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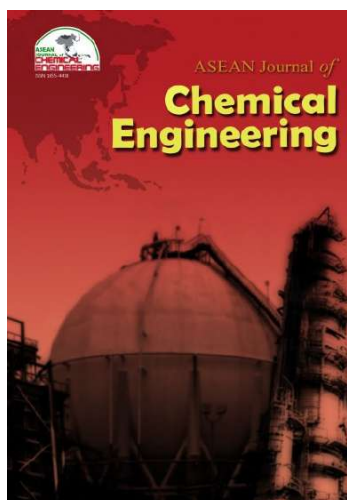
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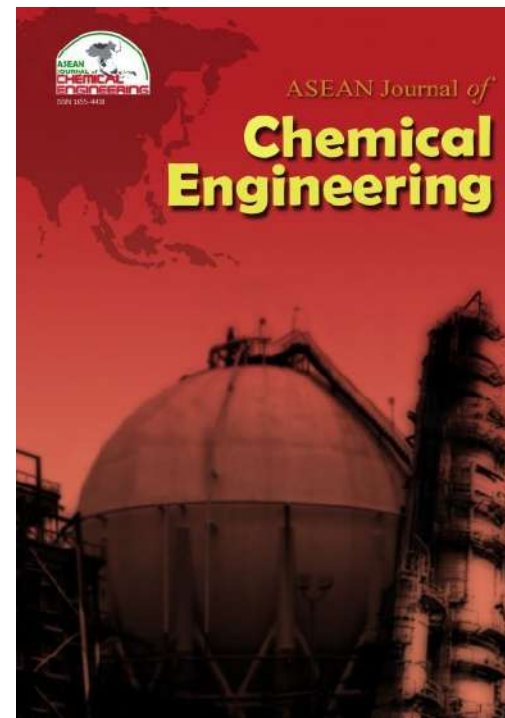
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The Effects of Particle Mesh and Temperature on Pyrolysis *Spirulina platensis* Residue (SPR): Pyrolysis Yield and Bio-Oil Properties

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Abstract. Microalgae is the third generation of biomass as renewable energy, a future energy source for making bio-oil. The purpose of this research is to examine the biomass from microalgae *Spirulina platensis* residue (SPR) using the pyrolysis process, to investigate the effect of particle mesh and temperature on the pyrolysis process, to determine the bio-oil properties, including density, pH, color, flame power, and conversion. Fixed bed reactor used for SPR pyrolysis with dimensions of 4.4 cm outside diameter, 4.0 cm inside diameter, and 60.0 cm reactor height. The temperature controls have been fitted from 300-600 °C combined with a 14-16 °C/minute heating rate. *Spirulina platensis* residue of 50 grams with various particle mesh (80 and 140 mesh) was fed to the reactor. From the experiment results, the particle mesh and temperature process are influenced by bio-oil yield, water phase, gas yield, biochar yield, conversion, and bio-oil properties, including density, pH, flame power, and color. One hundred forty mesh particles at a temperature of 500 °C showed the highest bio-oil yield with a yield of 22.92%, then the water, charcoal, and gas phases were 27.98, 18.84, and 30.26%, with a conversion of 81.16%. At the same time, 80 mesh particles at 500 °C yielded bio-oil, water, charcoal, and gas phases of 19.66, respectively; 23.10, 27.90, and 29.34%, with a conversion of 72.10%. In addition, density, pH, color, and flame power are described in this study.

Keywords: bio-oil yield, biochar, gas, water phase, pyrolysis, and *Spirulina platensis* residue.

INTRODUCTION

Microalgae is the third generation of biomass as renewable energy, a future energy source

(Chaiwong et al. 2013; Mathimani et al. 2019). In addition, microalgae can overgrow in wastewater with a high CO₂ level (Ferreira et al. 2021), dairy effluent (Vieira Costa et al.

2021) to reduce the cost of production, piggery wastewater (Ferreira et al. 2021) and can overgrow in open ponds (Fenton and ÓhUallacháin 2012).

Microalgae cultivation can reduce the greenhouse effect by using photobioreactors (Salazar et al. 2021; Kong et al. 2007). In photobioreactor, photosynthesis of microalgae takes place. Photosynthesis converts CO₂ in the air into O₂ with the help of light. The criterion by which the success rate of the process becomes is the amount of O₂ obtained. The greater the amount of O₂ produced, the more CO₂ is converted. This condition indicates that the photosynthesis process in the photobioreactor effectively reduces the concentration of CO₂ in the air and prevents global warming (Salazar et al. 2021; Huo et al. 2020). There are many advantages of microalgae as follows: high lipid content (Li et al. 2014), carbon hydrate (Aniza et al. 2021), protein, and other chemicals (Hallenbeck et al. 2016; Kadir et al. 2018). According to (Chowdhury and Loganathan 2019), the development of microalgae has many advantages over alternative biomass sources of energy, such as those with a high percentage of biomass production, struggling with food, and no need for a considerable amount of land to cultivate. Microalgae include lipids, fatty acids, and potential biofuel components (Jay et al. 2018; Chen et al. 2018). Lipids produced from microalgae have C16 and C18, which can be esterified to produce high-quality biodiesel (Mahmoud et al. 2015; Zighmi et al. 2017). Microalgae can have an oil concentration of up to 80% by mass of dry biomass (Schlagermann et al. 2012; Kong et al. 2007).

The pyrolysis process converts microalgae into three forms of sustainable energy: bio-oil, charcoal, and non-condensable gas. (Yang

et al. 2019; Azizi et al. 2020). The pyrolysis technique is a method for decomposing organic materials at room temperature using heat. (Jamilatun et al. 2019) has investigated the pyrolysis of residues of *Spirulina platensis* with a calorific value of bio-oil 20.46-33.62 MJ/kg. At the same time, (Chen et al. 2015) investigated the pyrolysis of residues of *Chlorella Vulgaris* (24.57 35.10 MJ/kg), *Chlorella sorokiniana* CY1 residue (20.24 MJ/kg), and *Nannochloropsis Oceanica* residue (32.3339 MJ/kg). Almost all bio-oil from microalgae has a high calorific value (Chen et al. 2015). The calorific value of bio-oil from microalgae is more significant than from lignocellulosic biomass sources. For coconut shells, the calorific value is 21.28 MJ/kg, while bagasse is 21.28 MJ/kg (Azeta et al. 2021). Each pyrolysis product has its benefits if further processed; for example, It is possible to utilize o-oil and o-gas as marine material. The water phase is used as an additive for food preservation and contains components as a supplement. In contrast, biochar may be an adsorbent in various applications, including food, sewage treatment, and chemicals.

Extraction of bio-oil from microalgae yields solid wastes that can be utilized as a source of raw materials for biofuel production (Trugnanasambantham et al. 2020). *Spirulina platensis* residue (SPR) is a solid residue with a high carbohydrate and protein content. Using solid leftovers of microalgae with low lipid content, such as *Spirulina* sp. (4-9 percent lipids), as a raw material for pyrolysis can boost bio-oil output by about 40% (Suganya et al. 2016). The drawback of bio-oil from microalgae is that the oxygenated and nitrogenated content is still high, which causes instability in its use.

Recently, much research has been conducted on microalgae to convert valuable

products. (Wang et al. 2013) reported similar observations in their experiment from *Chlorella Vulgaris* microalgae that contains: romantic, amides, amines, carboxylic acid, phenol, and other compounds. They also reported that microalgae could be converted by fast pyrolysis. A wide variety of parameters affect the performance of the pyrolysis, including temperature, size and shape, heating rate, residence duration, and catalyst, (Belotti et al. 2014; Ly et al. 2016; Yanik et al. 2013; Du et al. 2013). The temperature factor is crucial and essential because it can affect the composition of each product. Based on (Dutta et al. 2016) , secondary cracks will occur if the operating temperature increases beyond the optimum temperature, resulting in bio-oil output decreases while gas production rises.

This research aimed to investigate particle mesh and temperature process's effect on slow pyrolysis of microalgae *Spirulina platensis* with a range heating rate used is 14-16 °C/minute. The factors that affect the pyrolysis process of *Spirulina platensis* are reaction time, material size, pyrolysis temperature, heating rate, and material content (Jamilatun et al. 2020) . The particle mesh of 80 and 140 mesh was applied in the system, while the temperature was performed at 300, 400, 500, 550, and 600 °C. The dimensions of the fixed-bed reactor used are 4.4 cm outside diameter, 4.0 cm inside diameter, and 60.0 cm reactor height.

RESEARCH METHODOLOGY

Materials

This section summarizes that the primary raw material of this research was obtained from solid residues of *Spirulina platensis* and extracted with methanol (CH₃OH). Based on a

previous study (Jamilatun et al. 2019), the ultimate and HHV from SPR were analyzed to know the characteristic of the SPR, where HHV is the heat value obtained from combustion.

The component of SPR includes lipid of 0.09%, carbohydrate of 38.51, and protein of 49.60. The ultimate analysis of SPR contains sulfur of 0.55% and carbon of 41.36%. The hydrogen was 6.60%, the nitrogen was 7.17%, and oxygen was 35.33%. HHV of SPR is 18.21 MJ/kg. SPR as raw material in this research was used at 50 grams with the difference in size, including 140 and 80 mesh.

Procedures

The research procedure consists of 4 main steps: SPR preparation, pyrolysis preparation, pyrolysis process, and determining the properties of bio-oil (density, pH, color, and flammability).

SPR Preparation

The Chemical Engineering UGM Eco Mini Plant laboratory provided *Spirulina platensis* residue (SPR). SPR is mashed by pounding or using a blender. Then, sieving the SPR using a sieve of 80 and 140 mesh size. This sizing is to make a high heating transfer between SPR and Heat.

Preparation of Pyrolysis Equipment

This research is developed from a previous study (Bridgwater 2012; Jamilatun et al. 2020). The pyrolysis system in this research can be seen in Figure 1.

The design of the pyrolysis device can be seen in Figure 1. The reactor was installed at 4.4 cm outside diameter, 4.0 cm inside diameter, and 60.0 cm reactor height. The effect of temperature and pyrolysis time on heating speed can be determined by controlling the pyrolysis temperature. The

heater and thermocouple were used as the temperature control in this research.

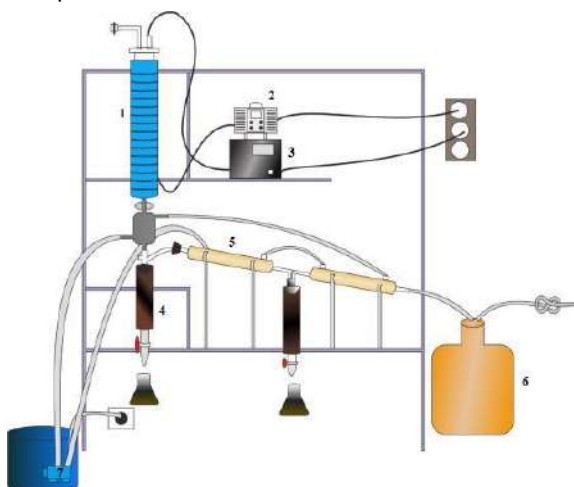


Fig. 1: Pyrolysis equipment

where: 1. Reactor

2. Heater

3. Thermocouple

4. Accumulator of bio-oil

5. Condenser

6. Accumulator of gas

7. Pump

Pyrolysis Process

This research was conducted with biomass (80 mesh and 140 mesh). The biomass was blended and sieved to get the different sizes of SPR. The SPR as a raw material was fed in the reactor with 80 mesh. Then the reactor is tightly closed. The heating rate was flowed from 30 °C with intervals of 5 minutes and increased using a voltmeter to the desired temperature (300–600 °C) at a speed of 14–16 °C/minutes. After the desired temperature is reached, it is maintained for about 1 hour to ensure the pyrolysis is complete. The liquid yields are collected in the accumulator while the gas yields are absorbed with air in gallons. Before testing with other size materials, the reactor must be refrigerated. Repeat the process for SPR with a particle mesh of 140

mesh with a temperature range of 300–600 °C. The results of bio-oil products, water phase, char, and gas are calculated using the following equation below. The value was calculated for each pyrolysis as described as follows:

$$Y_L = \frac{W_L}{W_M} \times 100 \% \quad (1)$$

$$Y_B = \frac{W_B}{W_M} \times 100 \% \quad (2)$$

$$Y_{WP} = \frac{W_{WP}}{W_M} \times 100 \% \quad (3)$$

$$Y_C = \frac{W_C}{W_M} \times 100 \% \quad (4)$$

$$Y_G = 100 \% - (Y_B + Y_{WP} + Y_C)\% \quad (5)$$

$$Y_L = Y_B + Y_{WP} \quad (6)$$

$$X = \frac{(W_B + W_{WP} + W_C)}{w_M} \times 100 \% \quad (7)$$

The following symbols are Y_L , Y_B , Y_{WP} , Y_C , and Y_G , which can be interpreted as the total yield of liquid, bio-oil, water, biochar, and gas phases, respectively. Meanwhile, W_M , W_L , W_B , W_{WP} , and W_C can be interpreted as the sample weight of SPR biomass, liquid product, bio-oil, water phase, and charcoal, respectively. Conversion using the symbol X .

Bio-oil properties

These properties are arranged in 4 types as follows:

a. Measure bio-oil density

The way to measure bio-oil density is first to weigh and record the weight of an empty bottle, then fill the bottle with distilled water according to the volume of bio-oil obtained. Then, consider the bottle that is filled with distilled water. Finally, calculate the density with the following formula:

$$\text{Density } (\rho) = \frac{\text{mass of biooil}}{\text{Volume of bio-oil}} \quad (8)$$

b. Bio-oil color

The colors of bio-oil in this section are investigated and recorded with notes and take a picture of bio-oil.

c. pH

Measure the pH of bio-oil using a pH meter by dipping the pH meter into bio-oil. Then note the pH value printed in the tool.

d. Bio-oil flammability

The technology to measure the flammability of bio-oil, the flame power is determined by burning a little of the bio-oil while calculating the time it takes for the bio-oil to ignite.

RESULTS AND DISCUSSION

Bio-oil products: Effect of pyrolysis temperature and particle mesh

A visual representation of this result can be seen in Figure 2. This research uses a temperature process from 300-600 °C and particle-sized of 80 mesh and 140 mesh. Each experiment showed that the yield of bio-oil at a particle mesh of 80 mesh and in the temperature ranges: 300, 400, 500, 550, and 600 °C was 12.54, 19.62, 19.66, 13.96, and 14.12%. Meanwhile, the bio-oil yield at 140 mesh and processing temperatures: of 300, 400, 500, 550, and 600 °C was 14.9, 22.24, 22.92, 15.18, and 15.66%. Based on these results, the bio-oil yield was influenced by the particle mesh SPR as a raw material in this research.

Based on Figure 3, During the reaction temperature process, the bio-oil increases from temperature 300 to 500 °C. The bio-oil yield decreases from temperature 500 to 600 °C. This condition occurs in both particle

meshes of biomass, 80 mesh and 140 mesh. The optimum reaction temperature of SPR occurs at temperatures 400 and 500 °C. This result is consistent with previous research (Angin 2013; Tokarchuk, et al. 2021; Mishra and Mohanty 2020). However, this phenomenon is affected by secondary cracking in which the pyrolysis yields are active to undergo secondary reaction to produce the high bio-oil. In addition, gas and charcoal production takes place in the primary cracking.

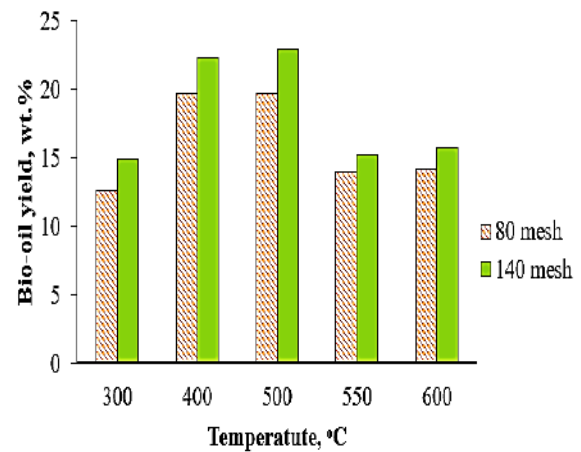


Fig. 2: Effect of temperature and SPR particle mesh on bio-oil yield

Following the experiment from (Ma et al. 2018; Treedet et al. 2020), the pyrolysis process increased with the increase of the thermal cracking reaction, which this condition increased the gas and liquid production. The decomposition of biomass leads to the formation of phenol and CH₄ (Jamilatun et al. 2019). Hence at a temperature of 500 °C, the maximum bio-oil is formed and will decrease at a temperature of 550 °C due to secondary reactions (Jamilatun et al. 2020).

The influence of particle mesh on bio-oil yield is identified as the more significant the particle mesh, the lower the bio-oil yields.

This result is relevant to previous research from (Qureshi et al. 2021), who also reported the effect of particle mesh of bagasse from 0.5 mm to 1.4 mm, with the result that the liquid yield increased from 25%-48%. Then the liquid product gradually decreased due to heat transfer limitation at the particle. A more significant temperature gradient was influenced by the increase in the particle mesh in the particles. At a specific time, the core temperature is lower than the surface of the biomass particles, which can cause an increase in biochar. At the same time, the production of gas and bio-oil decreases. Fine particles have sufficient surface area to interact with pyrolysis media to form volatile products such as gas, thus leaving the biomass matrix without experiencing secondary reactions (Aliyu et al. 2021).

Furthermore, it can be said that the particle mesh of the material is directly related to the area of the contact area, including affecting the heat transfer rate and mass. The relationship between particle mesh, contact area, and mass and heat transfer rates, namely, when a material has smaller particles, the surface area will be larger, which causes the contact area of the material to be more significant and ideal. So the distribution of heat and mass can run well into the particles, which causes the mass and heat transfer rates to run perfectly. And vice versa for large particle mesh.

Bio-oil products: Effect of temperature and particle mesh

The effect of temperature and SPR particle mesh on the yield water phase can be seen in Figure 3. Respectively. The water phase with a particle mesh of 80 was obtained at 21.08, 22.40, 23.10, 25.10, and 26.06% with temperatures of 300, 400, 500, 550, and

600 °C, respectively. At the same time, the water phase with a particle mesh of 140 mesh was obtained 25.82, 27.58, 27.98, 32.46, and 33.28%, respectively. Based on the results, it can be seen that the more significant the temperature, the water phase yield value increases; this is because the high temperature can convert the water phase that is in the SPR optimally (Jamilatun et al. 2020). Next, also secondary decomposition reactions occur when the pyrolysis temperatures are very high (Ly et al. 2016). This result was relevant to (Aguilar et al. 2015) , who investigated the pyrolysis effect in a fixed bed reactor with the result that the water phase increased from 17.99 to 21.05% at temperatures ranging from 400 to 500 °C. According to (Jamilatun et al. 2019), the larger the SPR particle mesh used, the lower the yield water phase. Because the particle mesh of the material is directly related to the contact area, it was affected by the speed of heat and mass transfer to convert SPR into a water phase. The yield water phase is also very dependent on the water content of the material and the formation of water during the pyrolysis process.

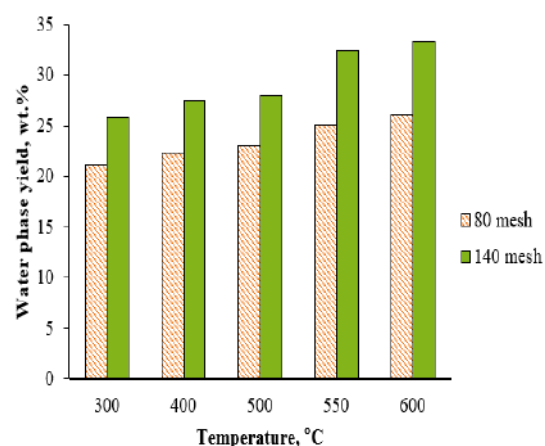


Fig. 3: Effect of temperature and SPR particle mesh on water phase yield

Biochar products: Effect of temperature and particle mesh.

The effect of temperature and SPR particle mesh on the yield of biochar is depicted in Figure 4. The experiment result indicated that the yield of biochar with a particle mesh of 80 mesh was 39.14, 30.12, 27.90, 27.68, and 24.1%, respectively. The biochar yield with a particle mesh of 140 mesh was 29.98, 20.58, 18.84, 17.72, and 14.56%, respectively. The temperature process was installed at 300, 400, 500, 550, and 600 °C. In Figure 4, it can be seen that the biochar yield gradually decreased when the temperature was too high and the particle of 80 mesh and 140 mesh. These results are relevant to (Sun et al. 2014). In general, moisture and hydration water loss occurs at a temperature under 250 °C. In contrast, temperatures above 150 °C decompose and transform into a vapor containing organic compounds and gases (Asadullah et al. 2007; Asadullah et al. 2013). In addition, the decreasing biochar yield from low to high temperature was affected by organic matter decomposed when the pyrolysis was at high temperature (Torri et al. 2016).

The effect of particle mesh on char yield indicated that the char yield depended on particle mesh. The result of particle mesh on the outcome of biochar shows that the development of charcoal depends on the particle mesh. Larger particles require more time to limit heat transfer between particles and supply heat due to the small contact surface area of the material. In addition, the larger biomass particle was supported for the secondary charcoal (Somerville and Deev 2020). This study is by (Jamilatun et al. 2019) the more significant the SPR particle mesh used, the greater the biochar yield obtained. An increase in particle mesh was affected by the more substantial temperature gradient in

the particles. Furthermore, the core temperature is lower than the surface of the biomass particles, which can cause an increase in charcoal, while the production of gas and bio-oil decreases (Garg et al. 2016; Jamilatun et al. 2020).

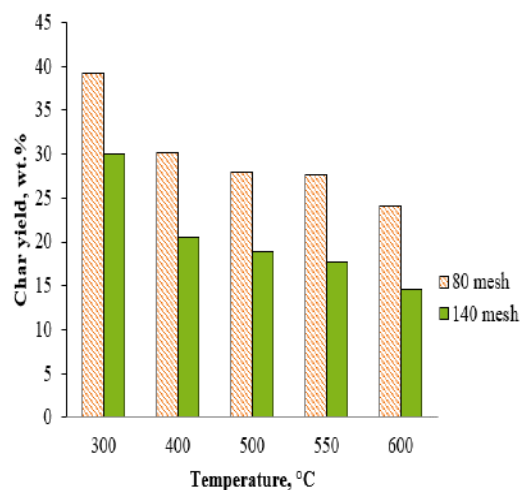


Fig. 4: Effect of temperature and SPR particle mesh on char yield

Gas Yield: Effect of temperature and particles mesh

Figure 5 shows the effect of temperature and biomass particles on gas yield. The experiment indicated that the gas yield with a particle mesh of 80 mesh was 27.24, 27.86, 29.34, and 33.26%, respectively. At the same time, the gas yield with a particle mesh of 140 mesh was 29.30, 29.60, 30.26, 34.64, and 36.5%, respectively. The pyrolysis temperature was applied at 300, 400, 500, 550, and 600 °C. The tendency of the higher the pyrolysis temperature, the greater the gas yield is depicted in Figure 5. According to (Garg et al. 2016), the gas yield was significantly affected by the loss of volatile matter or secondary decomposition at high temperatures, which causes increased gas production. These results are relevant to (Aguilar et al. 2015), who reported the effect of biomass particle mesh from Chinese tallow

trees. They investigated that the small particle takes place reasonable heat transfer rate causing a higher overall temperature. According to (Hong et al. 2017), the larger the SPR particle mesh used, the smaller the gas yield obtained. This phenomenon can occur because the decomposition process is less than optimal at a large particle mesh due to the small contact area. It can affect the low heat and mass transfer speed to convert SPR into gas.

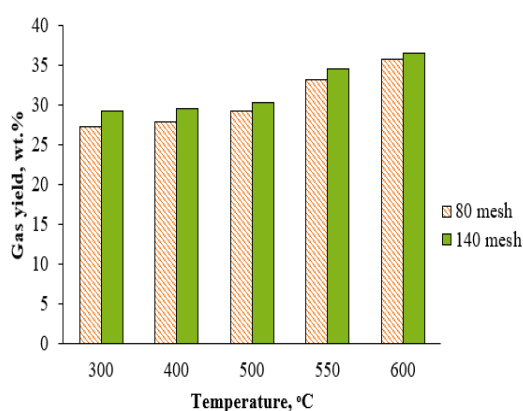


Fig. 5: Effect of temperature and SPR particle mesh on gas yield

Bio-oil conversion

Figure 6 shows the conversion of pyrolysis product with a particle mesh of 80 mesh and 140 mesh under temperature process at 300, 400, 500, 550, and 600 °C. The experiment results from the biomass particle mesh of 80 mesh were 60.86, 69.88, 72.10, 72.32, and 75.90%, respectively. At the same time, the conversion of pyrolysis products with a particle mesh of 140 mesh with the same temperature set was 70.02, 79.42, 81.16, 82.28, and 85.44%, respectively. Based on the pyrolysis results obtained, the higher the temperature, the higher the product conversion can be seen in Figure 6. This result was relevant to (Parthasarathy et al. 2021). They reported the conversion in the heating rate of 10, 20, and 50°C/min at a temperature

ranging from 200 to 530 °C. The larger the SPR particle mesh used, the smaller the conversion of pyrolysis products obtained. From Figure 6, it can be concluded that the relationship between temperature, particle mesh, and transformation of pyrolysis products, namely an increase in pyrolysis temperature. A smaller SPR particle mesh causes thermal decomposition to work more effectively, decreasing the SPR weight.

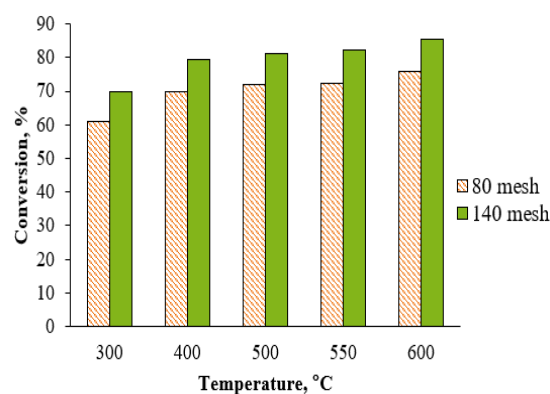


Fig. 6: Effect of temperature on various amounts of particle mesh on conversion

Density of Bio-oil

There are given in Table 1. the bio-oil density with particle mesh (80 mesh and 140 mesh) and temperature settings of 300, 400, 500, 550, and 600 °C. The research results indicate that the value of the density of bio-oil changes fluctuates with every temperature increase. Many chemical compounds cause the high density of bio-oil in the bio-oil, which has a high molecular weight. The more the presentation of a substance with a high molecular weight, the higher the density of the fuel solution. In contrast to petroleum fuels with a low percentage of heavy molecular substances, their density tends to be lighter. The chemical compound in power with high molecular weight will complicate

the evaporation process in the engine combustion chamber and tend not to burn completely. Previous research from (Kadir et al. 2018) explained why bio-oil density is 0.94-1.21 gram/ml. From table 2, the resulting density is 1.030-1.163 grams/ml. The smaller the density of bio-oil, the better it is used as a fuel because it is lighter. The effect of density on the power, namely the lower density, increased the ignition of the fuel. Hence, the energy quickly burned because high calorific value (Chukwunke et al. 2019).

Table 1. Density of bio-oil

Density of Bio-oil (gram/ml)		
Temperature (°C)	Particle mesh 80	(mesh) 140
300	1.081	1.049
400	1.154	1.030
500	1.035	1.032
550	1.163	1.069
600	1.086	1.073

pH of Bio-oil

The measuring pH in this research was conducted in 80 mesh and 140 mesh particles. The temperature process was set at 550 °C. This analysis was to know the pH level in bio-oil by using a pH meter. The experiment result found that the particle mesh of 80 mesh at a temperature of 550 °C with a pH level of 8.9. In comparison, the particle mesh of 140 at a temperature of 550 °C has obtained a pH level of 9.0. Relevant works by these researchers (Yang et al. 2019) are the leading methods on the pH of microalgae bio-oil at a pH of 8-9.9. In addition, previous research from (Borges et al. 2014) reported similar observations in bio-oil pH from *Chlorellas* of 9.33 and *Nannochloropsis* of 9.93. The pH alkaline of

bio-oil was affected by the presence of nitrogen in the bio-oil.

There are several ways of improving the bio-oil, including hydrotreating, hydrocracking, steam reforming, and others where compounds with high molecular weight are split into alkane compounds (Shan Ahamed et al. 2021). (Bertero et al. 2012) states that the acetic acid content in bio-oil depends on the biomass material; generally, the content is between 15-59% by weight.

Flame Power of Bio-oil

The bio-oil flame power test of *Spirulina platensis* residue (SPR) was carried out to determine the bio-oils ability to ignite when given a fire source. After the flame test experiment was carried out, the results showed that the flame power of bio-oil for temperatures of 300-600 °C is in a slow category (lights up for more than 6 seconds). This condition is due to the high content of phenol in the bio-oil. It is necessary to do further treatment to increase the flame power of the bio-oil. In contrast to the ignition power of gasoline or alcohol (0-2 seconds) (Santiyo et al. 2015). The flame of bio-oil in this research can be seen in Figure 7.



Fig. 7: Flame power of bio-oil

Color of Bio-oil

Based on the experiment results, the color of the bio-oil was blackish-brown which can be seen in Figure 8. When the temperature

increases, the black color level of bio-oil will be higher. These results were compared with previous research from (Chukwunke et al. 2019; Wądrzyk et al. 2018). The color of the bio-oil from (Wądrzyk et al. 2018) was dark-brown, while the bio-oil from (Chukwunke et al. 2019) was pale-brown. The different color of bio-oil was affected by the occurring reaction between carbohydrates and amino acid, which this reaction made the presence of Chromophore structure.



Fig. 8: Color of bio-oil

CONCLUSIONS

SPR has excellent potential to be developed as renewable energy using the pyrolysis method. Based on the research results, it was found that temperature and particle mesh greatly affected the pyrolysis yield. The higher the pyrolysis temperature, the higher the conversion, and the bigger the particle mesh, the lower the work of bio-oil, water phase, and gas for all temperatures tested. However, this is inversely proportional to the solid product, namely, biochar, where the more significant the particle mesh, the greater the biochar yield. The highest outcome of bio-oil products was at 500 °C with a particle mesh of 140 mesh of 22.92%. The optimal temperature for SPR pyrolysis is

at 500 °C. Product conversion is around 60-85%. The bio-oil product has properties such as a density of about 1.030-1.163 grams/ml, a pH of approximately 8-9, the color of the bio-oil is blackish-brown, and the flame power of the bio-oil is relatively slow-medium.

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