

2.2.2 Pelletizing of Biopellet

Biopellet pressing uses a fixed die rotary pelletizer machine, as shown in Figure 1. The pelletizer was powered by a 3-phase 10 HP electric motor with an inverter frequency of 25 Hz. The motor rotation average was 737 rpm, and the die rotation average was 74 rpm, with a biopellet capacity of 18.5 kg/hour. The diameter of the compressed hole was 11 - 12 mm. The pelletizer uses a roller wheel and dies component that rotates fixedly to compress the raw material towards the molding hole inside the die. Furthermore, the biopellet that came out from the pelletizer was removed from the excess water content through the solar drying during 1 - 2 day with temperature range from 32.94 - 33.87 ^{o}C.

2.2.3 Analysis Method

The characteristic and proximate analysis of tea dregs and crude glycerol biopellet consist of density (SNI 8021:2014), moisture content (AOAC 2005), volatile matter (ASTM D-5832:1998), ash content (ASTM E1755-01), calorific value (SNI 01-6235), and durability (DIN EN 14961-2). The results will be analyzed to determine the best biopellet formulation.

Figure 1. Fixed dies rotary pelletizer machine model TET-01

2.2.4 Combustion Rate Test

Combustion rate testing was carried out using a biomass stove. A sum of biopellet was weighed and then placed in the stove. The initial ignition of the biopellet uses a lighter. The combustion air speed used was 3.05 m/s with a fan blower. The qualitative parameters observed were ignition, smoke, ash content as waste, and combustion rate using equation (1).

$$Combustion\ rate = \frac{mass\ of\ burning\ biopellet\ (g)}{burning\ time\ (min)} \quad (1)$$

3. RESULTS AND DISCUSSION

3.1. Characterization and Properties of Biopellet

The biopellet surface with a low level of crude glycerol content has a smoother texture appearance than those with a high level. According to Gonzalez *et al*., (2020), the natural aggregation of biopellet derived from lignin can produce better mechanical interlocking. Meanwhile, biopellet with a high level of crude glycerol has a slightly rough surface, porous, and cracks on the surface. The visual appearance of biopellet can be seen in Figure 2. The biopellet with rough and broken surfaces was caused by the high-water content in the raw dough material, which affected by a high level of crude glycerol, so the densification process was not optimal. The lubricating properties of crude glycerol caused the pressure during the densification process to be imperfect (Gonzalez *et al*., 2020).

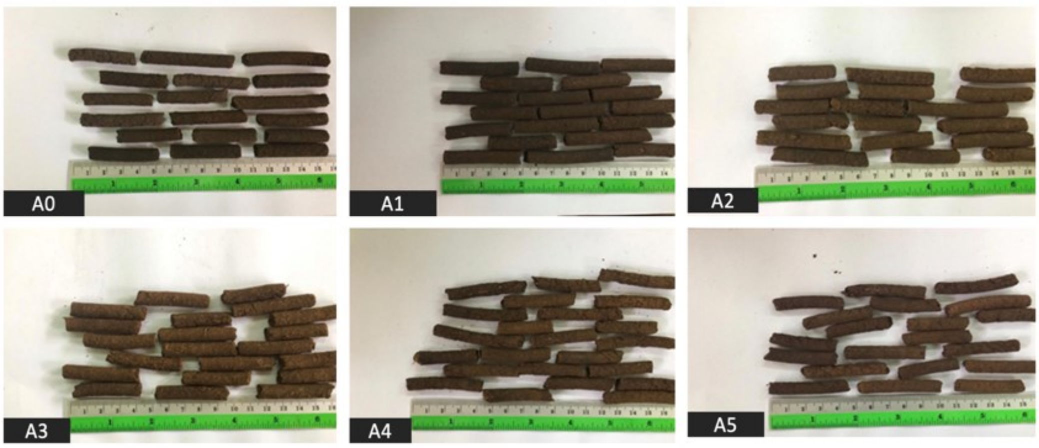

Figure 2. The visual appearance of tea dreg biopellets formulation

The physical appearance of biopellets is shown in Table 2. Referring to SNI 8675:2018, biopellet have a minimum standard density of 0.6 g/cm^3, maximum moisture content of 12%, maximum ash content of 5%, maximum volatile matter content of 80%, minimum fixed carbon of 14% and a minimum calorific value of 16.5 MJ/kg.

Table 2. Physical appearance of tea dregs biopellets

Composition level	Color	Physical appearance
A0 (control)	Deep black	Straight and smooth
A1	Black	Straight and smooth
A2	Faded black	Long and fine
A3	Faded black	Slightly rough
A4	Dark brown	Rough and brittle
A5	Dark brown	Rough and brittle

3.1.1 Density

The density of the tea dregs biopellet obtained was 0.85 - 1.13 g/cm^3, as shown in Figure 3. The lowest density was obtained from the composition level of A1, in comparison the composition level of A5 produced the highest density biopellet. According to the SNI, the density of the biomass pellet must be greater than 0.6 g/cm^3. All treatment level shows that tea dregs biopellet met the standards. The results of ANOVA test showed a p-value < 0.05, which indicated a significant difference in crude glycerol addition to the density of the tea dregs biopellet. The results of Duncan's test showed that the densities in the A0 treatment were significantly different from those of A1 and A2. However, the A0 treatment was not significantly different from those of A1, A2, and A3. The specific gravity, particle size, cellulose and lignin content were dominant factors that can affect the density of biopellet (Hendra, 2012). High crude glycerol levels in biopellet additive can increase the density and cause the biomass to expand. High quantities of glycerol can make the pellets denser, which causes the surface of the biopellet to enlarge. It is caused by the high water level of 10% and the glycerol concentration, which can bind the tea dregs particles. According to Saptoadi (2008), the smaller size and bond of biomass particles, the density will increase because it reduces the presence of voids and thus reduces water absorption.

3.1.2 Durability

Durability was an important factor in transporting and storing biopellet for the physical quality from external impact and pressure (Wistara *et al*., 2020). The high durability of the biopellet indicates that the quality was improving. According to the research findings, the durability of tea dregs biopellet ranged from 96.04% - 98.09% (Figure 3). The DIN EN 14961-2 Standards (2012) biopellet durability value must be greater than 96.5%. The lowest durability value was obtained from the A4 formulation, while the highest was obtained from the A2 formulation. The results of ANOVA test showed a p-value < 0.05, which indicated a significant difference in crude glycerol addition to the durability. The results of Duncan's test showed that the durability in the A0 treatment were significantly different from those of A1, A3, A4, and A5. However, the A0 treatment was not significantly different from the A2. Biopellet durability was influenced by adhesive, pressure, and pelletizing density. The pressure used in pelletizing produced a high density resulting in a high resistance value of the biopellet.

The use of glycerol additives can increase density, which also impacts increasing durability. It is related to the characteristic of glycerol, which can bind the biomass particles better. The durability test results were consistent with the findings of Gonzalez *et al.* (2020), who found no significant difference between biopellet with biodiesel waste as binder.

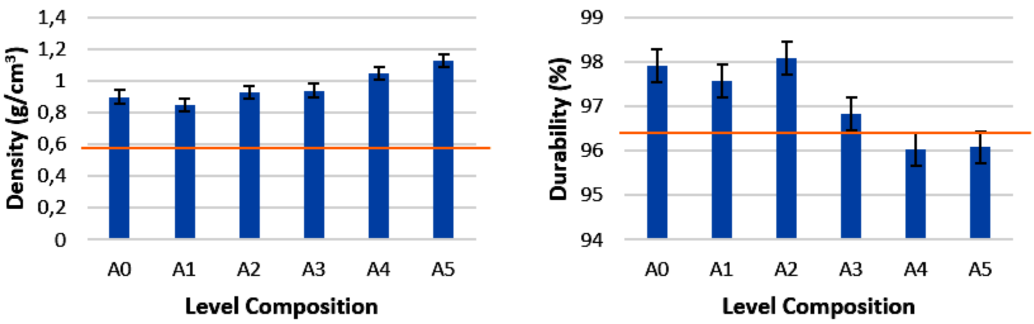

Figure 3. The effect of level composition of tea dregs and crude glycerol to the density and durability of biopellet (▬ SNI/ DEN-EN)

3.1.3 Moisture Content

The moisture content in the raw material of biomass can facilitate the formation of biopellet during the pelletizing process, but excess moisture content can reduce the dimensional quality (Prabawa & Miyono, 2018). According to the SNI, the moisture content of biomass pellets must be less than 12%. Meanwhile, the moisture content obtained in this study ranged from 7.56 - 12.16% (Figure 4). The lowest moisture content was obtained from the formulation with A1, while the highest was obtained from the formulation with A4. The results of ANOVA test showed a p-value < 0.05, which indicated a significant difference in crude glycerol addition to the moisture content of biopellet. The results of Duncan's test showed that the A0 treatment were significantly different from those of A1, A2, A3, A4, and A5. According to Litwiniak & Radomiak (2018) stated that using high amounts of biodiesel waste can increase the moisture content in biopellet because biodiesel waste contains around 10% water and has the hygroscopic characteristic of glycerin. Reducing the bound water on the biomass material can also be carried out through torrefaction treatment, decreasing the water content by up to 57.6% (Yulianto *et al*., 2020). Bantacut *et al*., (2016) added that drying temperature affects the moisture content of the biopellet. Moisture content that complies with standards was important to prevent pellet from swelling due to humidity during storage and shipping. High moisture content can make pellets susceptible to mould and microorganisms.

3.1.4 Volatile Matters

Volatile matter is an evaporated substance due to the decomposition of compounds still present in charcoal (Hendra & Pari, 2000). The volatile matter of biopellet was in the range of 77.05 - 79.74%. According to SNI standards, the maximum value of volatile matter content was 80%. The lowest volatile matter content was obtained with an A0 formulation, while the highest was obtained with an A5 formulation. All biopellet formulations level has met these requirements (Figure 4). The results of ANOVA test showed a p-value < 0.05, which indicated a significant difference in crude glycerol addition to the volatile mater of biopellet. The results of Duncan's test showed that the volatile maters in the A0 treatment were significantly different from those of A1, A2, A3, A4, and A5. However, the A2 treatment was not significantly different from the A3 and A5.

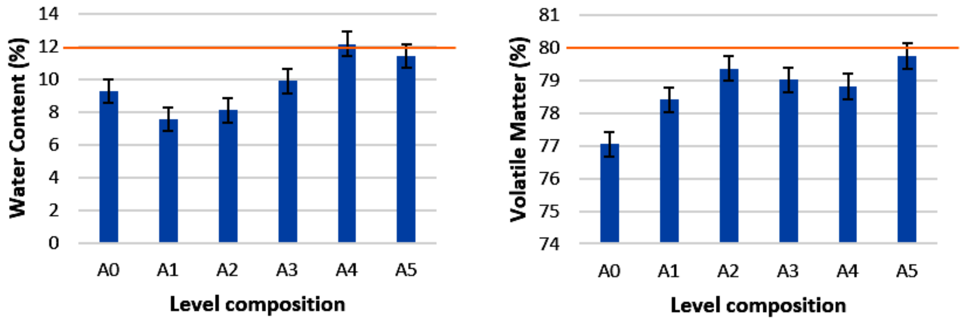

Figure 4. The effect of level composition of tea dregs and crude glycerol to the moisture content and volatile matter of biopellet (— SNI)

The phenomenon was presumably because the higher level of crude glycerol caused its volatile matter to evaporate. Therefore, adding high levels of biodiesel waste can increase volatile matter and fuel reactivity (Bartocci *et al*., 2018). The crude glycerol derived from biodiesel waste contains component of oils with the chemical formula of $C_3H_8O_3$, excess metanol (CH_3OH) and alkaline catalyst of NaOH. These component also have a volatile and flammable characteristics in burning process with a high calorific value that affect the volatile matter of pellet (Ilham *et al*., 2016). According to Huseini *et al*. (2015), volatile matter consists of flammable gases, including H_2, CO, methane, and easily condensable vapors. Hansen *et al*. (2009) states that the value of the volatile matter determines the burning time, burning rate, amount of smoke produced, and the efficiency of biopellet combustion. The level of volatile matter functions in the flame's stability and combustion.

3.1.5 Ash Content

Ash is a residue of minerals that have lost their carbon components. The main component of the ash is silica which has an unfavorable effect on the heating value and quality of burning pellets (Hendra, 2012). The ash content of the material directly proportional to silica content; the higher the silica content, the more ash is produced during the combustion process. Huseini *et al*. (2018) added that the constituents of ash consist of compounds of silicon, aluminum, iron, calcium, and small amounts of Na, Ti, K, Mg, and Mn, in the form of silicates, oxides, sulfates, and phosphates. Based on the proximate analysis, the ash content of tea dregs and crude glycerol biopellet obtained ranged from 4.25 - 7.75%. The SNI standard requires a maximum biopellet ash content of 5%. The lowest ash content was obtained from the formulation with an A5 formulation, while the highest ash content was obtained from the formulation with an A0 formulation. The results of ANOVA test showed a $p\text{-value} < 0.05$, which indicated a significant difference in crude glycerol addition to the ash content of biopellet. The results of Duncan's test showed that the A0 treatment were significantly different from those of A2, A3, A4, and A5. However, the A2 treatment was not significantly different from the A3. Based on the results (Figure 5), the increase and decrease in the resulting ash content were not significantly different between composition levels, the same adhesive content influenced this in each treatment. According to Nurmalasari & Afiah (2017), the higher the adhesive content, the higher ash content produced. The raw material component also influences the high or low ash content. According to Thompson & He (2006), many impurities substances are contained in biodiesel waste.

3.1.6 Calorific Value

The calorific value is the heat or energy released from fuel combustion and produces ash, CO_2, SO_2, nitrogen, and vapor (Patabang, 2011). The high calorific value of biopellet can minimize fuel requirement and make combustion more efficient (Jamilatun, 2012). The ignition speed of the biopellet is affected by the carbon content in the biopellets; the higher bound of carbon causes the heating value to increase so that the ignition speed of the biopellet tends to be faster (Onchieku *et al*., 2012). Based on the analysis, the calorific value was obtained between 15.37 - 16.38 MJ/kg (Figure 5). The SNI standard required a minimum calorific value of biomass pellet of 16.50 MJ/kg. The lowest calorific value was obtained from the formulation with an A5 level, while the highest was obtained from the formulation with a treatment A2 level of 10 % crude glycerol addition. The results of ANOVA test showed a p-value < 0.05, which indicated a significant difference in crude glycerol composition to the calorific value of biopellet. The results of Duncan's test showed that the A0 treatment were significantly different from those of A1 and A2. However, the A0 treatment was not significantly different from the A3, A4, and A5. From the treatment, crude glycerol addition can increase the calorific value at certain level on biopellet. Litwiniak & Radomiak (2018) added that the optimal percentage of adding biodiesel waste to biomass raw materials is around 20% or lower. Adding biodiesel waste at high levels can reduce the calorific value due to the presence of water and non-flammable substance contained in biodiesel waste. According to Afriani *et al*. (2017), the calorific value can be affected by the moisture content; the higher the water content in the biopellet, the lower the calorific value. This occurs because the heat energy produced by the biopellet is used to evaporate the water content before being used in the combustion process. Higher adhesive on biopellet content tends to lower the calorific value (Rahmadhani *et al*., 2017). Furthermore, the lignin content in raw materials also affects the heating value.

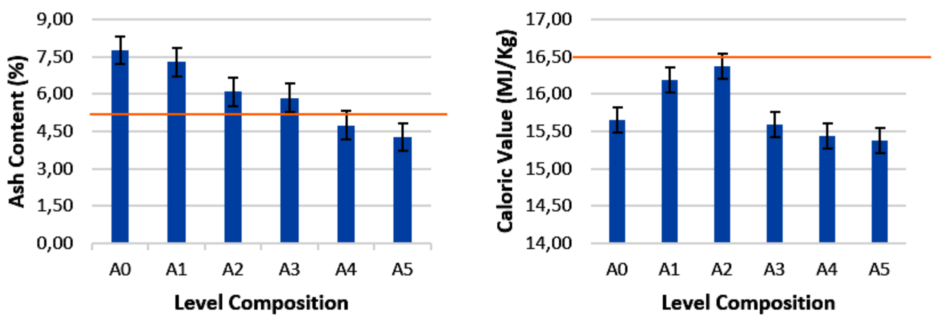

Figure 5. The effect of level composition of tea dregs and crude glycerol to the ash content and calorific value of biopellet (—— SNI)

3.1.7 Combustion Rate

The combustion rate of biopellet is the burning process that produces ashes, gases, and heat energy and counting by the mass amount of biopellet that burned in a unit of time. Based on the data, the combustion rate of biopellet was obtained from 28.70 - 35.93 g/minute and the average heat temperature was obtained from 557.25 - 713.75 ^{o}C (Figure 6). The highest combustion rate was obtained from the biopellet formulation of A5, while the lowest was obtained from the A2 level. The combustion rate of biopellet will affect fuel consumption speed. Formulation A2 has the best results based on the parameters of ease of ignition, and the temperature was relatively high and constant, with little ash, smoke, and long combustion time. The results of the qualitative testing of the combustion rate can be seen in Table 3.

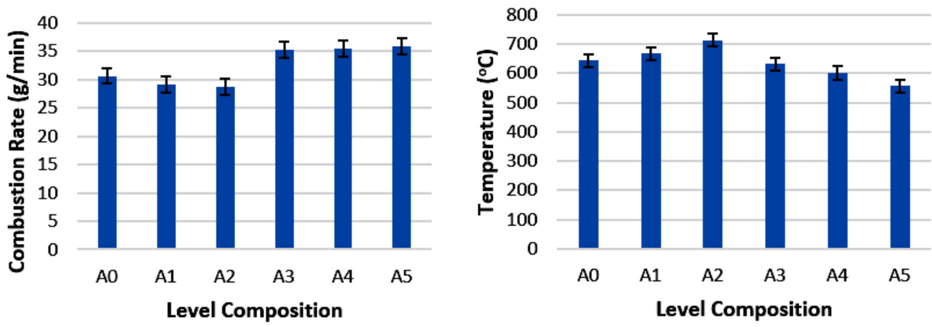

Figure 6. The effect of level composition of tea dregs and crude glycerol to the combustion rate and heat combustion temperature of biopellet

According to Sudiro & Sugiyanto (2014), the burning of biopellet is divided into three stages, the first is heating with a slow reduction of raw materials, the second is devolatilization which is indicated by a rapid decrease in the mass of raw material, and the third is burning carbon content. The difference in combustion rate between all biopellet formulations was also influenced by water content. The high-water content in the A3 -A5 treatment has come from the crude glycerol. High water content causes the combustion process to be inefficient because of the low heat produced and a large amount of smoke (Mahdie *et al*., 2018). The density of biopellet also affects the combustion rate. The high density of biopellet causes reduced air voids, and the combustion rate is slow (Purnomo *et al*., 2015).

Table 3. Qualitative performance of combustion of tea dregs and crude glycerol biopellet

Formulation level	Ignition convenience	Smoke	Ash
A0	fair	fair	fair
A1	fair	fair	poor
A2	excellent	good	fair
A3	good	bad	fair
A4	bad	poor	good
A5	poor	poor	good

Note: quantitative scale : (1) bad, (2) poor, (3) fair, (4) good, (5) excellent

3.2. Effect of Crude Glycerol Additives on Biopellet Quality

Biodiesel waste has proven to influence the quality of biopellet depend on control (A0). Adding crude glycerol up to 10% positively increased the biopellet density by 3.33%, the durability by 0.18%, volatile matter by 3.01%, calorific value by 4.62%, and decreased ash content by 6.10%. The water content of the biopellet decreased when crude glycerol was added by 5% - 10%. Moreover, the water content of the biopellet increased when the crude glycerol was added at levels above 10%. Adding crude glycerol below 10% positively decreases the biopellet water content by up to 12.75%.

The calorific value of biopellet increased when the crude glycerol was added at 5% and 10% but decreased when the crude glycerol was added at 15% - 25%. According to Bala -Litwiniak (2020) which states that adding 5% biodiesel waste can give a slight increase in calorific value and other properties. Generally, formulation A2 which adds 10% of crude glycerol as an additive to tea dregs biopellet, provides the most optimal quality improvement.

4. CONCLUSION

The tea dregs and crude glycerol biopellet had a density of 0.85 - 1.13 g/cm^3, a durability of 96.04 - 98.09%, a moisture content of 7.56 - 12.16%, a volatile matter content of 77 .05 - 79.74%, ash content of 4.25 - 7.75%, calorific value of 15.37 - 16.38 MJ/kg, and combustion rate of 28.70 - 35.93 g/min with average heat temperature of 557.25 - 713.75 ^{o}C. Based on proximate analysis data and SNI 8675:2018 standards, biopellet with the best formulation obtained was A2 level treatment with the addition of 10% crude glycerol, which had a positive effect on various properties. The addition of biodiesel waste or crude glycerol with high levels up to 15-25% can reduce the quality of biopellet.

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