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EFFECT OF GRAIN SIZE, TEMPERATURE AND CATALYST AMOUNT ON PYROLYSIS PRODUCTS OF *SPIRULINA PLATENSIS* RESIDUE (SPR)

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ABSTRACT

Spirulina platensis microalgae is one of the feedstocks used in the production of the third generation of biofuel. The extraction of its lipid for biodiesel leaves behind a residue, which can be treated by pyrolysis to create certain other value-added products. This paper discusses the effects of *Spirulina platensis* residue (SPR) with respect to grain size (0.105, 0.149 and 0.177 mm), temperature (300 to 600°C) and amount of catalyst (0, 10, 20 and 40 wt.%) on the characteristics of products (bio-oil, water phase, char and gas) obtained from pyrolysis in a fixed-bed reactor. The results of the study show that the higher the pyrolysis temperature, the higher the conversion. For the bio-oil product, the optimum temperature is 500°C, which produces a peak yield of 35.99 wt.%. The larger the grain size, the lower the bio-oil yield, gas water and gas, for all of the tested temperatures (300–600°C). The amount of catalyst and the pyrolysis temperature greatly influence the quality of bio-oil products, grouping them into the fractions of LPG (C ≤ 4), gasoline (C5–C11), biodiesel (C12–C18) and heavy naphtha (C ≥ 19). The tendency for LPG-Gasoline formation at optimum conditions, considering the use of a 10 wt.% catalyst at a temperature of 400–500°C, was reported.

Keywords: Alumina silica; Bio-oil pyrolysis; Fixed-bed; *Spirulina platensis* residue

1. INTRODUCTION

The massive exploitation of non-renewable natural resources may pose a threat to lives in the future arising from an increase in associated health issues and the greater risk of environmental damage that stems from the lower energy reserves and greater release of emissions during their processing (Anggorowati et al., 2018; Setyawan et al., 2018). There thus an urgent need to replace non-renewable energy sources with renewable ones. Biomass is one of the most potent renewable energy sources, with its development having now led to the third generation of biofuel (Purwanto et al., 2015; Supramono et al., 2015; Jamilatun et al., 2017a; Kusrini et al., 2018a).

The first-generation biofuels (*Fatty Acid Methyl Esters* (FAME) or biodiesel, corn ethanol and sugar alcohol) were produced from lignocellulosic sources in the form of vegetable oils, corn, sugar and others. Although safer from an environmental perspective, a negative impact on food security may have arisen due to competition with food for consumption (Naik et al., 2010; Maity

et al., 2014). Different from their predecessors, second-generation biofuels (hydrotreating oil, bio-oil, ethanol from lignocellulose, butanol and alcohol mixtures) were developed to address the aforementioned issue on biofuel production by using non-food lignocellulosic sources such as agricultural residues, forests, grasses, aquatic biomass, etc. Despite not competing for resources with food production as they are derived from renewable resources, and with lower costs of production, the raw material resources used in the first and second generations did require extensive land; hence, biofuel production per area is relatively low (Pradana et al., 2017a; Sudibyo et al., 2017). To that end, microalgae, which requires less land to produce, offers the potential to be developed as a resource for use in the third generation of biofuels (Jamilatun et al., 2017b; Pradana et al., 2017b). Microalgae can be converted into biofuels through either a thermal or biological process. Pyrolysis technology can be utilized to process microalgae into third-generation biofuels (Yuliansyah et al., 2015). The initial process begins with the extraction of *Spirulina platensis* microalgae to leave behind a solid residue known as *Spirulina platensis* residue (SPR). The process of pyrolysis can then be applied to convert this low-value material into highly valuable fuel products and chemicals.

Microalgae pyrolysis is influenced by many parameters, such as the biomass type, temperature, heating rate, residence time, size and shape, catalyst, etc., as partly reported by Zheng et al. (2018). Temperature is the most significant of these operating parameters and has a tremendous effect on the product composition; indeed, pyrolysis is generally carried out at a temperature range of 400–600°C (Kusrini et al., 2018b). Within this temperature range, the liquid phase is mostly produced, which accounts for around 60–70 of the wt.%. However, further increasing the temperature beyond its optimum will lead to secondary cracking, whereby tar from the primary cracking product will be converted into gas and char. The effect of secondary cracking is a decline in bio-oil products and an increase in gas products (Dickerson & Soria, 2013).

In a fast pyrolysis process, it is generally assumed that the increase in grain size produces a greater temperature gradient in the particles. At any given time, the core temperature is lower than that of the surface of the biomass particles. This can lead to an increase in charcoal, while the amounts of gas and bio-oil decrease. Small particles have an adequate surface area via which to interact with pyrolysis media to form volatile products, thus enabling them to leave the biomass matrix without experiencing any secondary reactions. However, in the case of slow pyrolysis, particle size shows a less significant effect on product yield (Yang et al., 2019).

The presence of an additional catalyst significantly affects the product yield in catalytic pyrolysis. The gas yield increases and charcoal is initially produced, the amount of which is subsequently increased with the addition of the catalyst. In addition, charcoal yield reaches a minimum of 9 wt.% when the mass ratio of the raw material/catalyst is 1:3. The addition of further catalyst increases the catalytic cracking rate, thereby aiding in the formation of gas compounds. Furthermore, the oil yield decreases as the loading of the catalyst increases. This phenomenon occurs due to secondary steam cracks (Qi et al., 2018).

This paper aims to characterize the SPR pyrolysis products (bio-oil, water phase, char and gas) in fixed-bed reactors with various temperatures, SPR grain sizes and number of catalysts used. The ultimate, proximate and calorific values of the raw materials in the form of SPR are analyzed, while the components of the alumina-silica catalyst are analyzed by SEM-EDX (Scanning Electron Microscopy Energy-Dispersive X-Ray Analysis). The pyrolysis products in the form of bio-oil will be analyzed with GC-MS (Gas Chromatography-Mass Spectrometry), with the components then grouped into the fractions of LPG (C ≤ 4), gasoline (C5-C11), biodiesel (C12-C18) and heavy naphtha (C ≥ 19).

2. METHODS

2.1. Materials

Spirulina platensis residue (SPR) was obtained from solid residues of *Spirulina platensis* extraction. Dry SPR was sieved and separated by grain size, i.e. 0.105, 0.149 and 0.177 mm, corresponding to the sizes required to pass through a 120, 100 and 80 mesh, respectively. SPR has certain characteristics relating to its components as obtained from: (a) ultimate analysis, where C, H, O, N and S are 41.36, 6.60, 35.33, 7.17 and 0.55 wt.%, respectively; and (b) proximate analysis, where carbohydrate, protein and lipids are 25.59, 49.60 and 0.09 wt.%, respectively (Jamilatun et al., 2017a). The aforementioned data on the characteristics were obtained from tests conducted by TEKMIRA, Bandung, Indonesia.

The catalyst used for this study was silica-alumina (SiO_2/Al_2O_3) obtained from PTPN V, Sei Galuh, Riau Indonesia. Analysis, performed by LPPT UGM, revealed the Si/Al catalyst to contain O, Al and Si in the respective proportions of 8.41, 55.78, 24.64 and 11.17 wt.%. In addition, the catalyst pore surface area, diameter and average pore volume were 240.553 m^2/g surface area, 3.3 nm and 0.199 cm^3/g total pore volume, respectively.

2.2. Methods

The cylinder reactor consists of upper and lower parts in which to place the SPR and catalyst, respectively. A condenser was also installed for the purpose of liquefying volatile matter. This reactor has ID L dimensions of 40 mm × 60 cm. Pyrolysis was run at temperatures between 300 and 600°C at a heating rate of 20–40°C/min. A schematic image of the fixed-bed reactor is shown in Figure 1 (Jamilatun et al., 2017a).

1. Thermocouple
2. Reactor
3. Sample SPR
4. Heater
5. Catalyst Silica-alumina
6. Condensor
7. Accumulator of gas
8. Accumulator of bio-oil
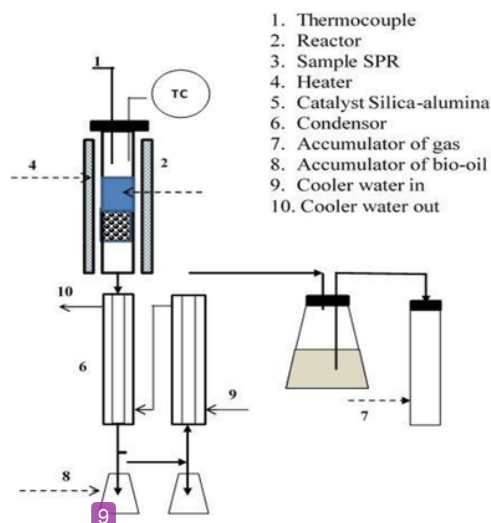9. Cooler water in
10. Cooler water out

Figure 1 Schematic diagram of the fixed-bed reactor

Fifty grams of SPR was inserted into the upper part of the reactor, which was then tightly closed. The amount of catalyst, which was contained in the lower part of the reactor, varied with reference to the cases of 10, 20 and 40 wt.%. Heating began from a temperature of 30°C and was increased to the desired temperature (300–600°C) at a rate of 3–35°C/minute. Once the desired temperature was reached, it was then maintained for a period of around 1 hour to ensure that the pyrolysis ran perfectly. The yields of bio-oil product, water phase, char and gas were calculated using the following equations, respectively (Jamilatun et al., 2017a).

$$Y_L = (W_L/W_M)x\ 100\ \% \tag{1}$$

$$Y_{Bo} = (W_{Bo}/W_M)\ x\ 100\ \% \tag{2}$$

$$Y_{Wp} = (W_A/W_M)\ x\ 100\ \% = Y_L - Y_{Bo} \tag{3}$$

$$Y_C = (W_C/W_M)\ x\ 100\ \% \tag{4}$$

$$Y_G = 1 - (Y_L + Y_C) \tag{5}$$

SPR (X) pyrolysis conversion can be determined using the following equation.

$$X = \frac{W_{Bo}+W_A+W_G}{W_M}x\ 100\ \% \tag{6}$$

In this case, W_M, W_L, W_{Bo}, W_{Wp}, W_C and W_G represent the weight of SPR, liquid, bio-oil, water phase, char and gas, respectively. While Y_L, Y_{Bo}, Y_{Wp}, Y_C and Y_G represent the yields of liquid, bio-oil, water phase, char and gas

3.   RESULTS AND DISCUSSION

3.1.   Characterization of Pyrolysis Products with Various SPR Grain Sizes and Temperatures

The yield results for pyrolysis products with varying sizes of SPR (0.105, 0.149 and 0.177 mm) and pyrolysis temperatures at a heating rate of 5–35°C/minute can be seen in Figure 2.

Figure 2a shows that the higher the pyrolysis temperature, the higher the bio-oil yield. The optimum temperature is 500°C. Any further increase in the temperature produces a lower bio-oil yield as a result of secondary cracking. This type of cracking results in the tar that was initially formed in the primary cracking being turned into gas and char. The evidence for this can be seen in Figures 2b –2d, where an increase in temperature generates a slightly higher water phase product, a much greater amount of gas at the temperature of 500°C and a sharply declining amount of char. In addition, the SPR grain size also affects the product yield. At each corresponding temperature, the larger the grain size, the smaller the yield of almost all products, except for char.

At 500ºC, the grain size increased from 0.105 to 0.177 mm, while the bio-oil yield was observed to decrease from 35.99 to 32.86 wt.%. In contrast, the percentage yield of char rose from 23.10 to 34.02 wt.%. Meanwhile, the others remained the same at all grain sizes, thus confirming the earlier study by Hu (2013). It was reported that the effect of grain size at a certain temperature (500ºC) will be stronger for bio-oil and char. Explaining this further, the increase in grain size produced a sharp decline in the bio-oil yield and a sharp rise in the char yield, whilst the gas and water phase yields remained fairly stable at all grain sizes.

It is generally assumed that an increase in grain size produces a greater temperature gradient in particles, so that at any given time the core temperature is lower than that at the surface of the biomass particles. Furthermore, it can lead to an increase in charcoal, while there are falls in the yield of gas and bio-oil. Small particles have a sufficient surface area to interact with pyrolysis media to form volatile products, thus leaving the biomass matrix without experiencing secondary reactions (Kan et al., 2016). Jalan and Srivastava (1999) stated that in the case of slow pyrolysis, particle size displayed a less significant effect on product yield. SPR pyrolysis conversion is calculated by summing the weight of the bio-oil, water phase and gas yields divided by the initial weight (Equation 6). An explanation of the increased pyrolysis conversion at temperatures of 300–600°C with variations in grain size can be seen in Figure 3.
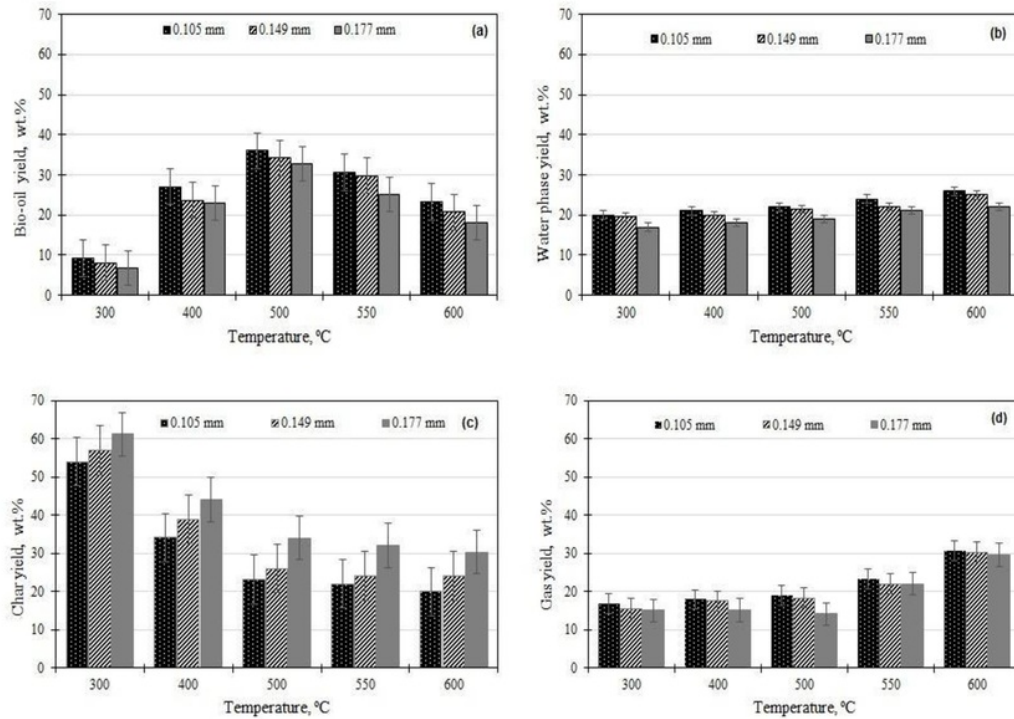
Figure 2 Effect of pyrolysis temperature and SPR grain size on yield of products: (a) yield of bio-oil; (b) yield of water phase; (c) yield of char; (d) yield of gas
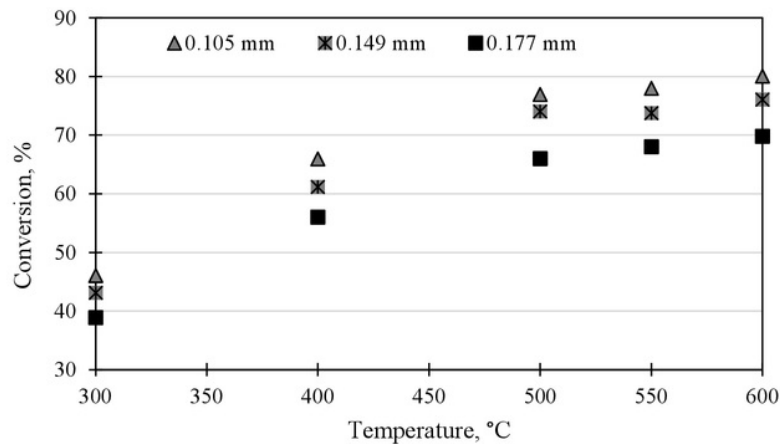

Figure 3 Relationship between grain size and conversion at various pyrolysis temperatures

Along with the increase in pyrolysis temperature and the smaller SPR grain size, the thermal decomposition is more effective; thus, the SPR weight decreases, causing the conversion to increase. The increased liquid and gas products indicate that the speed of the decomposition reaction increases in line with the increasing temperature and smaller grain size (Kan et al., 2016). Considering the low thermal conductivity of biomass, the biomass particles that provide rapid

heating to achieve a high bio-oil yield should be very small. Although a high heating rate can be achieved in a pyrolysis reactor, the low thermal conductivity of biomass inhibits temperature gradients across particles. Jalan and Srivastava (1999) observed that the pyrolysis process was controlled by primary pyrolysis reactions and external heat transfer for small particle sizes (< 1 mm), while for large particle sizes (> 1 mm) the process was controlled by heat transfer in conjunction with both primary and secondary pyrolysis.

3.2. Characterization of Pyrolysis Products with Varying Amounts of Catalyst

Figure 4 presents the pyrolysis product yields plotted on the y-axis against the catalyst percentage (0–40 wt.%). The pyrolysis process was initially conducted at four different temperatures of 400, 500, 550 and 600°C using the fixed variable of grain size set to 0.105 mm.
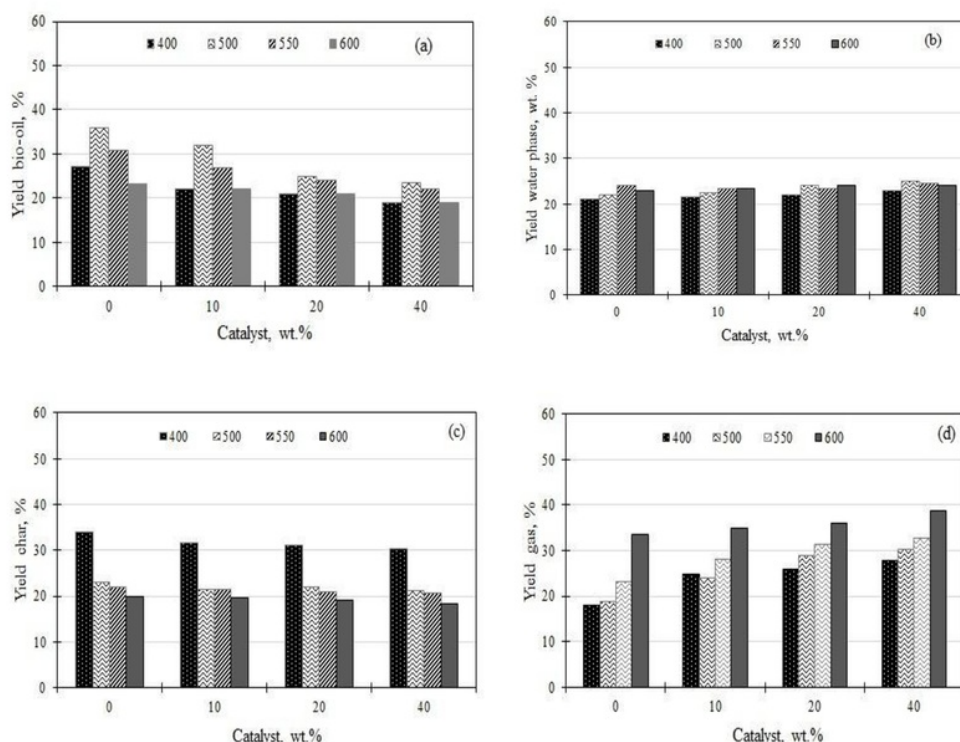

Figure 4 The effect of using catalysts on product composition in pyrolysis with an SPR grain size of 0.105 mm: (a) yield of bio-oil; (b) yield of water phase; (c) yield of char; (d) gas yield

From Figure 4a, for instance, the yield of bio-oil products at 500°C can be seen in the following case order, starting with the largest: without catalyst (35.99 wt.%) > with a catalyst of 10 wt.% (32.00 wt.%) > 20 wt.% (25.00 wt.%) > 40 wt.% (23.50 wt.%). Thus, an increase in the number of catalysts will eventually reduce the yield of bio-oil for all pyrolysis temperatures (400–600°C).

As presented in Figure 4b, the ranges for the water phase with the use of catalysts from 0 to 40% are from about 22.00±1.00% to 23.50±0.50% in a linear increasing order. This means that an increase in the amount of catalysts used creates a tendency for the yield of the water phase to increase. This finding is aligned with that from a study by Aysu (2015), where an increasing amount of catalysts led to deoxygenation (dehydration and hydrodeoxygenation) reactions occurring more quickly and effectively, thus generating increased water formation.

From Figures 4d and 4e, it can be explained that there is a decrease in the char yield and a sharp rise in the gas yield for all temperatures (400–600°C) in line with an increase in the number of catalysts. An increase in the number of catalysts will lead to an increase in conversion, where the number of non-condensable and condensable gas products increases, causing the char yield to decrease. The increase in gas yield (non-condensable gas) is caused by decarbonylation and decarboxylation reactions, while the increase in water is due to hydrodeoxygenation and hydrogenation reactions. Condensable gas (bio-oil) is influenced by all reactions, including cracking, hydrocracking, deamination, decarbonylation, decarboxylation, hydrodeoxygenation, hydrogenation and cyclisation (Dickerson & Soria, 2013; Qi et al., 2018).

Figure 5 shows the relationship between the number of catalysts and its conversion within the temperature range of 400–600°C. It can be seen that an increase in temperature from 400 to 600°C leads to an approximate 1% increase in the amount of converted product, from ±70 to 80%, regardless of the division into bio-oil, water phase, char and gas, as previously shown in Figure 4. In addition, the influence of the number of catalysts on the conversion is more pronounced at a lower temperature (at 400°C) than at a higher temperature. Interestingly, the catalytic pyrolysis process may not significantly influence the conversion, but instead tends to markedly affect the desired composition percentage. (Aysu, 2015).
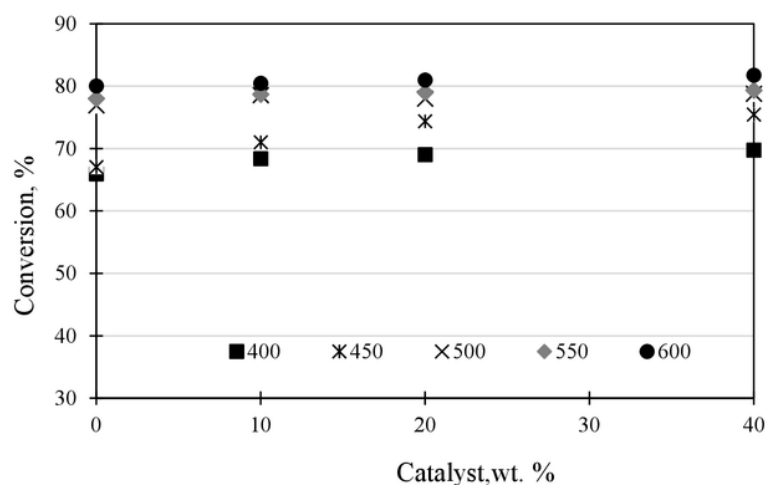

Figure 5 Relationship between the number of catalysts and conversion at various pyrolysis temperatures with an SPR grain size of 0.105 mm

3.3. Grouping of Bio-oil Fraction based on the Amount of C Atoms

The grouping into LPG, gasoline, diesel and heavy naphtha fractions is based merely on the number of C atoms available, as observed in the compounds contained in bio-oil, without considering the groups of alcohol, aldehydes, ketones, acids, nitrogenated or other compounds. From the results of GC-MS, more than 100 bio-oil constituents are obtained. The grouping of bio-oil products into the fractions of LPG (C ≤ 4), gasoline (C5–C11), diesel (C12–C18) and heavy naphtha (C ≥ 19) was performed by categorizing the number of C atoms in bio-oil constituent compounds. The grouping of the results into fractions at 400, 500, 550 and 600ºC pyrolysis temperatures can be observed in Table 1. From the table, it can be seen that LPG products highly dominate the products in pyrolysis with or without catalysts for all temperatures tested. It can be explained that by employing the pyrolysis process, the complex compounds contained in carbohydrates and proteins can be decomposed into smaller-size condensable compounds. These condensed liquids are mainly composed of compounds having C ≤ 4.

By using the catalyst, we can see that the fractions other than LPG (gasoline, diesel and heavy naphtha) begin to grow, despite LPG remaining as the dominant fraction. From the results of GC-MS, it can be seen that the LPG fraction comprises a compound with $C \leq 4$, likely by a methanol component, which has high potential as a product for use as a fuel or additive (Jamilatun et al., 2017a; Sun et al., 2019). The use of catalysts in pyrolysis is aimed at improving the quality of bio-oil by increasing the aromatic and short chain compounds $C \leq 4C$ and C5–C11 through cracking and deoxygenation reactions. The use of a catalyst significantly affects the composition of the product and increases conversion with increasing temperatures (Aysu, 2015). From catalytic and non-catalytic pyrolysis, it is expected that fuel with a fraction between LPG and gasoline will be produced. This study derived the optimum condition, with the best results obtained with the use of 10 wt.% catalyst at a temperature of 400–500°C.

Table 1 Bio-oil fraction without and with catalyst

Bio-oil fraction	Temperature, °C			
	400	500	550	600
Without catalyst				
LPG	96.30	80.16	85.50	98.32
Gasoline	1.73	4.14	9.19	0.33
Diesel	1.91	12.45	4.97	0.00
Heavy naphtha	0.09	3.25	0.33	1.36
Catalyst, 10 wt.%				
LPG	45.48	55.31	81.28	66.23
Gasoline	35.70	28.95	6.17	23.54
Diesel	14.73	12.92	9.28	9.59
Heavy naphtha	3.10	2.82	3.27	14.48
Catalyst, 20 wt.%				
LPG	73.03	69.64	86.36	22.58
Gasoline	5.03	18.91	4.66	58.70
Diesel	24.19	10.62	7.47	15.88
Heavy naphtha	2.40	0.84	1.51	2.87
Catalyst, 40 wt.%				
LPG	69.23	78.97	78.54	81.23
Gasoline	7.10	3.36	5.71	3.04
Diesel	8.39	14.36	12.50	13.83
Heavy naphtha	1.14	3.31	13.83	1.95

4. CONCLUSION

As a raw material source in the production of third-generation biofuel, *Spirulina platensis* residue offers the potential to be developed on a large scale due to its ease of cultivation, simple processing, pyrolysis technology and low cost. Temperature and the grain size of SPR microalgae affect the composition of the product. Bio-oil yield will increase from 9.22 to 33.99 wt.% at a temperature of 300–500°C, then fall to 23.34 wt.% at 600°C with an SPR grain size of at least 0.105 mm. This positive-peak curvature trend also applies to the sizes of 0.149 and 0.177 mm. However, the larger the grain size, the lower the bio-oil yield. An increase in grain size seems to produce a fall in the water phase, gas and its total conversion, whereas the opposite is shown for the yield of char. This applies to all temperatures (300–600°C).

The use of a catalyst affects the composition of the product. A greater amount of catalysts (0–40 wt.%) appears likely to result in a lower yield of bio-oil and char, with greater amounts of converted product obtained in the gas phase and fairly constant amounts in the water phase.

Based on the grouping of the number of C atoms in the bio-oil constituent, the dominant fraction is the LPG-Gasoline fraction, with methanol being the most highly available component. In the optimum condition at temperatures of 400–500°C, pyrolysis with the use of 10 wt.% catalyst produced about 45.48–55.31 wt.% of LPG fraction and 35.70–28.95 wt.% of gasoline fraction.

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